Supplementary Material

An efficient synthesis of 3-ethoxypyrrolidine-2,5-diones and *cis*-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-diones from *β*-cyanocarboxylic acids

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General Experimental Methods

3-Ethoxypyrrolidine-2,5-diones **6a-k,** *cis*-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-diones **7a-k**, and *bis*-3-ethoxypyrrolidine-2,5-diones **11-13** were identified by ^{1}H and ^{13}C NMR techniques, mass spectrometry (GC-MS), and High Resolution Mass Spectrometry analysis. The correct assignment of the ^{1}H NMR spectra of the products **6a-k**, **7a-k**, and **11-13** was carried out using 2D COSY experiment.

The synthesis of 4-alkoxyvinyl trichloromethyl ketones **1** and **2** and the synthesis of *β*-cyanocarboxylic acids **3** and **4** are reported in the literature.[1,2] The High Resolution Mass Spectra (HRMS) were registered on a LC-MS-TOF Bruker Daltonics Micro Ic from the Department of Chemistry of University of São Paulo (USP), São Paulo, SP, Brazil. The mass spectra were registered on an HP 5973 MSD connected to an HP 6890 GC. The GC was equipped with a split-splitless injector, auto-sampler, cross-linked HP-5 capillary column (30 m, 0.32 mm of internal diameter), and helium was used as the carrier gas. The ^{1}H and ^{13}C NMR spectra were acquired on a Bruker DPX200 or DPX400 spectrometer in $CDCl_3$ with TMS as the internal reference.

Synthesis of N-Alkyl(aryl)-3-ethoxypyrrolidine-2,5-diones 6a-k and N-alkyl(aryl)-cis-2,3,3a,6a-tetrahydrofuro[2,3-c]pyrrole-4,6(5H)-diones 7a-k, General procedure:

To a solution of 3-cyano-3-ethoxypropionic acid **3** or 2-cyanotetrahydrofuran-3-carboxylic acid **4** (5.0 mmol) in water (10 mL), amines **5a-k** (5.0 mmol) was added. The reaction mixture was stirred at 140°C until all the water was distilled off. After the distillation of the water, the temperature of the oil bath was raised to 180°C and the reaction was maintained at this temperature for 1.5 hours. The reaction was cooled to room temperature and chloroform (30 mL) was added to the reaction flask in order to solubilize the remaining oil in the flask. The solution was dried with anhydrous sodium sulfate, filtered and evaporated. Compounds **6** and **7** were obtained as brown or yellow oils in good yields and were purified by column chromatography on silica gel as the stationary phase and 50% chloroform/ethyl acetate solution as the mobile phase.

Spectrometric dada of compounds 6a-k and 7a-k (^{1}H NMR, ^{13}C NMR, GC-MS, and HRMS):

3-Ethoxy-1-(pyridin-2-ylmethyl)pyrrolidine-2,5-dione (6a). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.26 (t, 3H, $J_{H7\text{-}H6}$ = 7.0, H–7), 2.73 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.2, $J_{H4\text{-}H3}$ = 4.4, H–4), 3.12 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.3, $J_{H4'\text{-}H3}$ = 8.2, H–4'), 3.63-3.78 (m, 1H, H–6), 3.95-4.10 (m, 1H, H–6'), 4.43 (dd, 1H, $J_{H3\text{-}H4'}$ = 8.1, $J_{H3\text{-}H4}$ = 4.4, H–3), 4.83 (s, 2H, H–8), 7.14-7.26 (m, 2H, H–11, H–13), 7.64 (td, 1H, $J_{H12\text{-}H11,\,H13}$ = 7.8, $J_{H12\text{-}H10}$ = 1.8, H–12), 8.51 (d, 1H, $J_{H10\text{-}H11}$ = 4.8, H–10). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 15.1 (C–7), 36.3 (C–4), 43.2 (C–8), 66.9 (C–6), 73.4 (C–3), 121.8 (C–11), 122.5 (C–13), 136.6 (C–12), 149.5 (C–10), 154.1 (C–9), 174.0 (C–2), 175.7 (C–5). GC-MS

(EI, 70eV) *m/z* (%): 190 (MH^+ - 45, 100), 162 (4), 135 (38), 93 (47). GC-MS (CI) *m/z* (%): 235 (MH^+, 100), 263 (10). HRMS (ESI-TOF) Calcd for $C_{12}H_{15}N_2O_3$: $[M+H]^+$ 235.1082. Found: 235.1087.

3-Ethoxy-1-(pyridin-3-ylmethyl)pyrrolidine-2,5-dione (6b). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.24 (t, 3H, $J_{H7\text{-}H6}$ = 7.2, H–7), 2.65 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.4, H–4), 3.03 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.4, $J_{H4'\text{-}H3}$ = 8.0, H–4'), 3.64-3.72 (m, 1H, H–6), 3.95-4.02 (m, 1H, H–6'), 4.32 (dd, 1H, $J_{H3\text{-}H4'}$ = 8.4, $J_{H3\text{-}H4}$ = 4.0, H–3), 4.66 (s, 2H, H–8), 7.24-7.27 (m, 1H, H–12), 7.71 (dt, 1H, $J_{H13\text{-}H12}$ = 7.6, $J_{H13\text{-}H10,\ H11}$ = 2,4, H–13), 8.53 (d, 1H, $J_{H11\text{-}H12}$ = 6.4, H–11), 8.63 (s, 1H, H–10). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 15.0 (C–7), 36.3 (C–4), 39.7 (C–8), 67.1 (C–6), 73.2 (C–3), 123.5 (C–12), 131.1 (C–9), 136.7 (C–13), 149.4 (C–11), 150.1 (C–10), 173.7 (C–2), 175.3 (C–5). GC-MS (EI, 70eV) *m/z* (%): 190 (MH^+ - 45, 100), 162 (4), 135 (18), 92 (57), 72 (29). HRMS (ESI-TOF) Calcd for $C_{12}H_{15}N_2O_3$: $[M+H]^+$ 235.1082. Found: 235.1079.

3-Ethoxy-1-(pyridin-4-ylmethyl)pyrrolidine-2,5-dione (6c). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.25 (m, 3H, $J_{H7\text{-}H6}$ = 7.0, H–7), 2.69 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.2, $J_{H4\text{-}H3}$ = 4.2, H–4), 3.07 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.1, $J_{H4'\text{-}H3}$ = 8.2, H–4'), 3.62-3.77 (m, 1H, H–6), 3.93-4.08 (m, 1H, H–6'), 4.36 (dd, 1H, $J_{H3\text{-}H4'}$ = 7.9, $J_{H3\text{-}H4}$ = 4.2, H–3), 4.65 (s, 2H, H–8), 7.24 (d, 2H, $J_{H10\text{-}H11}$ = 6.0, H–10), 8.56 (d, 2H, $J_{H11\text{-}H10}$ = 6.0, H–11). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 15.0 (C–7), 36.3 (C–4), 41.0 (C–8), 67.1 (C–6), 73.2 (C–3), 123.2 (C–10), 143.7 (C–9), 150.1 (C–11), 173.6 (C–2), 175.3 (C–5). GC-MS (EI, 70eV) *m/z* (%):190 (MH^+ - 45, 100), 161 (10), 135 (7), 92 (9), 72 (22), 55 (11). HRMS (ESI-TOF) Calcd for $C_{12}H_{15}N_2O_3$: $[M+H]^+$ 235.1082. Found: 235.1080.

3-Ethoxy-1-(phenylmethyl)pyrrolidine-2,5-dione (6d). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.23 (t, 3H, $J_{H7\text{-}H6}$ = 6.8, H–7), 2.63 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.4, $J_{H4\text{-}H3}$ = 4.0, H–4), 3.00 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.0, $J_{H4'\text{-}H3}$ = 8.4, H–4'), 3.63-3.71 (m, 1H, H–6), 3.95-4.02 (m, 1H, H–6'), 4.29 (dd, 1H, $J_{H3\text{-}H4'}$ = 8.4, $J_{H3\text{-}H4}$ = 4.0, H–3), 4.65 (s, 2H, H–8), 7.26-7.38 (m, 5H, H–10, H–11, H–12). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.0 (C–7), 36.2 (C–4), 42.2 (C–8), 66.9 (C–6), 73.2 (C–3), 127.9 (C–12), 128.6 (C–10), 128.7 (C–11), 135.3 (C–9), 173.9 (C–2), 175.6(C–5). GC-MS (EI, 70eV) *m/z* (%): 233 (M^+, 3), 189 (MH^+ - 45, 100), 132 (100), 91 (100), 55 (54). HRMS (ESI-TOF) Calcd for $C_{13}H_{16}NO_3$: $[M+H]^+$ 234.1130. Found: 234.1127.

3-Ethoxy-1-(2-phenylethyl)pyrrolidine-2,5-dione (6e). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.23 (t, 3H, $J_{H7\text{-}H6}$ = 7.2, H–7), 2.57 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.4, H–4), 2.87-2.97 (m, 3H, H–9, H–4'), 3.60-3.67 (m, 1H, H–6), 3.74 (t, 2H, $J_{H8\text{-}H9}$ = 8.0, H–8), 3.90-3.98 (m, 1H, H–6'), 4.23 (dd, 1H, $J_{H3\text{-}H4'}$ = 8.0, $J_{H3\text{-}H4}$= 4.0, H–3), 7.19-7.31 (m, 5H, H–11, H–12, H–13). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.0 (C–7), 33.3 (C–9), 36.1 (C–4), 39.6 (C–8), 66.7 (C–6), 73.0 (C–3), 126.6 (C–13), 128.6 (C–11), 128.7 (C–12), 137.5 (C–10), 174.0 (C–2), 175.6 (C–5). GC-MS (EI, 70eV) *m/z* (%): 247 (M^+, 1), 203 (MH^+ - 45, 100), 104 (100), 71 (10), 55 (14). GC-MS (CI) *m/z* (%): 248 (MH^+, 100), 276 (14). HRMS (ESI-TOF) Calcd for $C_{14}H_{18}NO_3$: $[M+H]^+$ 248.1286. Found: 248.1287.

3-Ethoxy-1-(2-dimethylaminoethyl)pyrrolidine-2,5-dione (6f). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.25 (t, 3H, $J_{H7\text{-}H6}$ = 7.2, H–7), 2.25 (s, 6H, H–10, H–10'), 2.51 (t, 2H, $J_{H9\text{-}H8}$ = 6.4, H–9), 2.62 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.4, H–4), 3.02 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.0, $J_{H4'\text{-}H3}$ =

8.0, H–4'), 3.63 (t, 2H, J_{H8-H9} = 6.4, H–8), 3.65-3.72 (m, 1H, H–6), 3.95-4.03 (m, 1H, H–6'), 4.33 (dd, 1H, $J_{H3-H4'}$ = 8.4, J_{H3-H4} = 4.0, H–3). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.1 (C–7), 36.2 (C–4), 36.4 (C–8), 45.3 (C–10, C–10'), 56.0 (C–9), 66.8 (C–6), 73.2 (C–3), 174.3 (C–2), 176.0 (C–5). GC-MS (EI, 70eV) *m/z* (%): 214 (M^+, 4), 71 (6), 58 (100). HRMS (ESI-TOF) Calcd for $C_{10}H_{19}N_2O_3$: $[M+H]^+$ 215.1395. Found: 215.1393.

1-(2-Diethylaminoethyl)-3-ethoxypyrrolidine-2,5-dione (6g). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.01 (t, 6H, $J_{H11-H10}$ = 7.2, H–11, H–11'), 1.25 (t, 3H, J_{H7-H6} = 7.2, H–7), 2.54-2.67 (m, 7H, H–10, H–10', H–4, H–9), 3.02 (dd, 1H, $J_{H4'-H4}$ = 18.0, $J_{H4'-H3}$ = 8.0, H–4'), 3.61 (t, 2H, J_{H8-H9} = 6.8, H–8), 3.65-3.72 (m, 1H, H–6), 3.95-4.03 (m, 1H, H–6'), 4.32 (dd, 1H, $J_{H3-H4'}$ = 8.4, J_{H3-H4} = 4.0, H–3). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.9 (C–11, C–11'), 15.1 (C–7), 36.2 (C–9), 36.5 (C–4), 47.0 (C–10, C–10'), 49.2 (C–8), 66.8 (C–6), 73.2 (C–3), 174.2 (C–2), 175.9 (C–5). GC-MS (EI, 70eV) *m/z* (%): 242 (M^+, 3), 124 (5), 86 (100), 58 (13). HRMS (ESI-TOF) Calcd for $C_{12}H_{23}N_2O_3$: $[M+H]^+$ 243.1708. Found: 243.1700.

3-Ethoxy-1-(2-hydroxyethyl)pyrrolidine-2,5-dione (6h). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.26 (t, 3H, J_{H7-H6} = 7.0, H–7), 2.67 (dd, 1H, $J_{H4-H4'}$ = 18.2, J_{H4-H3} = 4.2, H–4), 3.06 (dd, 1H, $J_{H4'-H4}$ = 18.0, $J_{H4'-H3}$ = 8.2, H–4'), 3.62-3.78 (m, 5H, H–6, H–8, H–9), 3,90-4,07 (m, 1H, H–6'), 4.36 (dd, 1H, $J_{H3-H4'}$ = 8.2, J_{H3-H4} = 4.2, H–3). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 15.1 (C–7), 36.2 (C–4), 41.3 (C–8), 59.9 (C–9), 67.0 (C–6), 73.2 (C–3), 174.9 (C–2), 176.4 (C–5). GC-MS (EI, 70eV) *m/z* (%): 143 (MH^+-45, 100), 116 (10), 97 (27), 55 (43). HRMS (ESI-TOF) Calcd for $C_8H_{14}NO_4$: $[M+H]^+$ 188.0923. Found: 188.0926.

3-Ethoxy-1-(3-hydroxypropyl)pyrrolidine-2,5-dione (6i). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.25 (t, 3H, J_{H7-H6} = 6.8, H–7), 1.80 (qui, 2H, $J_{H9-H8,\ H10}$ = 6.4, H–9), 2.66 (dd, 1H, $J_{H4-H4'}$ = 18.4, J_{H4-H3} = 4.0, H–4), 3.03 (dd, 1H, $J_{H4'-H4}$ = 18.0, $J_{H4'-H3}$ = 8.0, H–4'), 3.58 (t, 2H, J_{H8-H9} = 6.0, H–8), 3.66-3.71 (m, 3H, H–6, H–10), 3.95-4.03 (m, 1H, H–6'), 4.15 (s, 1H, OH), 4.33 (dd, 1H, $J_{H3-H4'}$ = 8.4, J_{H3-H4} = 4.0, H–3). ^{13}C NMR (50 MHz, $CDCl_3$,): δ = 15.0 (C–7), 30.2 (C–9), 35.1 (C–4), 36.2 (C–8), 58.9 (C–10), 66.9 (C–6), 73.2 (C–3), 174.9 (C–2), 176.5 (C–5). GC-MS (EI, 70eV) *m/z* (%): 202 (MH^+, 1), 157 (MH^+-45, 67), 139 (100), 111 (62), 72 (52), 55 (56). HRMS (ESI-TOF) Calcd for $C_9H_{16}NO_4$: $[M+H]^+$ 202.1079. Found: 202.1078.

3-Ethoxy-1-(2-hydroxyprop-1-yl)pyrrolidine-2,5-dione (6j - *major compound*). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.20-1.29 (m, 6H, H–7, H–10), 2.67 (dd, 1H, $J_{H4-H4'}$ = 18.2, J_{H4-H3} = 4.4, H–4), 3.07 (dd, 1H, $J_{H4'-H4}$ = 18.2, $J_{H4'-H3}$ = 8.2, H–4'), 3.56 (d, J_{H8-H9} = 6.4, H–8), 3.62-3.77 (m, 1H, H–6), 3.91-4.07 (m, 2H, H–6', H–9), 4.37 (dd, 1H, $J_{H3-H4'}$ = 8.0, J_{H3-H4} = 4.2, H–3).^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.1 (C–7), 21.1 (C–10), 36.2 (C–4), 46.0 (C–8), 65.9 (C–6), 67.0 (C–9), 73.2 (C–3), 174.9 (C–2), 176.5 (C–5). **(6j - *minor compound*):** δ RMN ^{1}H (200 MHz, $CDCl_3$): 1.20-1.29 (m, 6H, H–7, H–10), 2.67 (dd, 1H, $J_{H4-H4'}$ = 18.2, J_{H4-H3} = 4.4, H–4), 3.06 (dd, 1H, $J_{H4'-H4}$ = 18.2, $J_{H4'-H3}$ = 8.0, H–4'), 3.56 (d, J_{H8-H9} = 6.4, H–8), 3.62-3.77 (m, 1H, H–6), 3.91-4.07 (m, 2H, H–6', H–9), 4.36 (dd, 1H, $J_{H3-H4'}$ = 8.2, J_{H3-H4} = 4.2, H–3). δ RMN ^{13}C (100 MHz, $CDCl_3$): 15.1 (C–7), 21.1 (C–10), 36.2 (C–4), 46.0 (C–8), 65.9 (C–6), 67.0 (C–9), 73.2 (C–3), 175.0 (C–2), 176.6 (C–5). GC-MS (EI, 70eV) *m/z* (%): 186 (M^+-15, 2), 157 (MH^+-

45, 100), 111 (96), 85 (23), 55 (81). HRMS (ESI-TOF) Calcd for $C_9H_{16}NO_4$: $[M+H]^+$ 202.1079. Found: 202.1084.

3-Ethoxy-1-((R)-1-hydroxybutan-2-yl)pyrrolidine-2,5-dione (6k - *major compound*). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.89 (t, 3H, $J_{H10\text{-}H9}$ = 7.4, H–10), 1.25 (t, 3H, $J_{H7\text{-}H6}$ = 7.0, H–7), 1.68-1.88 (m, 2H, H–9), 2.65 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.2, H–4), 3.04 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.0, $J_{H4'\text{-}H3}$ = 8.2, H–4'), 3.61-3.79 (m, 2H, H–6, H–8), 3.90-4.20 (m, 3H, H–6', H–11), 4.28-4.37 (m, 1H, H–3). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 10.6 (C–10), 15.1 (C–7), 20.8 (C–9), 36.1 (C–4), 56.1 (C–11), 61.9 (C–8), 66.9 (C–6), 72.9 (C–3), 175.1 (C–2), 176.8 (C–5). **(6k - *minor compound*):** δ RMN ^{1}H (200 MHz, $CDCl_3$): 0.89 (t, 3H, $J_{H10\text{-}H9}$ = 7.4, H–10), 1.25 (t, 3H, $J_{H7\text{-}H6}$ = 7.0, H–7), 1.68-1.88 (m, 2H, H–9), 2.65 (dd, 1H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.2, H–4), 3.04 (dd, 1H, $J_{H4'\text{-}H4}$ = 18.0, $J_{H4'\text{-}H3}$ = 8.2, H–4'), 3.61-3.79 (m, 2H, H–6, H–8), 3.90-4.20 (m, 3H, H–6', H–11), 4.28-4.37 (m, 1H, H–3). δ RMN ^{13}C (100 MHz, $CDCl_3$): 10.6 (C–10), 15.1 (C–7), 20.9 (C–9), 36.2 (C–4), 56.1 (C–11), 62.1 (C–8), 66.9 (C–6), 73.1 (C–3), 175.4 (C–2), 177.0 (C–5). GC-MS (EI, 70eV) *m/z* (%): 216 (MH^+, 1), 171 (MH^+-45, 100), 153 (30), 116 (27), 98 (24), 72 (39), 55 (52). HRMS (ESI-TOF) Calcd for $C_{10}H_{18}NO_4$: $[M+H]^+$ 216.1236. Found: 216.1238.

5-(Pyridin-2-ylmethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7a). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.26-2.37 (m, 2H, H–3, H–3'), 3.47 (ddd, 1H, $J_{H2\text{-}H2'}$ = 9.2, $J_{H2\text{-}H3}$ = 7.0, $J_{H2\text{-}H3'}$ = 2.8, H–2), 3.79 (m, 1H, H–3a), 4.10 (ddd, 1H, $J_{H2'\text{-}H2}$ = 9.0, $J_{H2'\text{-}H3}$ = 7.2, $J_{H2'\text{-}H3'}$ = 2.8, H–2'), 4.83 (s, 2H, H–7), 4.87 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a), 7.14-7.22 (m, 2H, H–10, H–12), 7.64 (td, 1H, $J_{H11\text{-}H10,\,H12}$ = 7.6, $J_{H11\text{-}H9}$ = 1.8, H–11), 8.46 (d, 1H, $J_{H9\text{-}H10}$ = 4.0, H–9). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 30.2 (C–3), 43.3 (C–3a), 44.9 (C–7), 68.0 (C–2), 77.7 (C–6a), 121.7 (C–10), 122.6 (C–12), 136.7 (C–11), 149.4 (C–9), 153.5 (C–8), 174.9 (C–6), 177.0 (C–4). GC-MS (EI, 70eV) *m/z* (%): 233 (MH^+, 36), 204 (57), 189 (100), 134 (41), 92 (51), 70 (88). HRMS (ESI-TOF) Calcd for $C_{12}H_{13}N_2O_3$: $[M+H]^+$ 233.0926. Found: 233.0920.

5-(Pyridin-3-ylmethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7b): Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.21-2.32 (m, 2H, H–3, H–3'), 3.37-3.57 (m, 2H, H–3a, H–2), 4.05 (dt, 1H, $J_{H2'\text{-}H2}$ = 10.2, $J_{H2'\text{-}H3,\,H3'}$ = 4.6, H–2'), 4.68 (s, 2H, H–7), 4.78 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a), 7.27 (dd, 1H, $J_{H11\text{-}H12}$ = 7.6, $J_{H11\text{-}H10}$ = 5.0, H–11), 7.72 (dt, 1H, $J_{H12\text{-}H11}$ = 7.6, $J_{H12\text{-}H9,\,H10}$ = 2.2, H–12), 8.55 (d, 1H, $J_{H10\text{-}H11}$ = 3.6, H–10), 8.64 (d, 1H, $J_{H9\text{-}H12}$ = 1.2, H–9). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.9 (C–3), 40.0 (C–3a), 44.9 (C–7), 68.2 (C–2), 77.7 (C–6a), 123.6 (C–11), 131.1 (C–8), 136.5 (C–12), 149.5 (C–10), 150.0 (C–9), 174.2 (C–6), 176.5 (C–4). GC-MS (EI, 70eV) *m/z* (%): 232 (M^+, 97), 204 (20), 187 (20), 135 (47), 92 (51), 70 (100). HRMS (ESI-TOF) Calcd for $C_{12}H_{13}N_2O_3$: $[M+H]^+$ 233.0926. Found: 233.0919.

5-(Pyridin-4-ylmethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7c). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.22-2.33 (m, 2H, H–3, H–3'), 3.34-3.64 (m, 2H, H–3a, H–2), 4.01-4.14 (m, 1H, H–2'), 4.65 (s, 2H, H–7), 4.80 (d, 1H, $J_{H6a\text{-}H3a}$ = 7,2, H–6a), 7.25 (d, 2H, $J_{H9\text{-}H10}$ = 6.2, H–9), 8.56 (d, 2H, $J_{H10\text{-}H9}$ = 5.8, H–10). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.9 (C–3), 41.3 (C–3a), 44.9 (C–7), 68.3 (C–2), 77.7 (C–6a), 123.2 (C–9), 144.2 (C–8), 149.8 (C–10), 174.2 (C–6), 176.5 (C–4). GC-MS (EI, 70eV) *m/z* (%): 232 (M^+, 100), 204 (41), 135 (23), 92 (14), 70 (100). HRMS (ESI-TOF) Calcd for $C_{12}H_{13}N_2O_3$: $[M+H]^+$ 233.0926. Found: 233.0923.

5-(1-Phenylmethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7d). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.17-2.28 (m, 2H, H–3, H–3'), 3.31-3.55 (m, 2H, H–3a, H–2), 3.97-4.07 (m, 1H, H–2'), 4.65 (s, 2H, H–7), 4.74 (d, 1H, $J_{H6a-H3a}$ = 7,2, H–6a), 7.27-7.33 (m, 5H, H–9, H–10, H–11). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 29.9 (C–3), 42.4 (C–3a), 44.7 (C–7), 68.0 (C–2), 77.6 (C–6a), 128.0 (C–11), 128.6 (C–9), 128.6 (C–10), 135.4 (C–8), 174.5 (C–6), 176.7 (C–4). GC-MS (EI, 70eV) *m/z* (%): 231 (M^+, 34), 203 (37), 186 (21), 91 (100), 70 (58). HRMS (ESI-TOF) Calcd for $C_{13}H_{14}NO_3$: $[M+H]^+$ 232.0973. Found: 232.0965.

5-(2-Phenylethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7e). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.08-2.18 (m, 2H, H–3, H–3'), 2.93 (t, 2H, J_{H8-H7} = 7,2, H–8), 3.23-3.35 (m, 2H, H–3a, H–2), 3.80 (t, 2H, J_{H7-H8} = 6.6, H–7), 3.96 (ddd, 1H, $J_{H2-H2'}$ = 9.0, J_{H2-H3} = 6.8, $J_{H2-H3'}$ = 3.0, H–2'), 4.68 (d, 1H, $J_{H6a-H3a}$ = 7.2, H–6a), 7.19-7.32 (m, 5H, H–10, H–11, H–12). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 29.9 (C–3), 33.1 (C–8), 39.7 (C–3a), 44.6 (C–7), 67.7 (C–2), 77.5 (C–6a), 126.8 (C–12), 128.5 (C–10), 128.8 (C–11), 137.2 (C–9), 174.7 (C–6), 176.8 (C–4). GC-MS (EI, 70eV) *m/z* (%): 245 (M^+, 8), 104 (100), 91 (21), 91 (31), 69 (17). HRMS (ESI-TOF) Calcd for $C_{14}H_{16}NO_3$: $[M+H]^+$ 246.1130. Found: 246.1120.

5-(2-Dimethylaminoethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7f). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.23 (s, 6H, H–9, H–9'), 2.26-2.28 (m, 2H, H–3, H–3'), 2.51 (t, 2H, J_{H8-H7} = 6.4, H–8), 3.36-3.41 (m, 2H, H–3a, H–2), 3.64 (t, 2H, J_{H7-H8} = 6.4, H–7), 4.04-4.08 (m, 1H, H–2'), 4.77 (d, 1H, $J_{H6a-H3a}$ = 7.2, H–6a). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 30.1 (C–3), 36.7 (C–3a), 44.7 (C–7), 45.3 (C–9, C–9'), 55.7 (C–8), 67.7 (C–2), 77.6 (C–6a), 175.0 (C–6), 177.2 (C–4). GC-MS (EI, 70eV) *m/z* (%): 212 (M^+, 2), 70 (4), 58 (100). HRMS (ESI-TOF) Calcd for $C_{10}H_{17}N_2O_3$: $[M+H]^+$ 213.1239. Found: 213.1236.

5-(2-Diethylaminoethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7g). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.96 (t, 6H, J_{H10-H9} = 7.2, H–10, H–10'), 2.19-2.31 (m, 2H, H–3, H–3'), 2.51 (q, 4H, J_{H9-H10} = 7.2, H–9, H–9'), 2.61 (t, 2H, J_{H8-H7} = 6.6, H–8), 3.31-3.41 (m, 1H, H–3a), 3.58-3.70 (m, 3H, H–7, H–2), 4.01-4.11 (m, 1H, H–2'), 4.76 (d, 1H, $J_{H6a-H3a}$ = 7.2, H–6a). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 11.7 (C–10, C–10'), 30.0 (C–3), 36.7 (C–3a), 44.6 (C–8), 46.6 (C–9, C–9'), 49.1 (C–7), 67.7 (C–2), 77.6 (C–6a), 174.9 (C–6), 177.1 (C–4). GC-MS (EI, 70eV) *m/z* (%): 241 (MH^+, 7), 86 (100), 70 (12), 58 (15). HRMS (ESI-TOF) Calcd for $C_{12}H_{21}N_2O_3$: $[M+H]^+$ 241.1552. Found: 241.1556.

5-(2-Hydroxyethyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7h). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.22-2.33 (m, 2H, H–3, H–3'), 2.83 (s, 1H, OH), 3.40-3.49 (m,1H, H–3a), 3.55-3.81 (m, 5H, H–7, H–8, H–2), 4.03-4.12 (m, 1H, H–2'), 4.81 (d, 1H, $J_{H6a-H3a}$ = 7.2, H–6a). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 30.0 (C–3), 41.4 (C–3a), 44.8 (C–7), 59.5 (C–8), 68.0 (C–2), 77.7 (C–6a), 175.7 (C–6), 177.8 (C–4). GC-MS (EI, 70eV) *m/z* (%): 186 (MH^+, 10), 142 (100), 70 (57), 56 (29). HRMS (ESI-TOF) Calcd for $C_8H_{12}NO_4$: $[M+H]^+$ 186.0766. Found: 186.0759.

5-(3-Hydroxypropyl)-2,3,3a,6a-tetrahydrofuro[2,3-*c*]pyrrole-4,6(5*H*)-dione (7i). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.80 (qui, 2H, $J_{H8-H7,H9}$ = 6.0, H–8), 2.25-2.31 (m, 2H, H–3, H–3'), 3.38-3.46 (m, 1H, H–3a), 3.57 (t, 2H, J_{H9-H8} = 6.0, H–9), 3.59-3.65 (m, 1H, H–2), 3.68 (t,

2H, $J_{H7\text{-}H8}$ = 6.4, H–7), 4.05-4.10 (m, 1H, H–2'), 4.78 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.9 (C–3), 30.3 (C–8), 35.9 (C–3a), 44.8 (C–7), 59.3 (C–9), 68.1 (C–2), 77.7 (C–6a), 175.4 (C–6), 177.6 (C–4). GC-MS (EI, 70eV) *m/z* (%): 199 (M^+, 2), 181 (14), 155 (43), 70 (100). HRMS (ESI-TOF) Calcd for $C_9H_{14}NO_4$: $[M+H]^+$ 200.0923. Found: 200.0924.

5-(2-Hydroxyprop-1-yl)-2,3,3a,6a-tetrahydrofuro [2,3-*c*]pyrrole-4,6(5*H*)-dione (7j - major compound). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.22 (d, 3H, $J_{H9\text{-}H8}$ = 6.2, H–9), 2.22-2.34 (m, 2H, H–3, H–3'), 3.38-3.48 (m,1H, H–3a), 3.56-3.69 (m, 3H, H–7, H–8), 3.98-4.14 (m, 2H, H–2, H–2'), 4.81 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.2 (C–9), 30.1 (C–3), 44.7 (C–3a), 46.1 (C–7), 65.7 (C–8), 68.0 (C–2), 77.6 (C–6a), 175.5 (C–6), 177.7 (C–4). **(7j - minor compound).** δ RMN ^{1}H (200 MHz, $CDCl_3$)1.22 (d, 3H, $J_{H9\text{-}H8}$ = 6.2, H–9), 2.22-2.34 (m, 2H, H–3, H–3'), 3.38-3.48 (m,1H, H–3a), 3.56-3.69 (m, 3H, H–7, H–8), 3.98-4.14 (m, 2H, H–2, H–2'), 4.82 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a). δ RMN ^{13}C (100 MHz, $CDCl_3$): 21.2 (C–9), 30.1 (C–3), 44.8 (C–3a), 46.1 (C–7), 65.8 (C–8), 68.0 (C–2), 77.7 (C–6a), 175.5 (C–6), 177.8 (C–4). GC-MS (EI, 70eV) *m/z* (%): 198 (M^--H, 1), 155 (100), 142 (49), 127 (14), 99 (44), 69 (28). HRMS (ESI-TOF) Calcd for $C_9H_{14}NO_4$: $[M+H]^+$ 200.0923. Found: 200.0926.

5-[(R)-1-Hydroxybutan-2-yl]-2,3,3a,6a-tetrahydro-furo[2,3-*c*]pyrrole-4,6(5*H*)-dione (7k - major compound). Yellow oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 0.87 (t, 3H, $J_{H9\text{-}H8}$ = 7,2, H–9), 1.67-1.77 (m, 1H, H–8), 1.79-1.92 (m, 1H, H–8'), 2.24-2.31 (m, 2H, H–3, H–3'), 2.93 (s, 1H, OH), 3.37-3.42 (m, 1H, H–3a), 3.59-3.65 (m, 1H, H–10), 3.74 (dd, 1H, $J_{H10'\text{-}H10}$ = 12.0, $J_{H10'\text{-}H7}$ = 3.6, H–10'), 3.96-4.02 (m, 1H, H–7), 4.05-4.17 (m, 2H, H–2, H–2'), 4.75 (d, 1H, $J_{H6a\text{-}H3a}$ = 7.2, H–6a). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 10.6 (C–9), 20.7 (C–8), 30.2 (C–3), 44.4 (C–3a), 56.4 (C–7), 61.6 (C–10), 67.8 (C–2), 77.2 (C–6a), 176.0 (C–6), 178.2 (C–4). **(7k - minor compound).** δ RMN ^{1}H (200 MHz, $CDCl_3$): 0.87 (t, 3H, $J_{H9\text{-}H8}$ = 7.2, H–9), 1.67-1.77 (m, 1H, H–8), 1.79-1.92 (m, 1H, H–8'), 2.24-2.31 (m, 2H, H–3, H–3'), 2.93 (s, 1H, OH), 3.37-3.42 (m, 1H, H–3a), 3.59-3.65 (m, 1H, H–10), 3.74 (dd, 1H, $J_{H10'\text{-}H10}$ = 12.0, $J_{H10'\text{-}H7}$ = 3.6, H–10'), 3.96-4.02 (m, 1H, H–7), 4.05-4.17 (m, 2H, H–2, H–2'), 4.77 (d, 1H, $J_{H6a\text{-}H3a}$ = 8.0, H–6a). δ RMN ^{13}C (100 MHz, $CDCl_3$): 10.6 (C–9), 20.7 (C–8), 30.2 (C–3), 44.6 (C–3a), 56.4 (C–7), 61.7 (C–10), 67.8 (C–2), 77.3 (C–6a), 176.0 (C–6), 178.2 (C–4). GC-MS (EI, 70eV) *m/z* (%): 213 (M^+, 1), 182 (21), 142 (100), 84 (50), 70 (34). HRMS (ESI-TOF) Calcd for $C_{10}H_{16}NO_4$: $[M+H]^+$ 214.1079. Found: 214.1074.

Synthesis of *bis*-3-ethoxypyrrolidine-2,5-diones 11-13, General procedure

Compounds **11-13** were obtained according to the procedure described in this paper to obtain the compounds **6** and **7**, but in these cases, 3-cyano-3-ethoxypropionic acid **3** (5.0 mmol) was dissolved in water (10 mL) and reacted with the diamines 1,2-diaminoethane (**8**), 1,3-diaminopropane (**9**) or 1,4 diaminobutane (**10**) (2.5 mmol). The *bis*-3-ethoxypyrrolidine-2,5-diones **11-13** were obtained as brown oils in moderate yields. Compounds **11-13** were considered pure and were not submitted to further purification.

Spectrometric dada of compounds 11-13 (^{1}H NMR, ^{13}C NMR, GC-MS, and HRMS)

1,1'-(Ethane-1,2-diyl)bis(3-ethoxypyrrolidine-2,5-dione) (11). Brown oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.24 (t, 6H, $J_{H7\text{-}H6}$ = 7.0, H–7), 2.59 (dd, 2H, $J_{H4\text{-}H4'}$ = 18.0, $J_{H4\text{-}H3}$ = 4.4, H–4), 2.93 (dd, 1H, $J_{H4'\text{-}H4}$ = 8.2, $J_{H4'\text{-}H3}$ = 3.8, H–4'), 3.02 (dd, 1H, $J_{H4'\text{-}H4}$ = 8.0, $J_{H4'\text{-}H3}$ = 3.8, H–4'), 3.59-3.77 (m, 6H, H–6, H–8), 3.82-4.01 (m, 2H, H–6'), 4.24-4.32 (m, 2H, H–3). ^{13}C NMR (50 MHz, $CDCl_3$): δ = 15.1 (C–7), 36.0 (C–4), 36.7 (C–8), 66.8 (C–6), 73.2 (C–3), 174.4 (C–2), 174.5 (C–2), 176.1 (C–5), 176.2 (C–5). GC-MS (EI, 70eV) *m/z* (%): 312 (M^+, 1), 268 (MH^+-45, 51), 224 (100), 187 (30), 125 (39), 72 (34), 55 (38). HRMS (ESI-TOF) Calcd for $C_{14}H_{20}N_2O_6Na$: $[M+H]^+$ 335.1219. Found: 335.1216.

1,1'-(Propane-1,3-diyl)bis(3-ethoxypyrrolidine-2,5-dione) (12). Brown oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.25 (t, 6H, $J_{H7\text{-}H6}$ = 7.0, H–7), 1.95 (qui, 2H, $J_{H9\text{-}H8}$ = 7.0, H–9), 2.62 (dd, 2H, $J_{H4\text{-}H4'}$ = 18.2, $J_{H4\text{-}H3}$ = 4.2, H–4), 3.03 (dd, 2H, $J_{H4'\text{-}H4}$ = 18.2, $J_{H4'\text{-}H3}$ = 8.0, H–4'), 3.50 (t, 4H, $J_{H8\text{-}H9}$ = 7.0, H–8), 3.60-3.75 (m, 2H, H–6), 3.90-4.05 (m, 2H, H–6'), 4.33 (dd, 2H, $J_{H3\text{-}H4'}$ = 8.2, $J_{H3\text{-}H4}$ = 4.0, H–3). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.1 (C–7), 25.3 (C–9), 28.2 (C–9), 35.8 (C–8), 36.2 (C–4), 66.9 (C–6), 73.2 (C–3), 174.2 (C–2), 174.3 (C–2), 175.9 (C–5). GC-MS (EI, 70eV) *m/z* (%): 326 (M^+, 1), 282 (MH^+-45, 100), 238 (74), 184 (29), 139 (43), 72 (56), 55 (53). HRMS (ESI-TOF) Calcd for $C_{15}H_{22}N_2O_6Na$: $[M+H]^+$ 349.1376. Found: 349.1374.

1,1'-(Butane-1,4-diyl)bis(3-ethoxypyrrolidine-2,5-dione) (13). Brown oil; ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.25 (t, 6H, $J_{H7\text{-}H6}$ = 7.0, H–7), 1.57 (m, 4H, H–9), 2.62 (dd, 2H, $J_{H4\text{-}H4'}$ = 18.2, $J_{H4\text{-}H3}$ = 4.2, H–4), 3.01 (dd, 2H, $J_{H4'\text{-}H4}$ = 18.2, $J_{H4'\text{-}H3}$ = 8.0, H–4'), 3.51 (m, 4H, H–8), 3.60-3.75 (m, 2H, H–6), 3.91-4.05 (m, 2H, H–6'), 4.3 (dd, 2H, $J_{H3\text{-}H4'}$ = 8.0, $J_{H3\text{-}H4}$ = 4.2, H–3). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 15.1 (C–7), 24.7 (C–9), 36.2 (C–4), 37.9 (C–8), 66.9 (C–6), 73.2 (C–3), 174.2 (C–2), 175.9 (C–5). GC-MS (EI, 70eV) *m/z* (%): 340 (M^+, 1), 296 (MH^+-45, 77), 252 (20), 72 (62), 55 (100). HRMS (ESI-TOF) Calcd for $C_{16}H_{24}N_2O_6Na$: $[M+H]^+$ 363.1532. Found: 363.1527.

NMR spectra of compounds 6a-k:

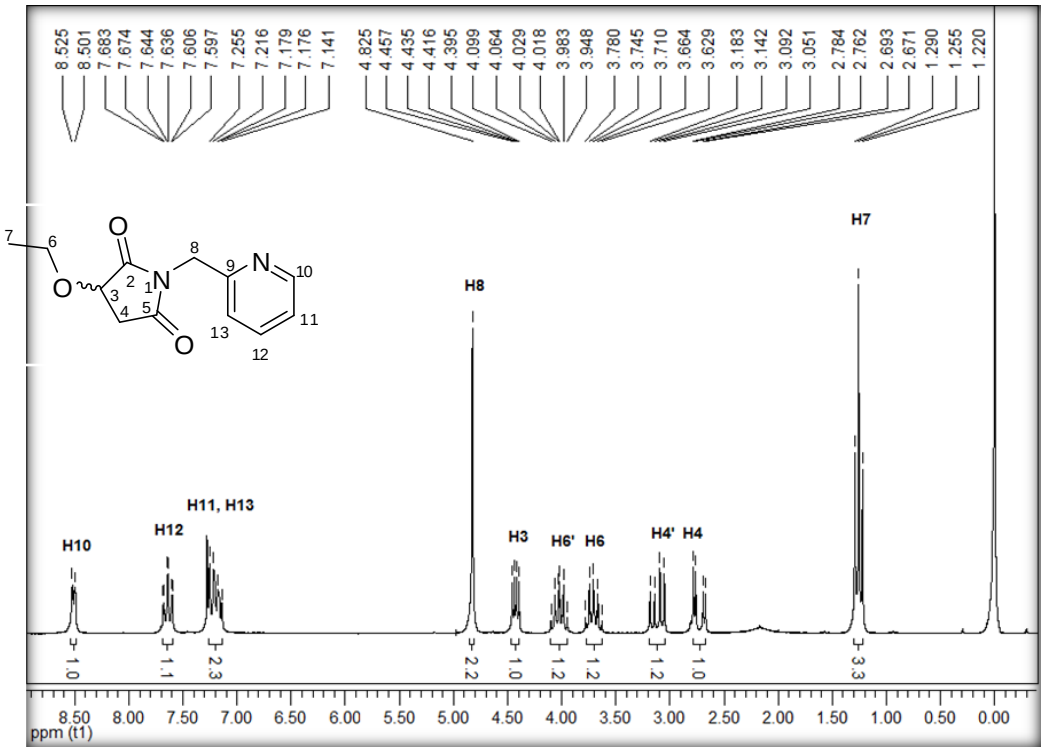

Figure S1. ^{1}H NMR (400MHz, $CDCl_3$) spectrum of compound **6a**.

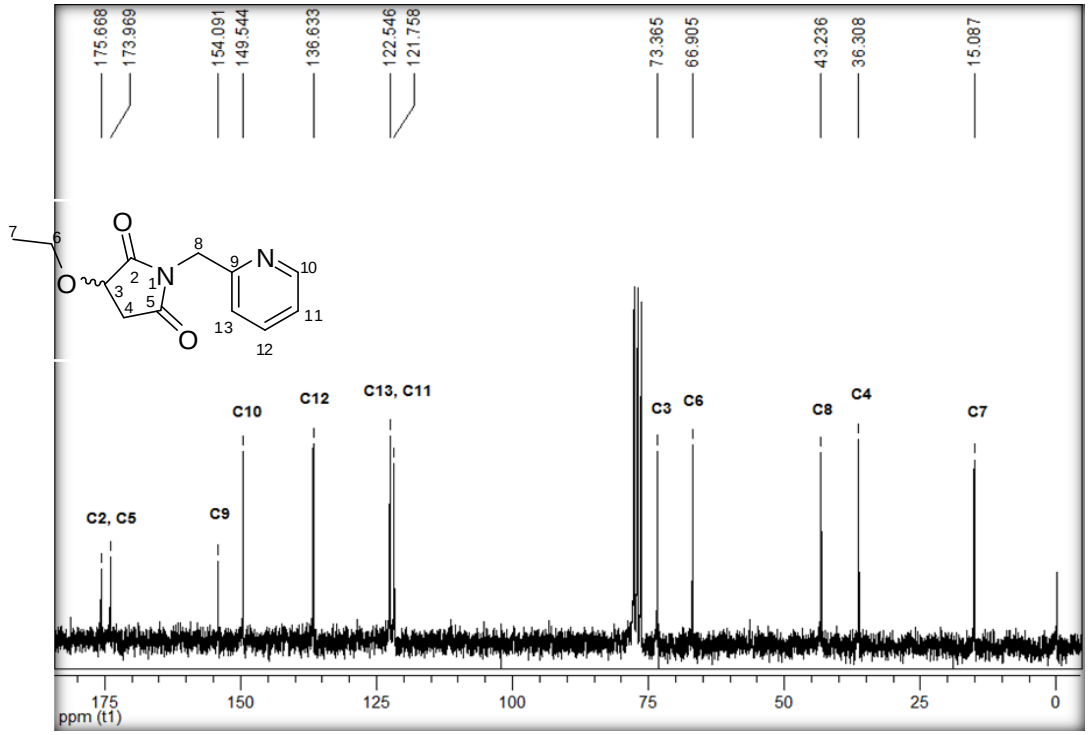

Figure S2. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6a**.

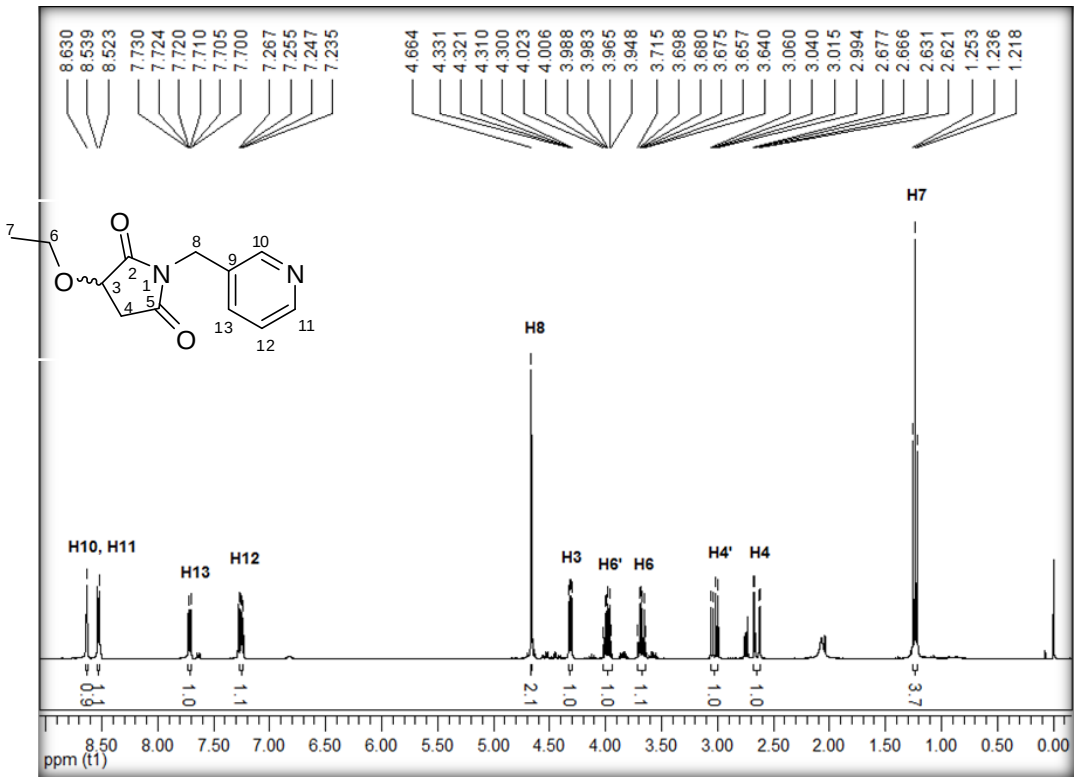

Figure S3. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6b**.

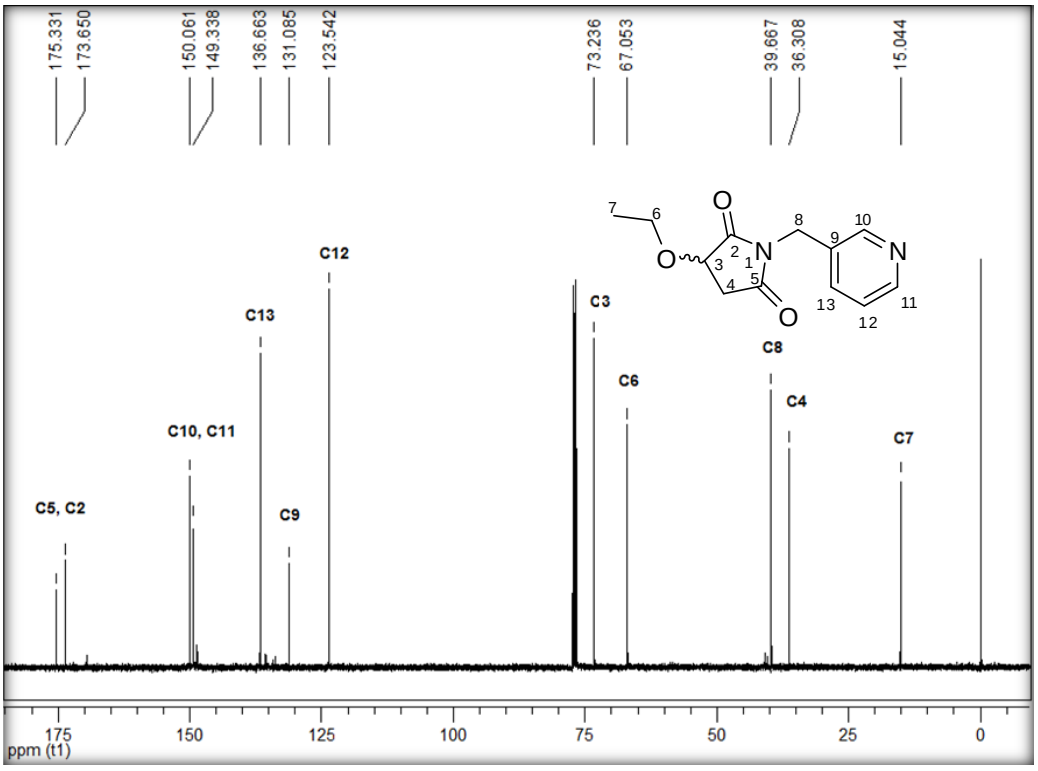

Figure S4. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **6b**.

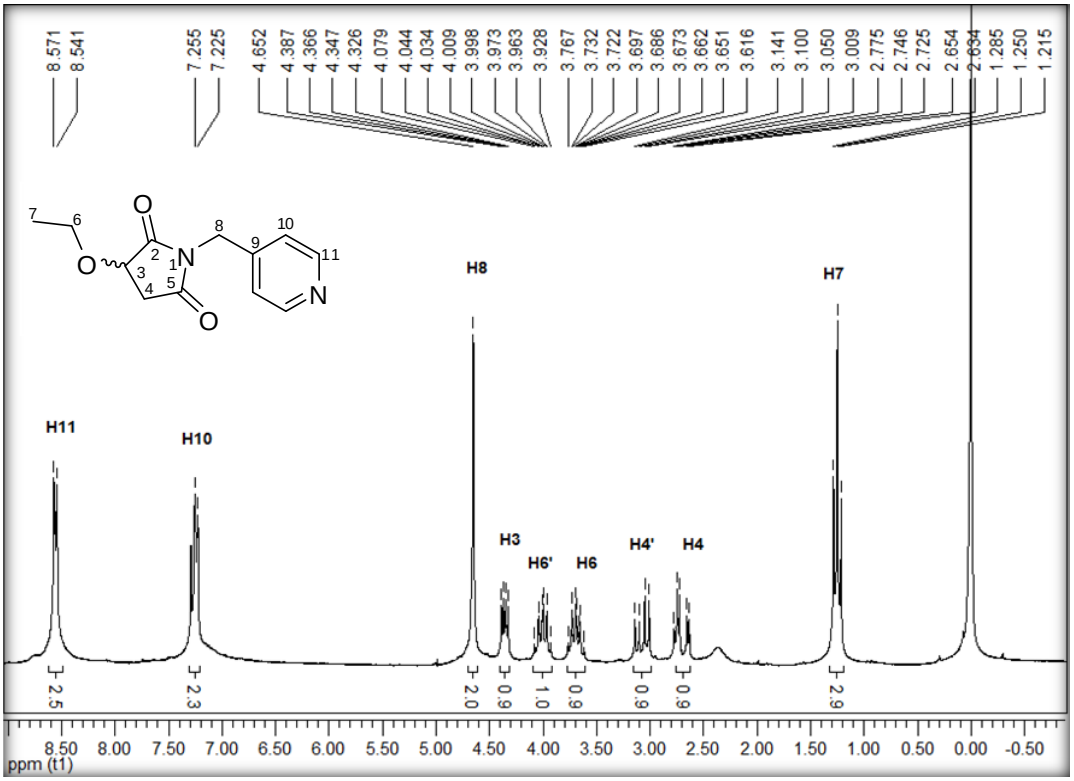

Figure S5. ^{1}H NMR (200 MHz, $CDCl_3$) spectra of compound **6c**.

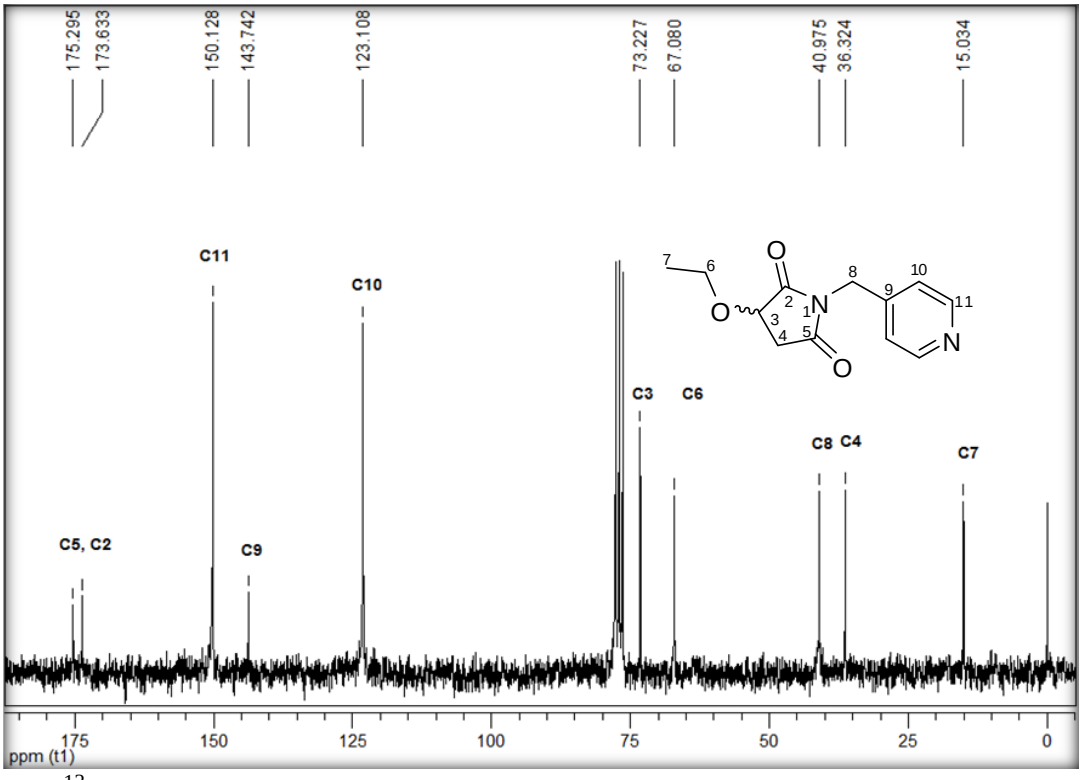

Figure S6. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **6c**.

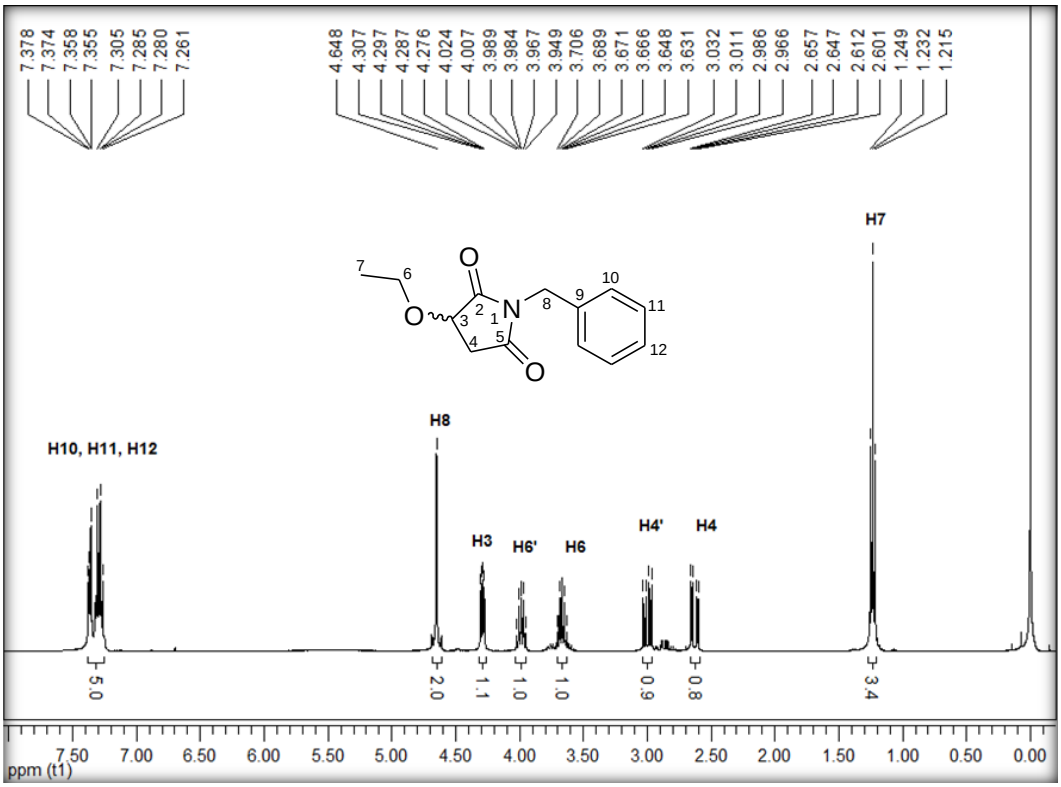

Figure S7. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6d**.

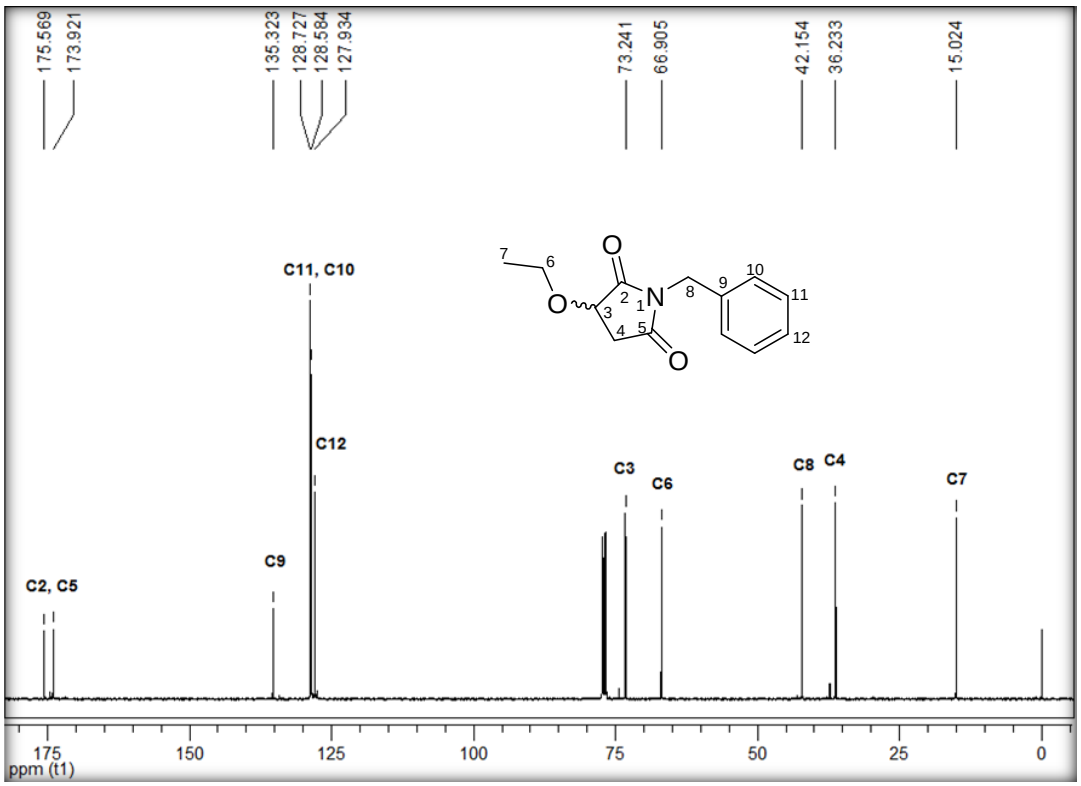

Figure S8. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6d**.

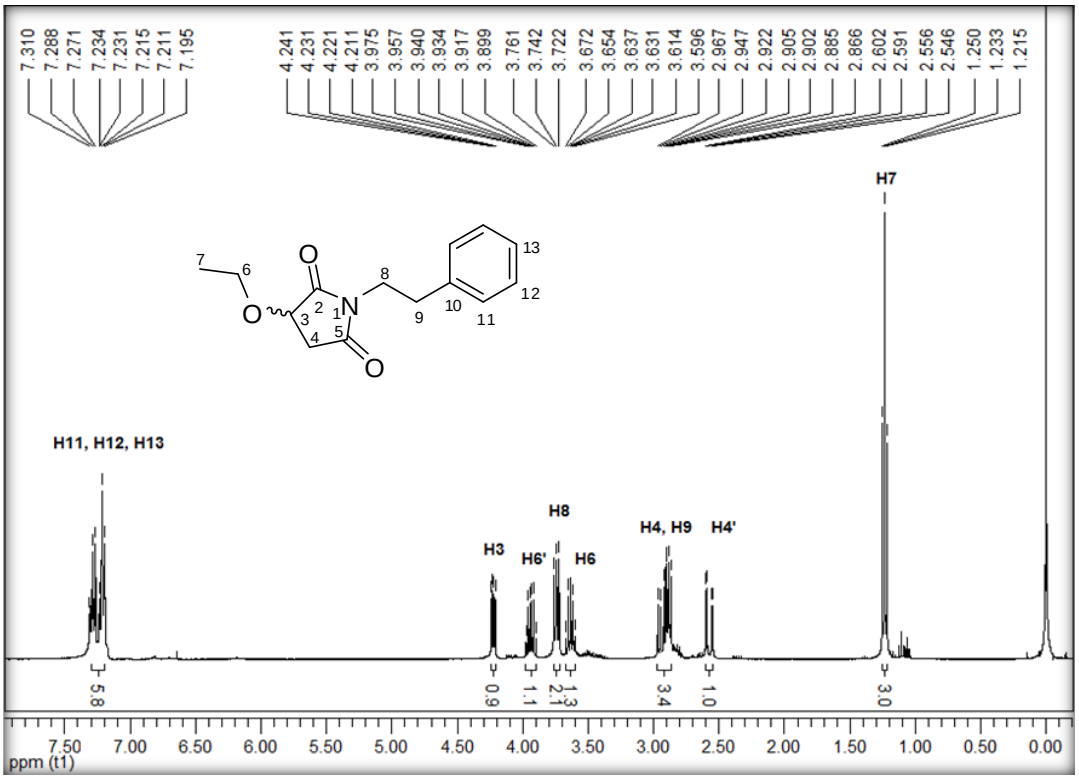

Figure S9. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **6e**.

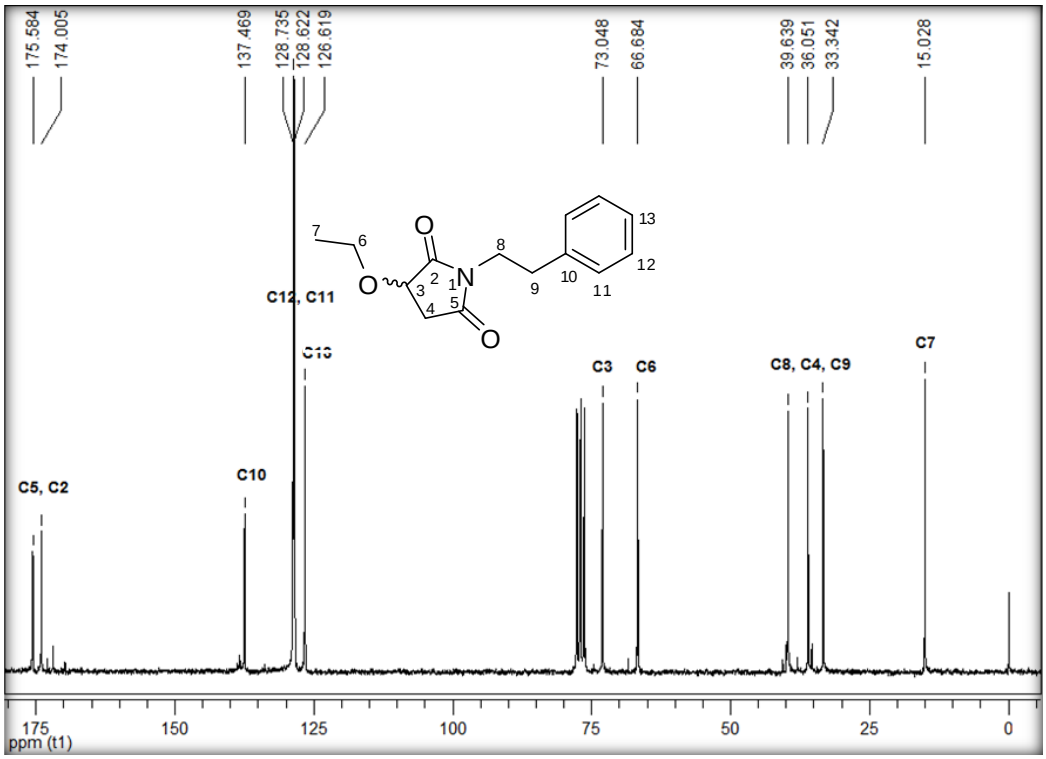

Figure S10. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6e**.

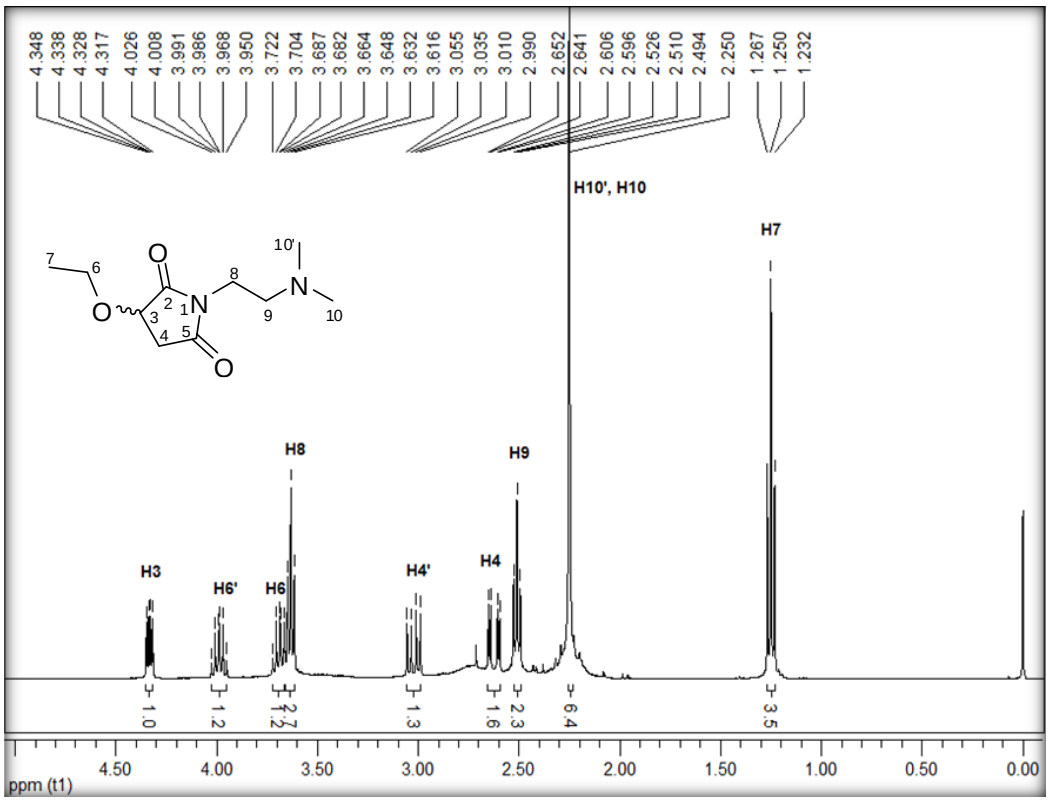

Figure S11. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound **6f**.

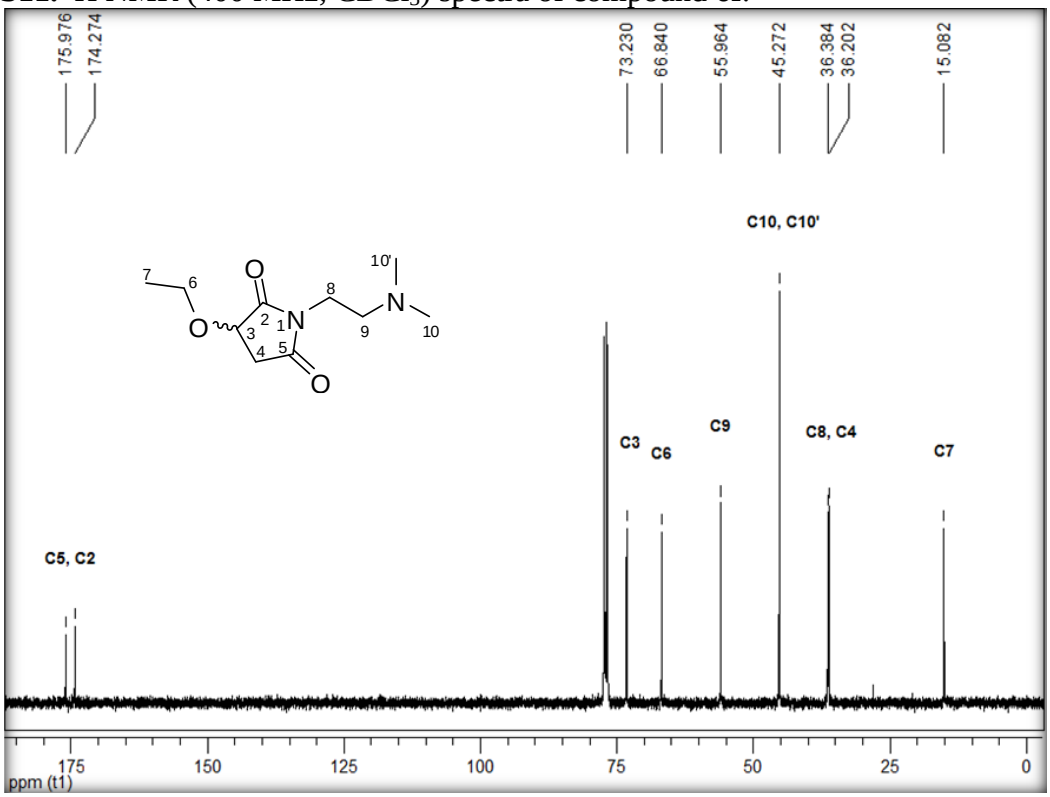

Figure S12. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6f**.

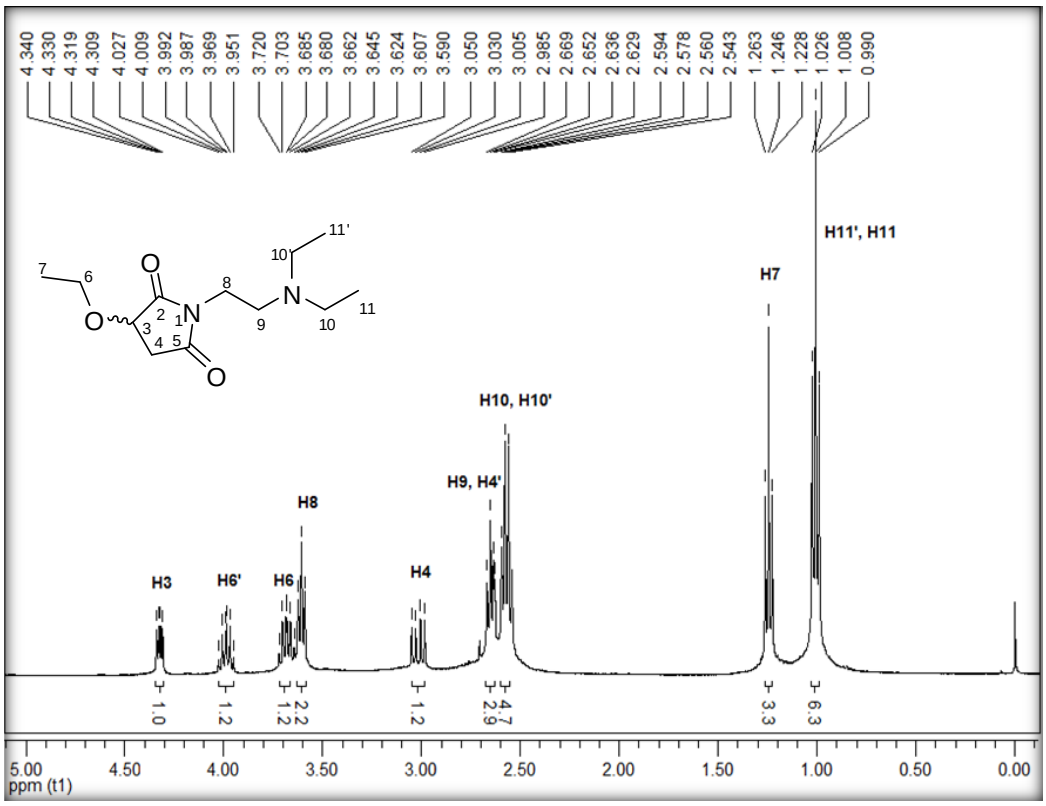

Figure S13. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6g**.

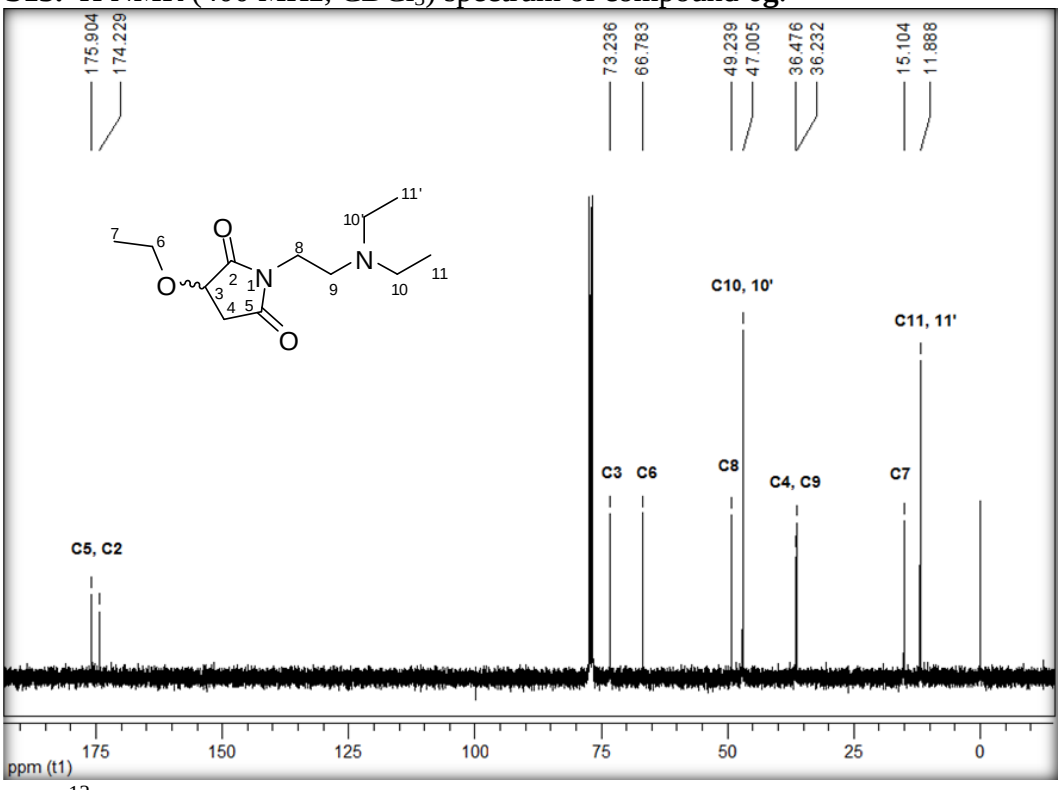

Figure S14. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6g**.

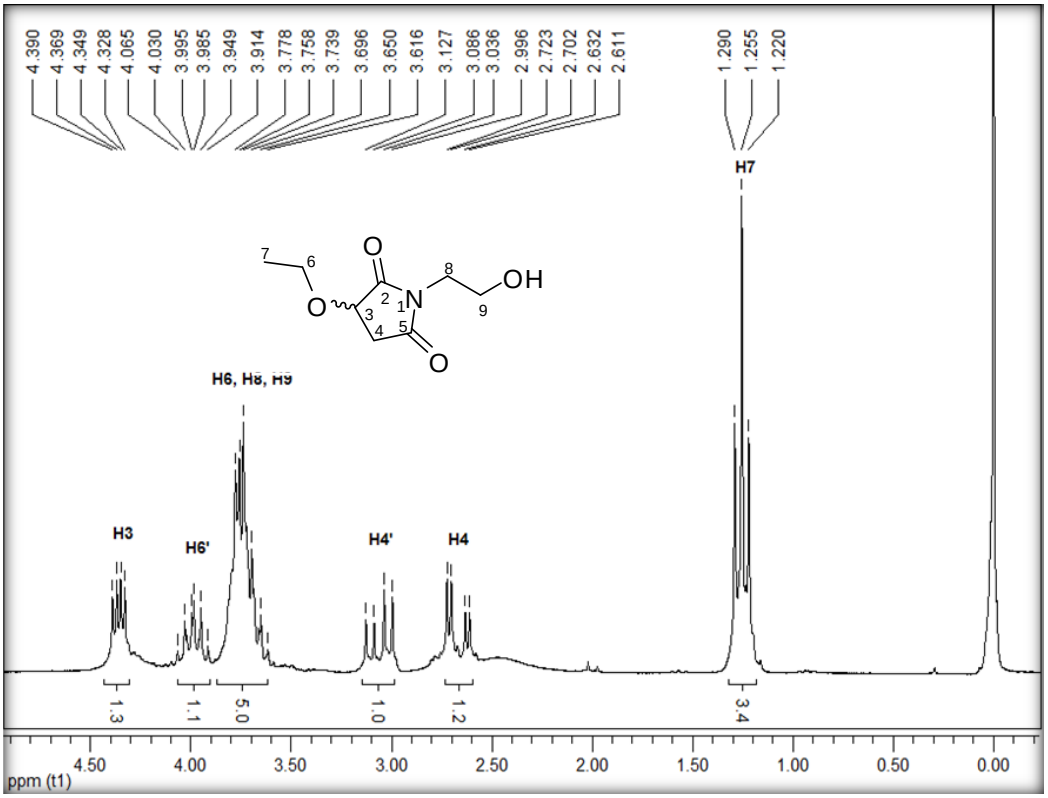

Figure S15. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **6h**.

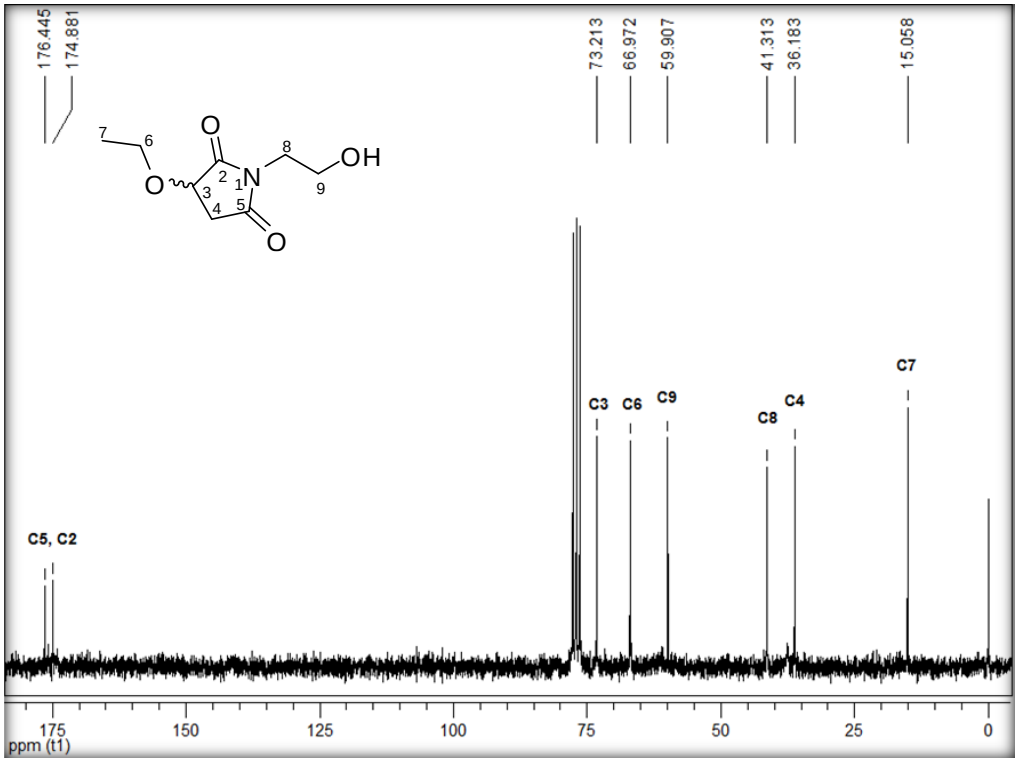

Figure S16. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **6h**.

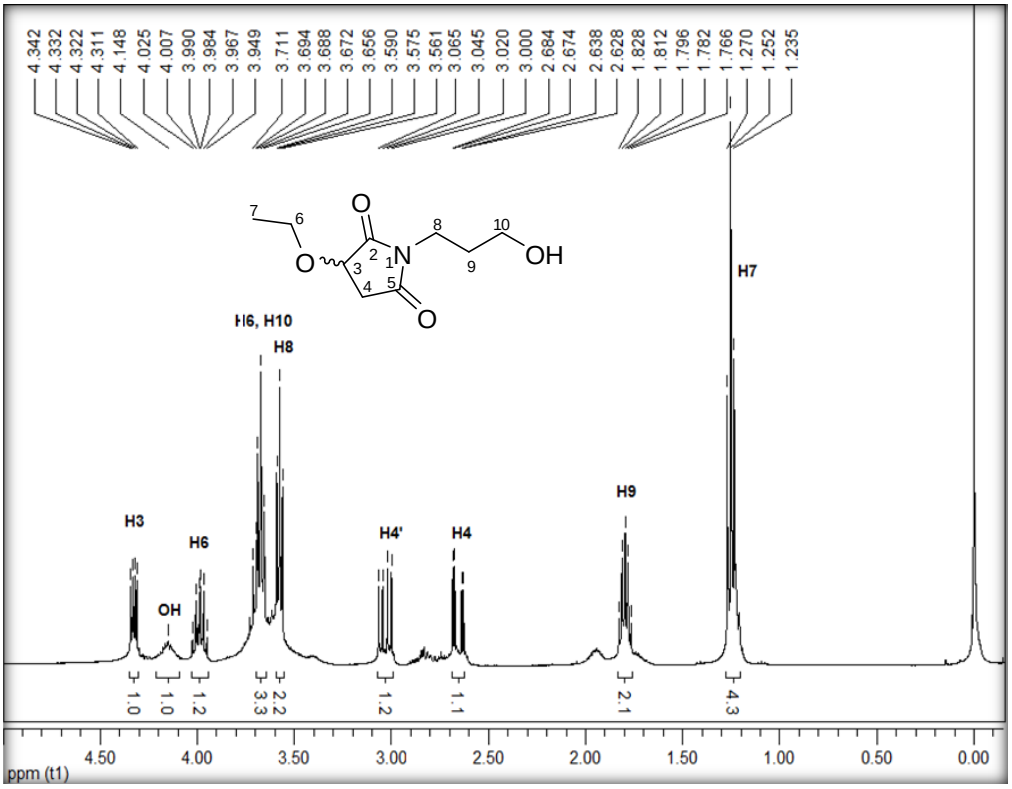

Figure S17. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6i**.

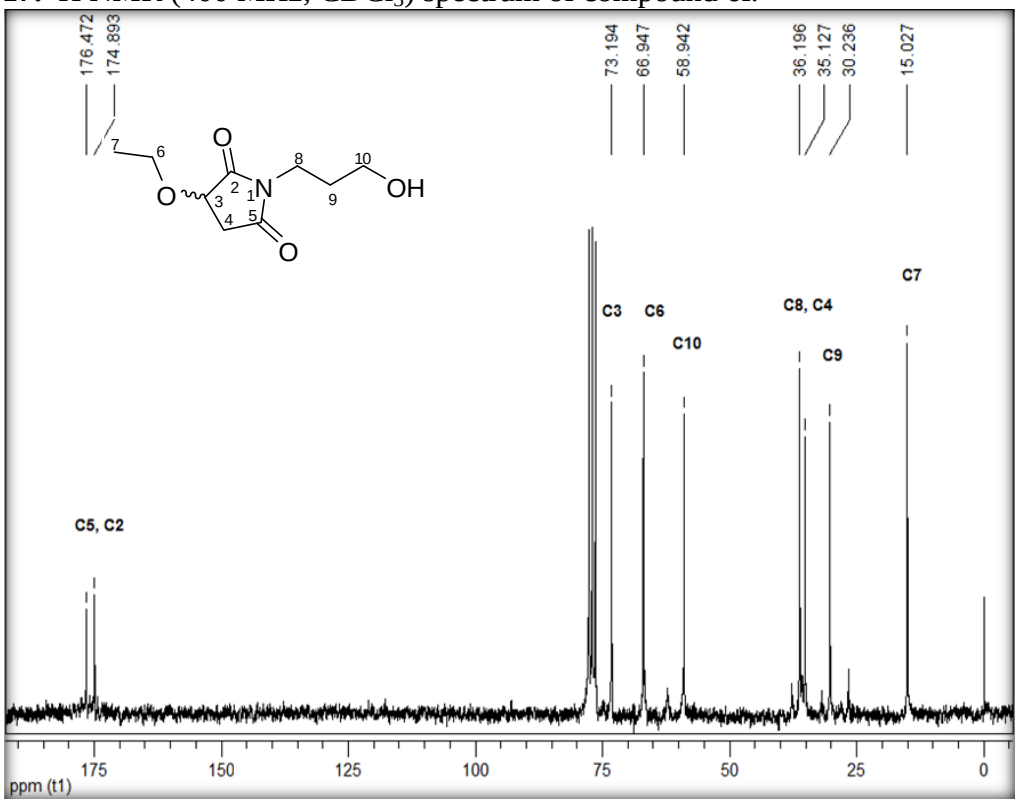

Figure S18. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **6i**.

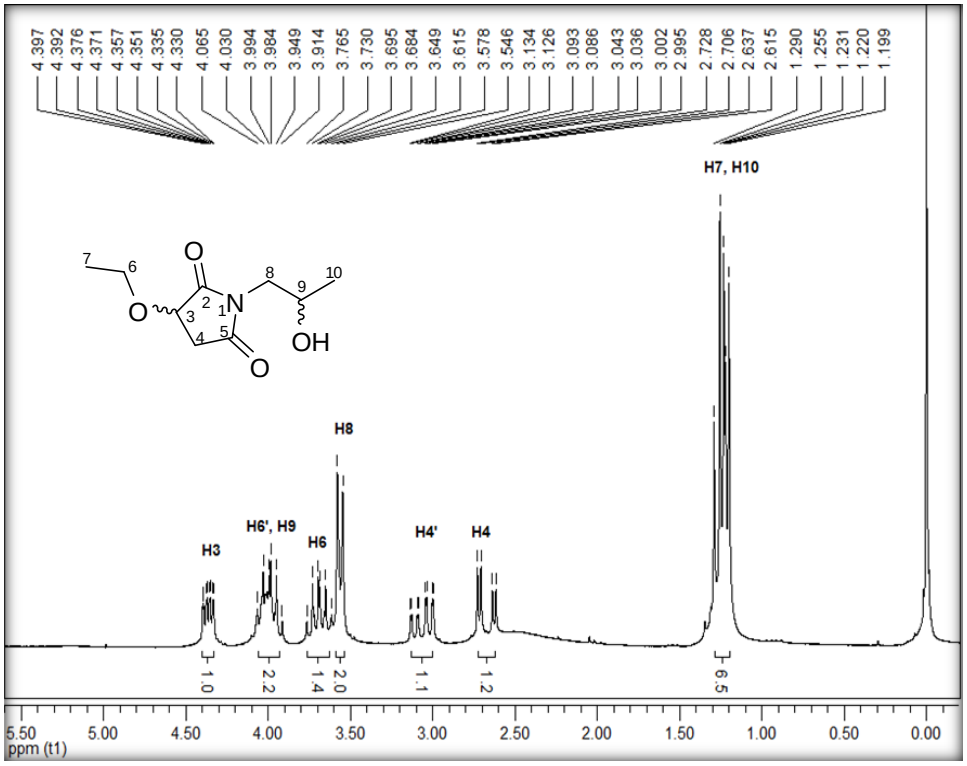

Figure S19. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **6j**.

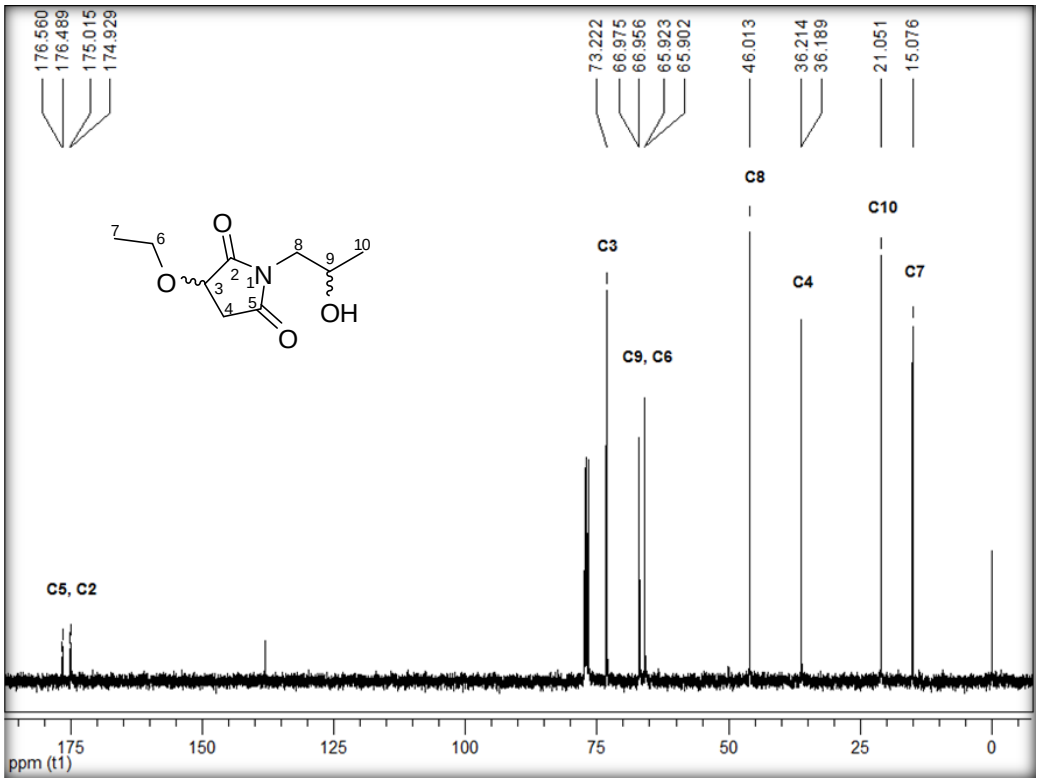

Figure S20. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6j**.

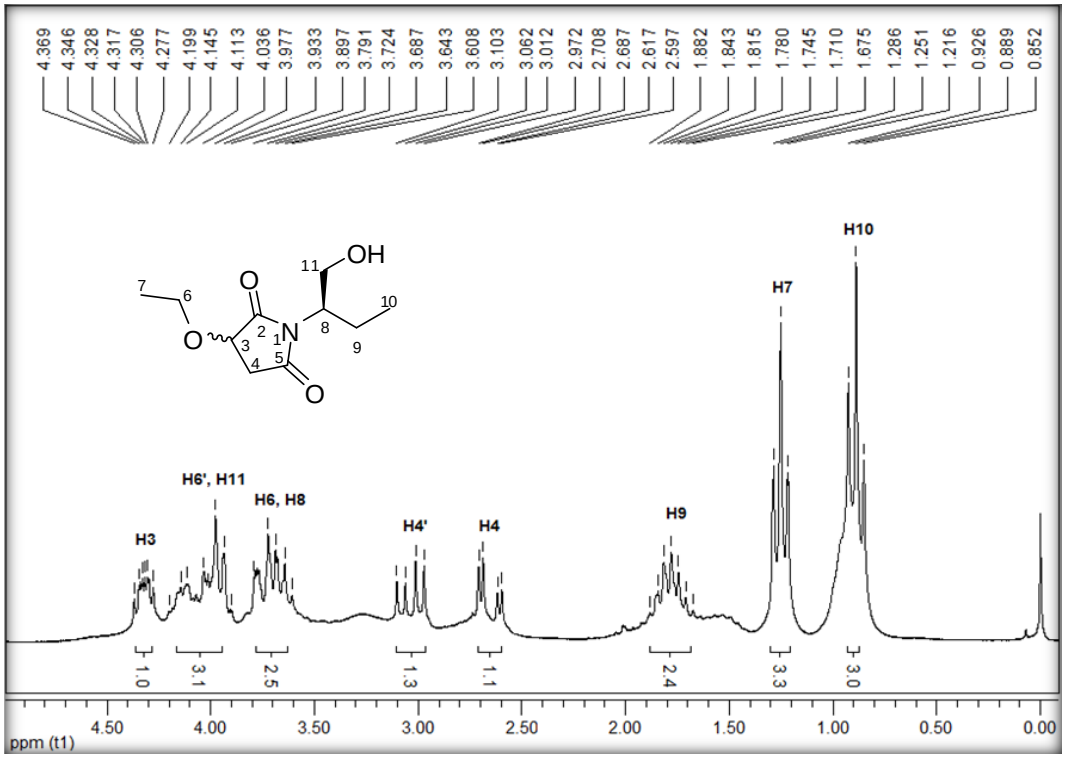

Figure S21. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **6k**.

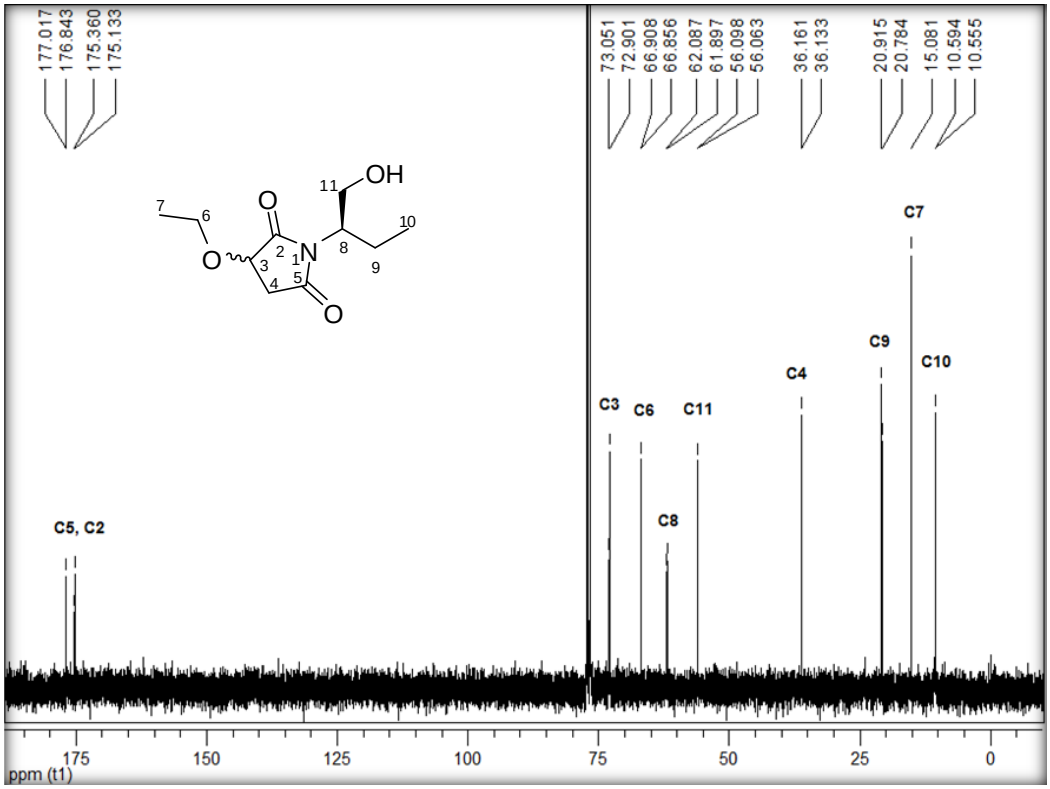

Figure S22. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **6k**.

NMR spectra of compounds 7a-k

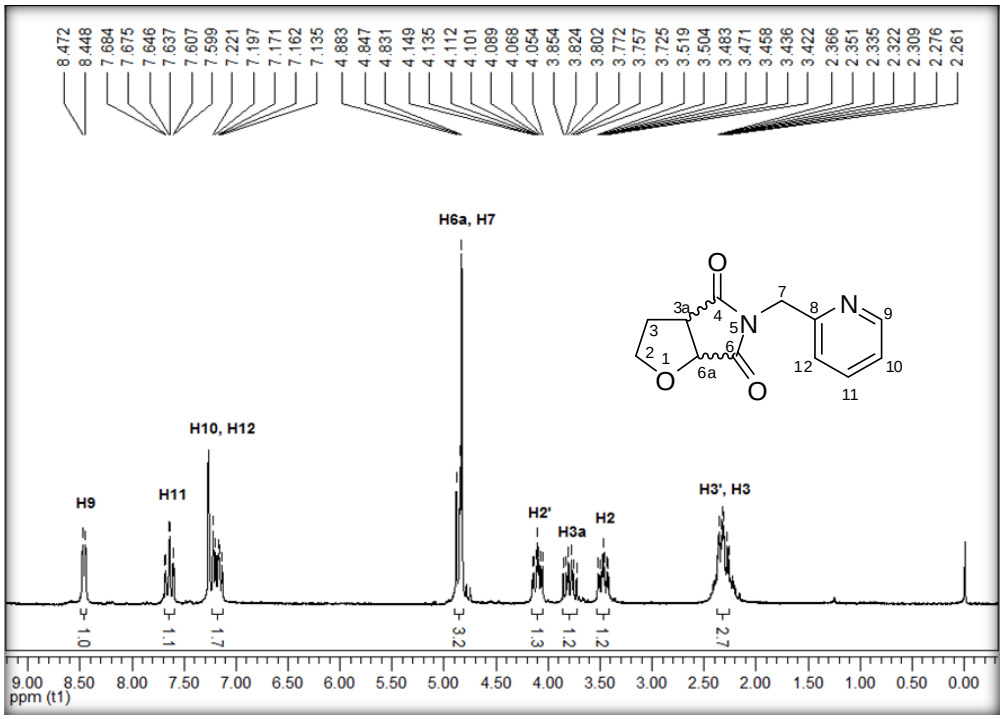

Figure S23. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7a**.

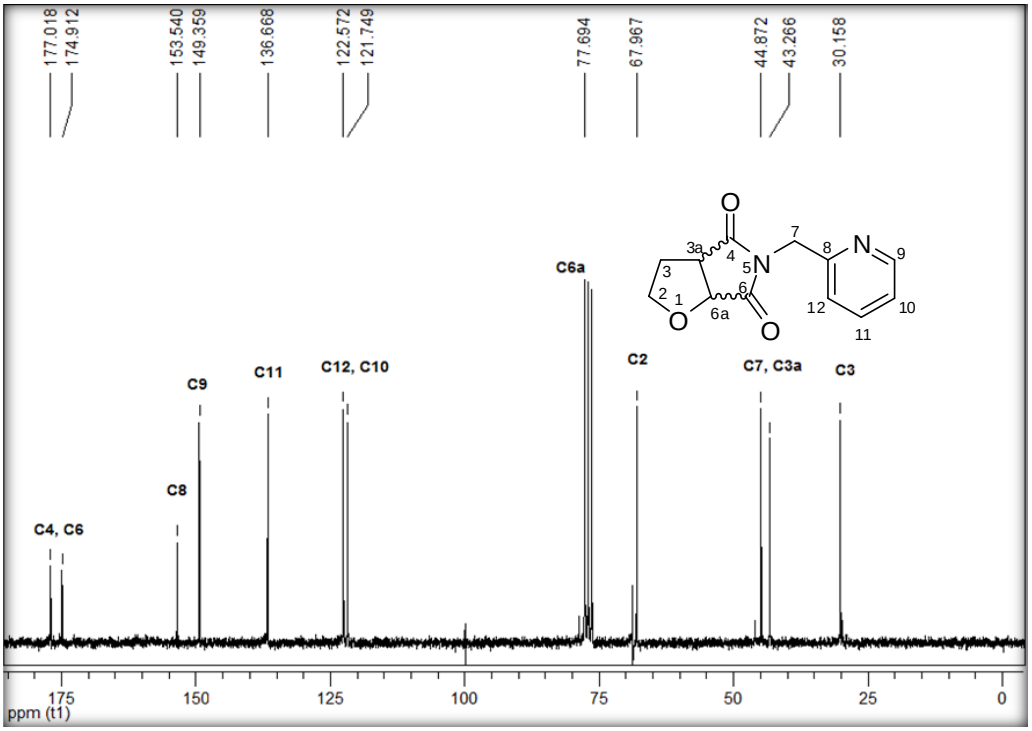

Figure S24. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **7a**.

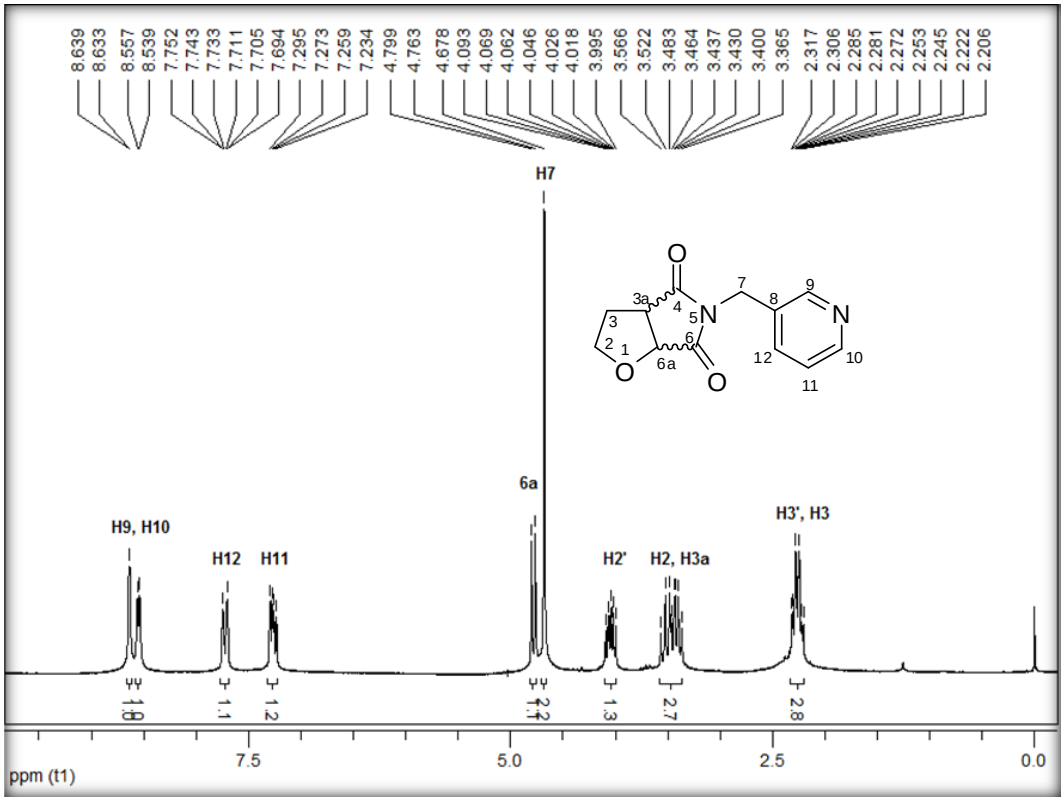

Figure S25. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7b**.

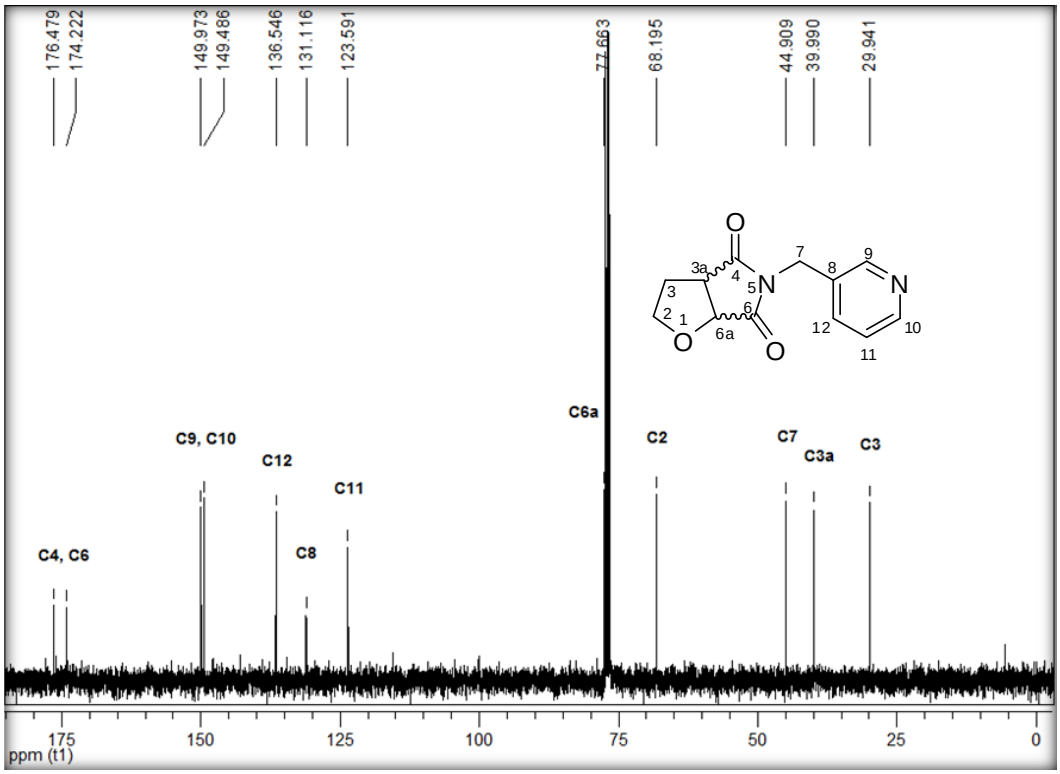

Figure S26. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7b**.

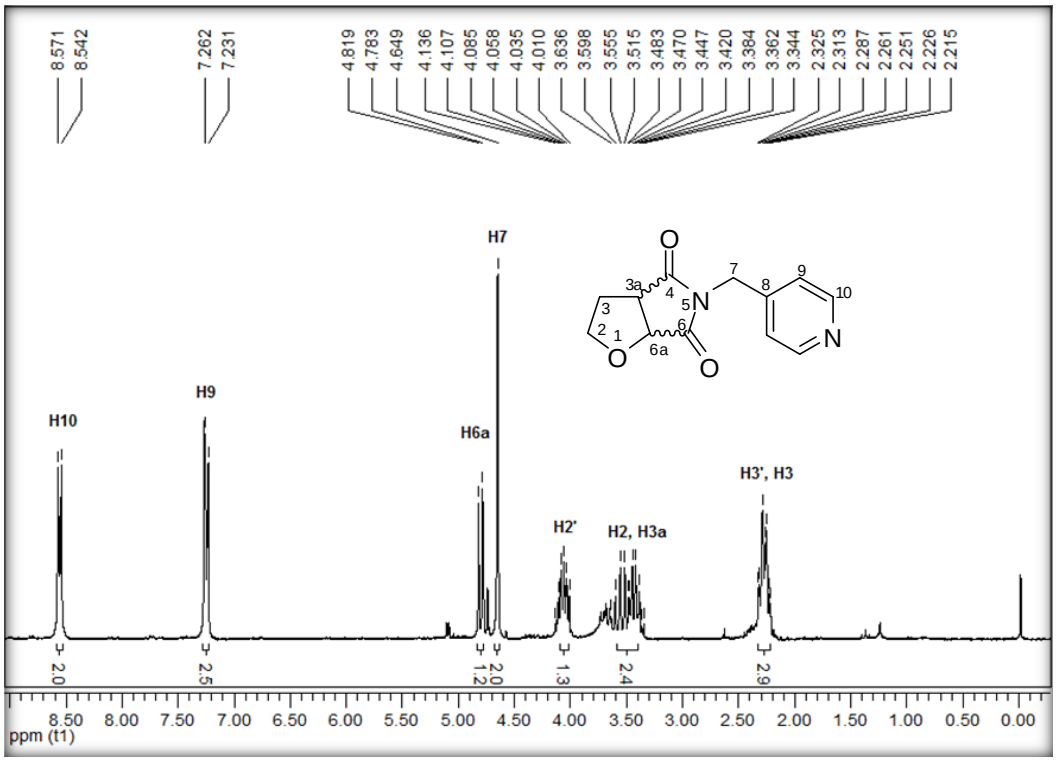

Figure S27. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7c**.

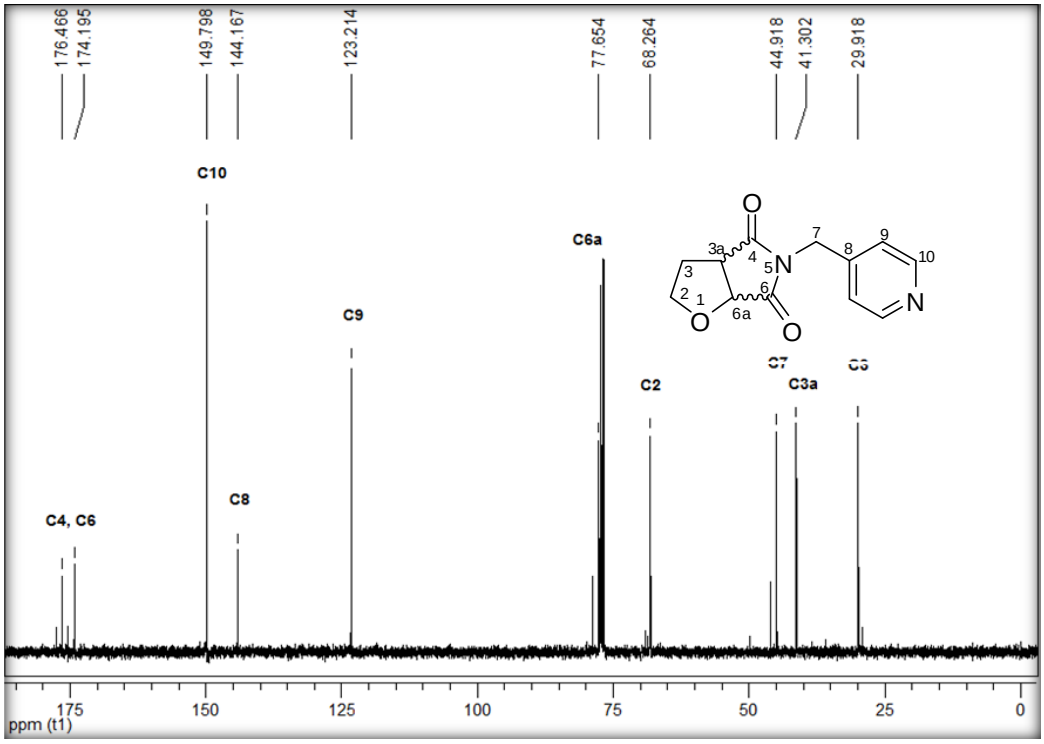

Figure S28. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7c**.

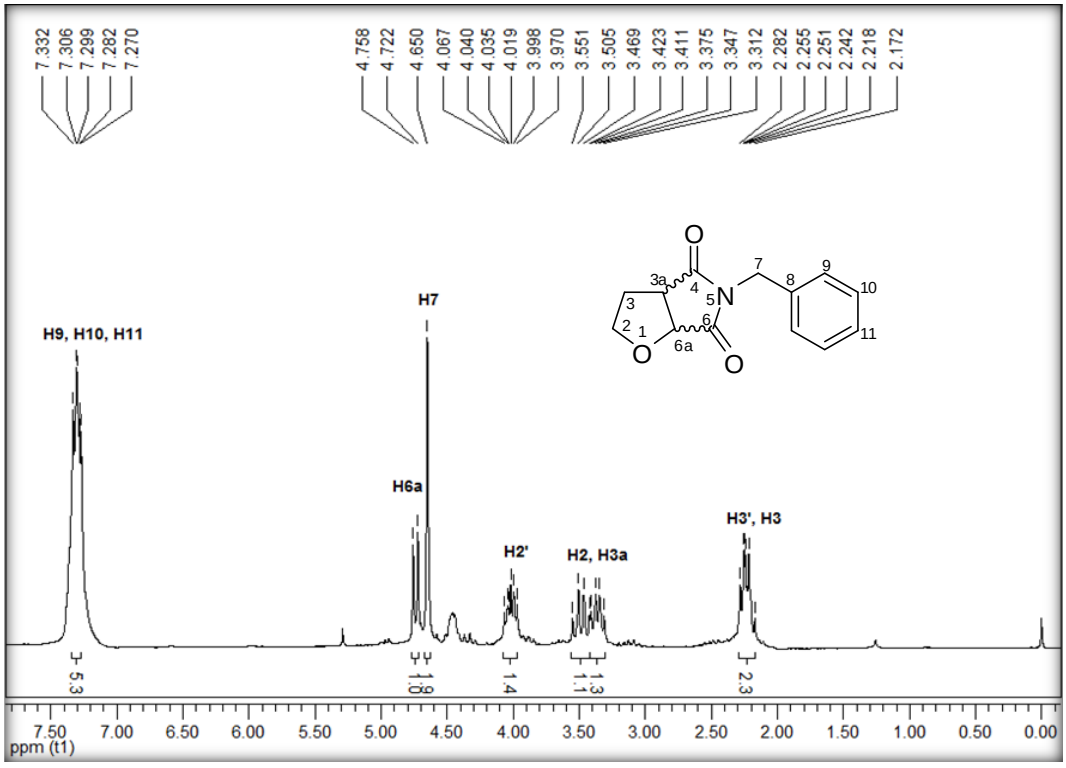

Figure S29. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7d**.

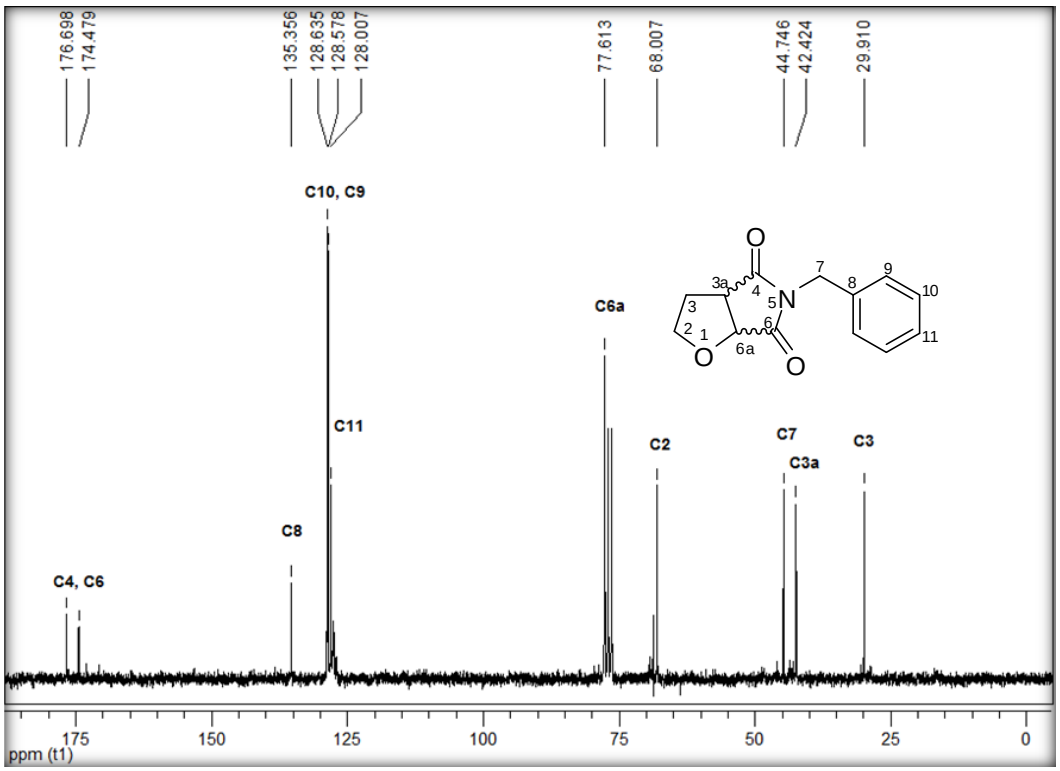

Figure S30. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **7d**.

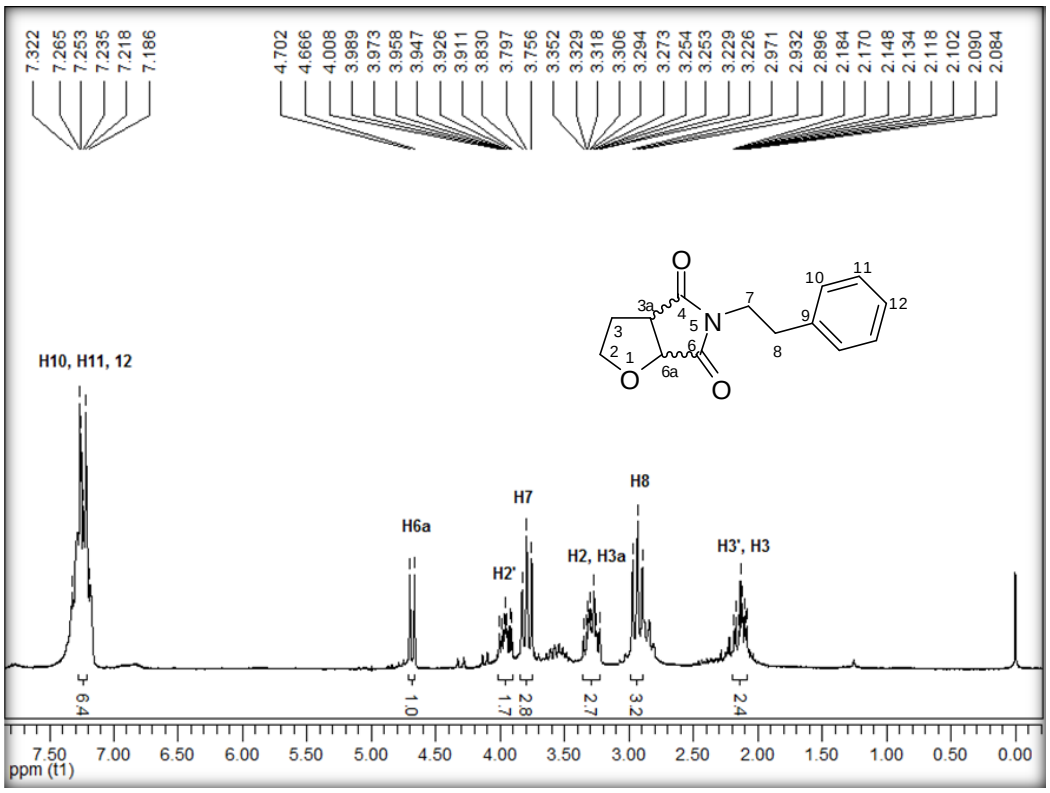

Figure S31. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7e**.

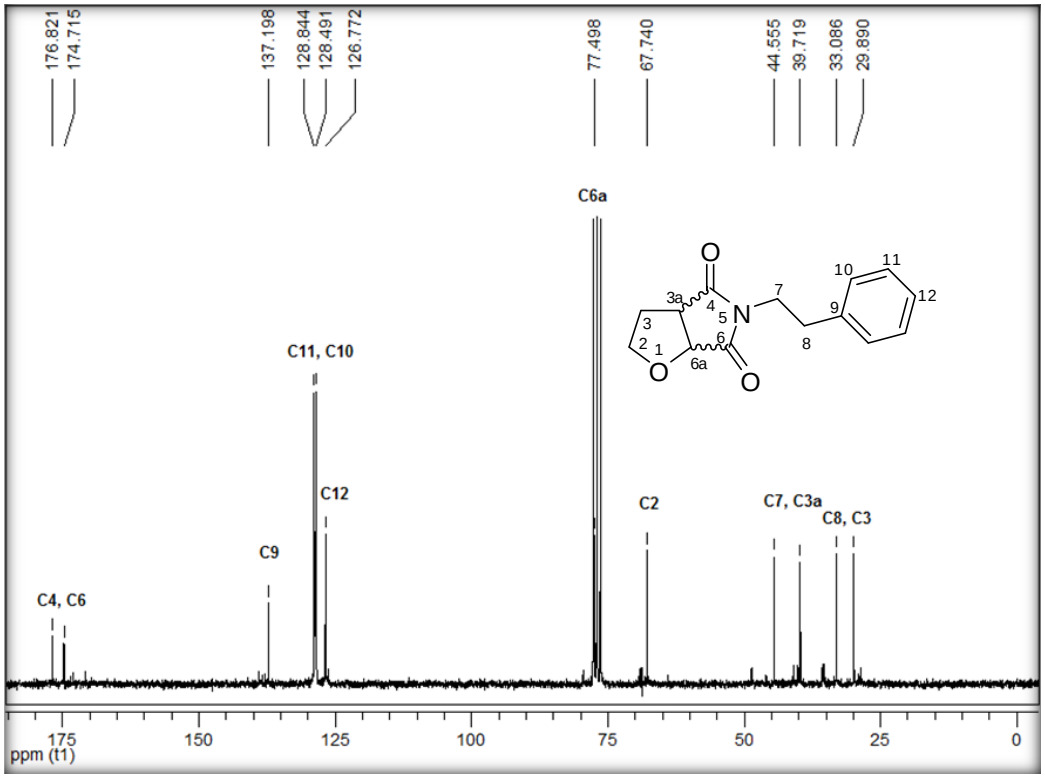

Figure S32. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **7e**.

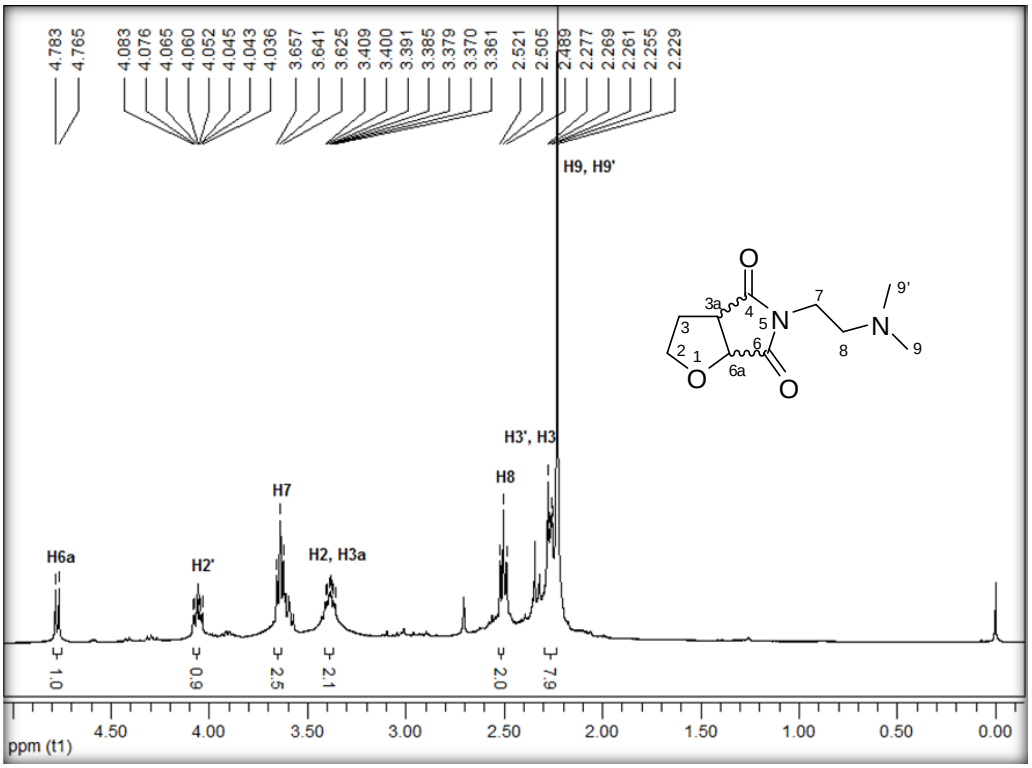

Figure S33. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound **7f**.

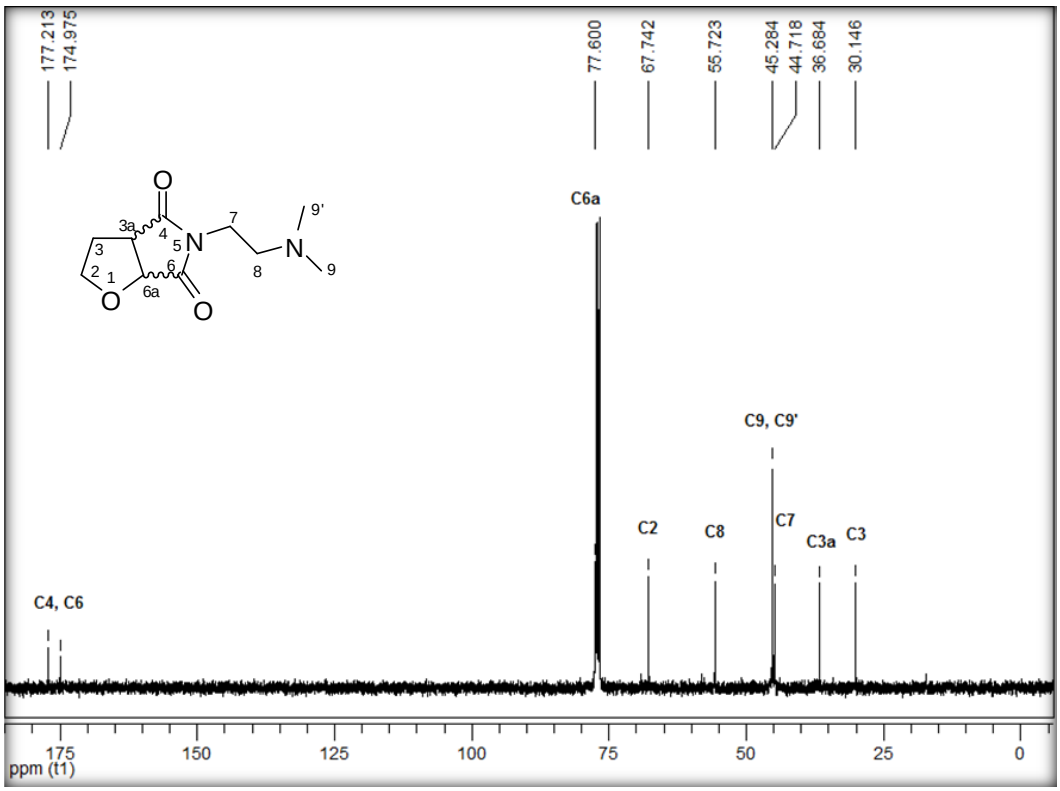

Figure S34. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7f**.

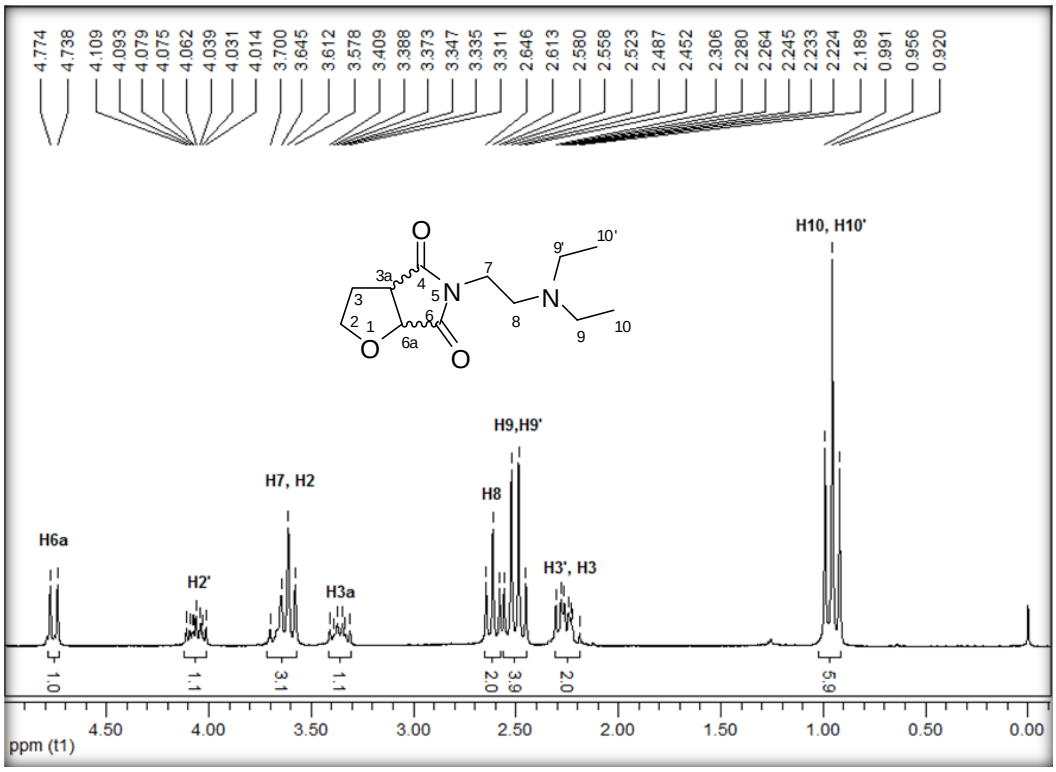

Figure S35. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7g**.

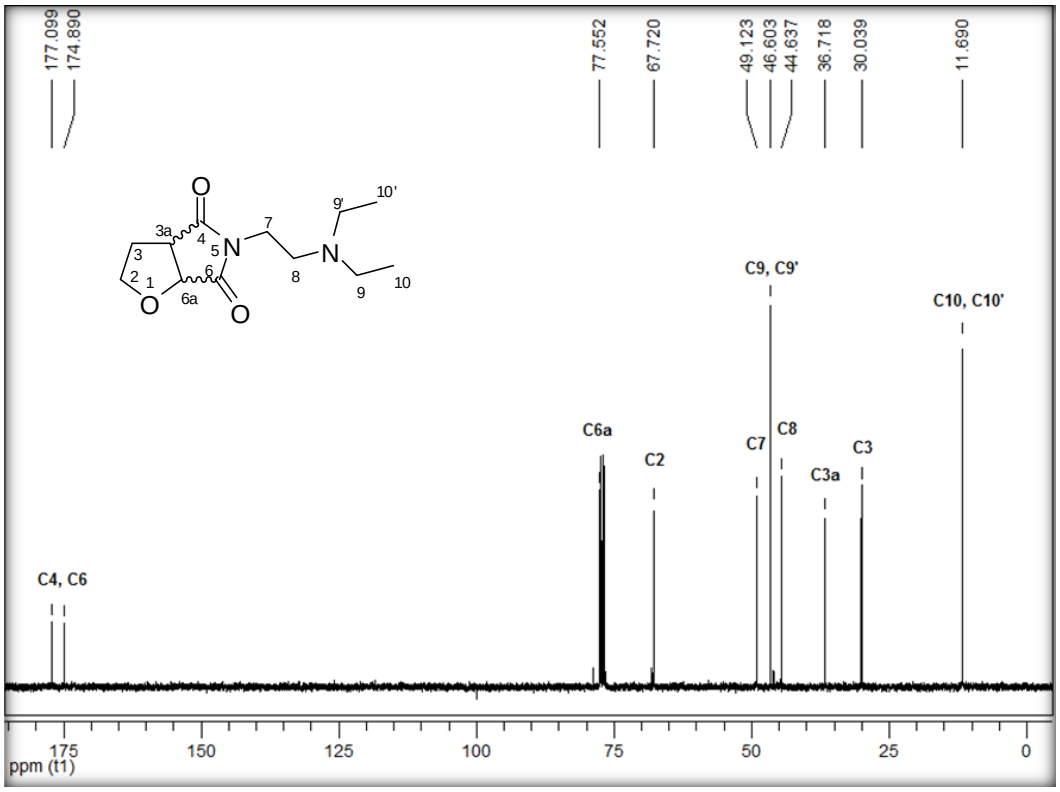

Figure S36. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7g**.

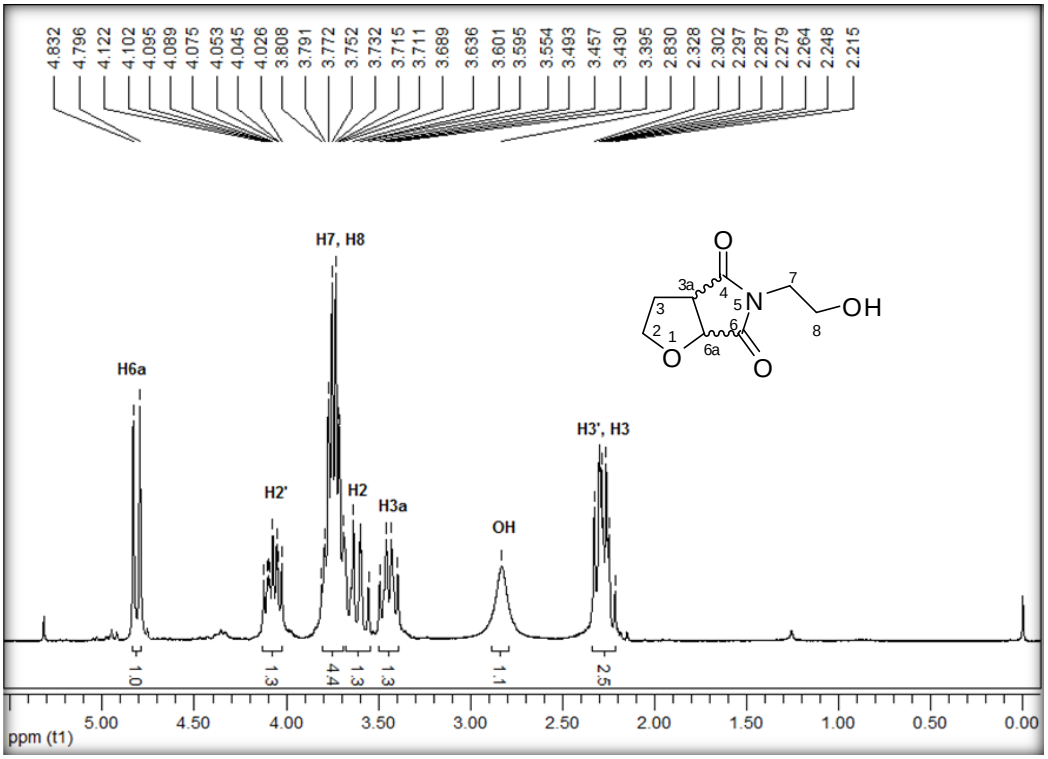

Figure S37. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7h**.

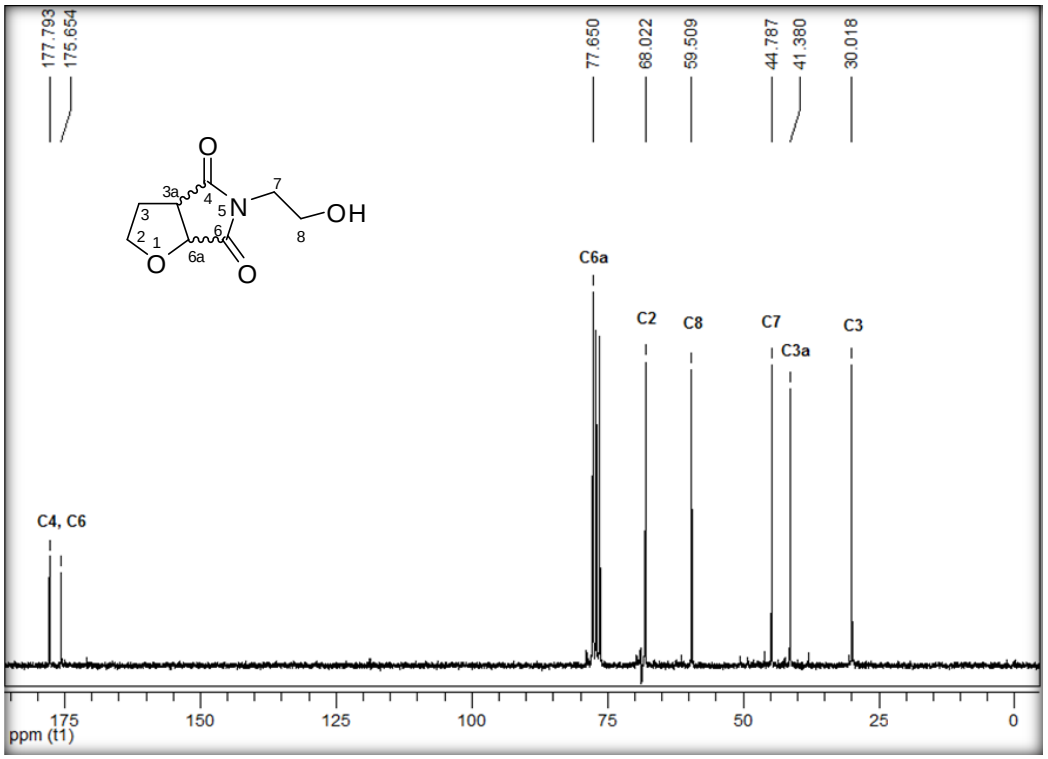

Figure S38. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **7h**.

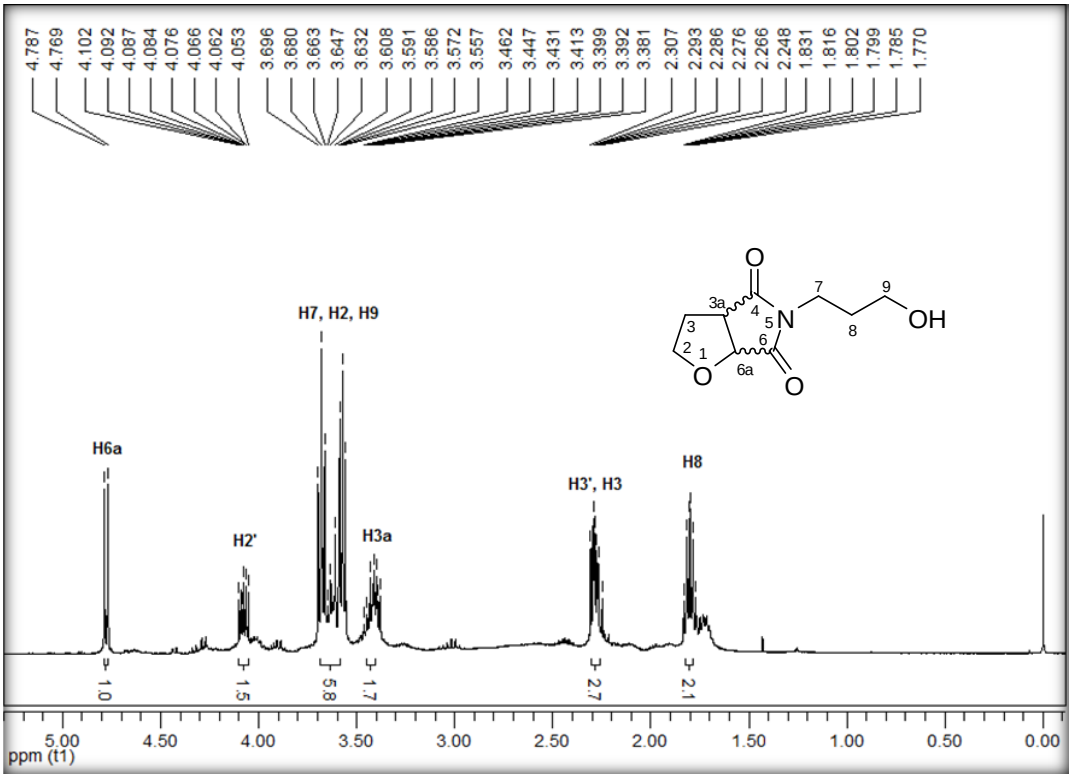

Figure S39. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **7i**.

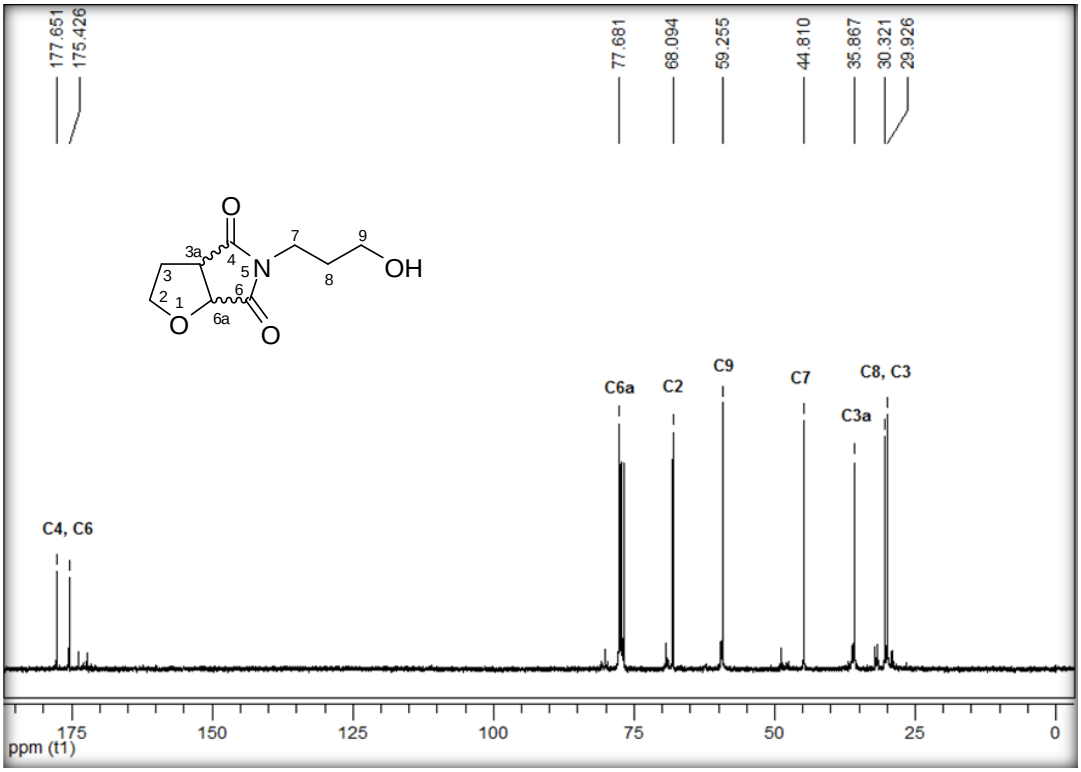

Figure S40. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7i**.

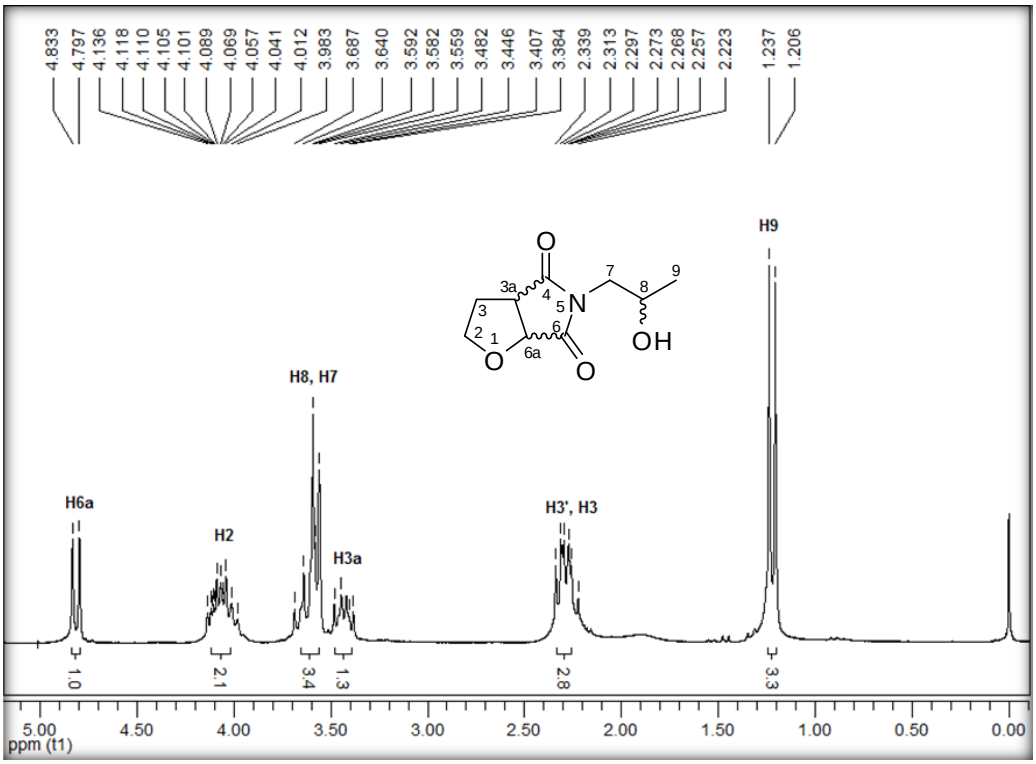

Figure S41. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7j**.

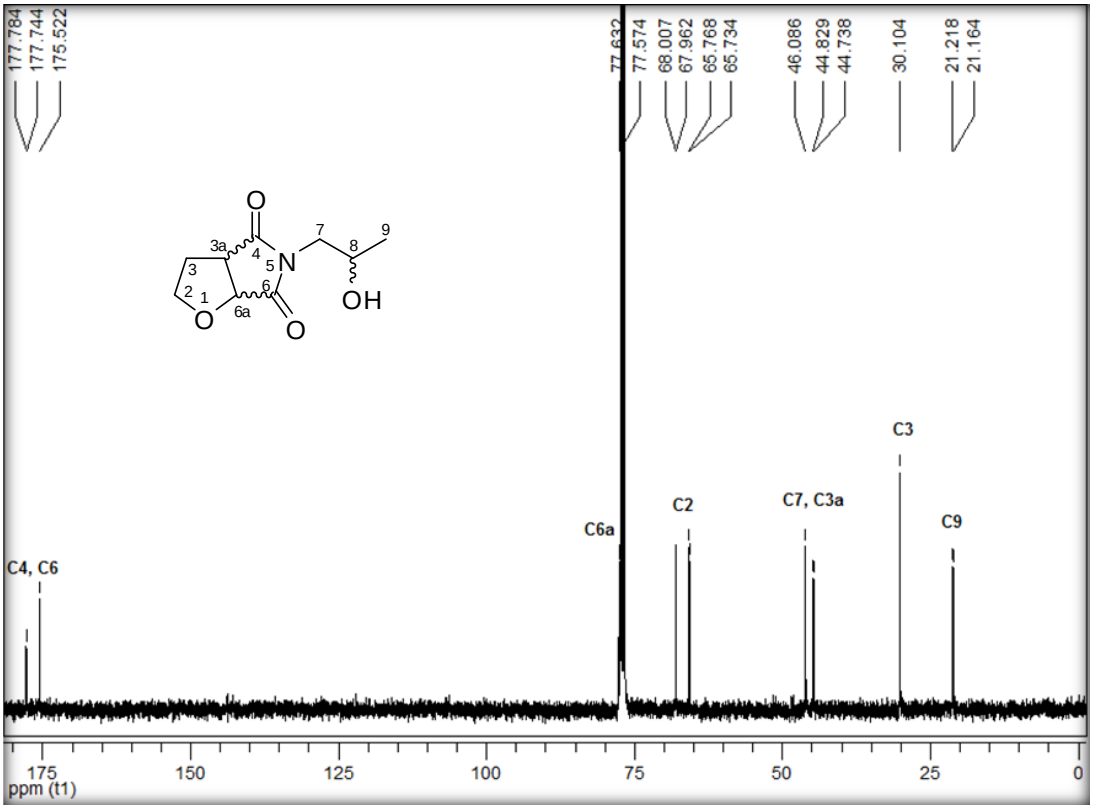

Figure S42. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7j**.

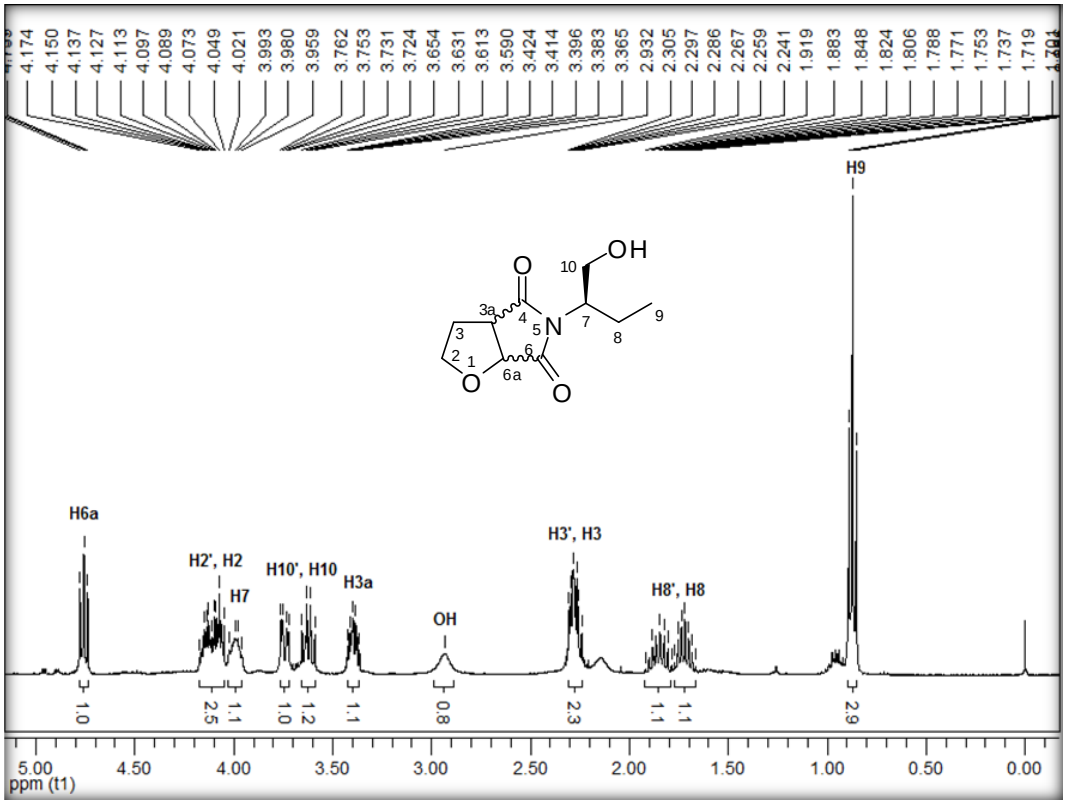

Figure S43. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **7k**.

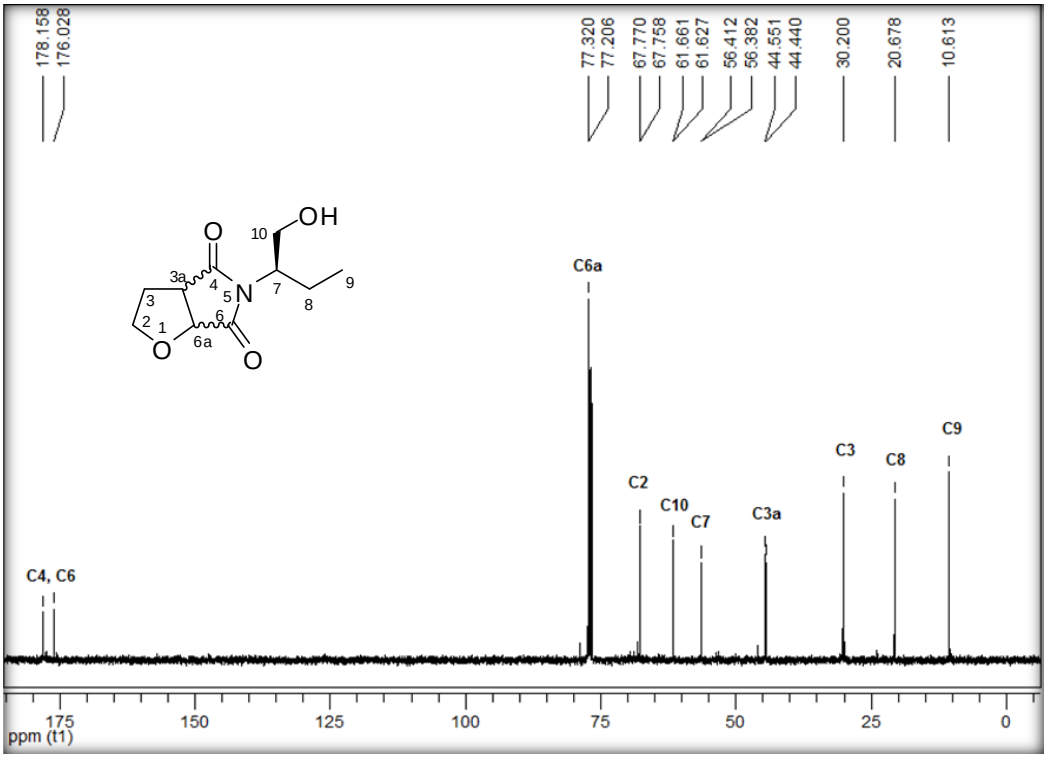

Figure S44. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **7k**.

NMR spectra of compounds 11-13

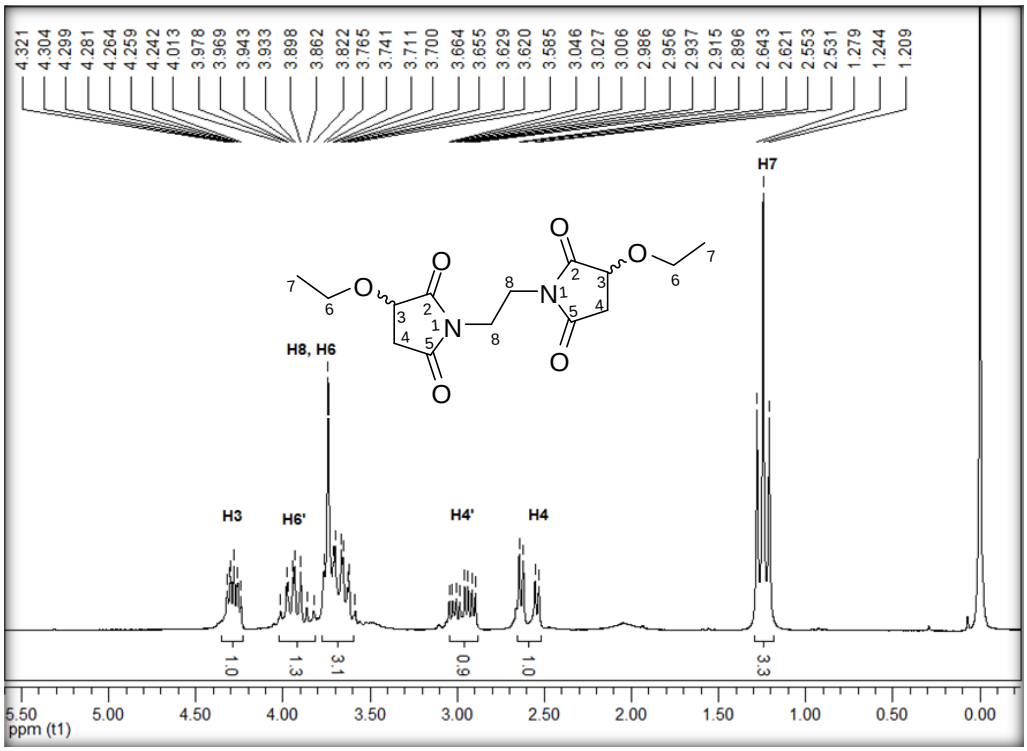

Figure S45. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **11**.

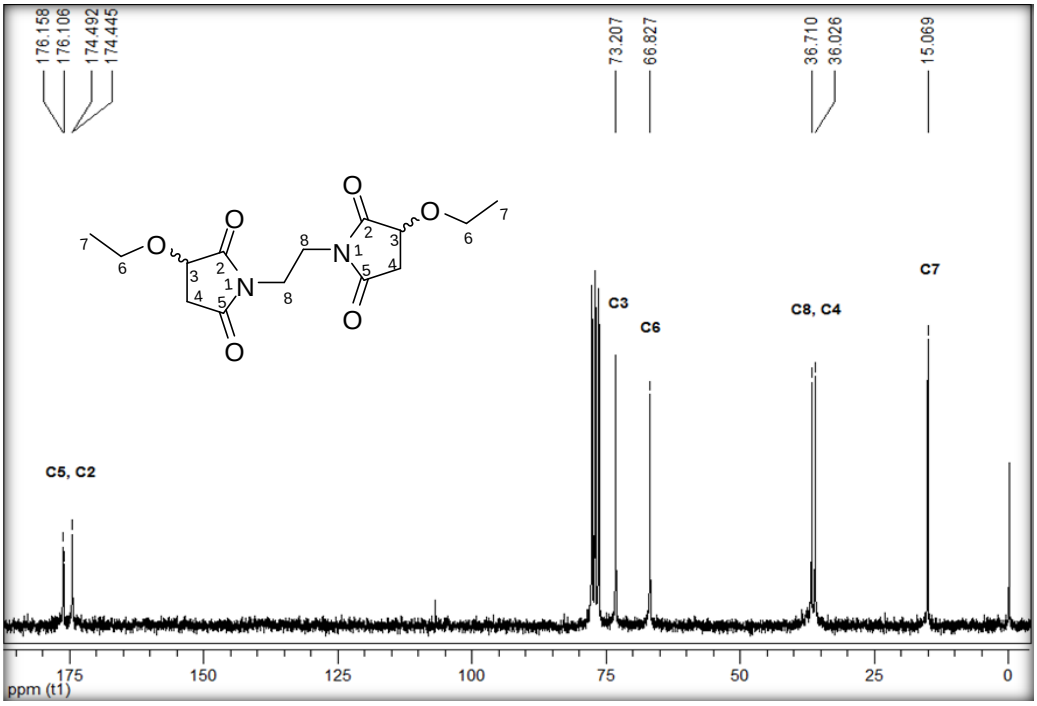

Figure S46. ^{13}C NMR (50 MHz, $CDCl_3$) spectrum of compound **11**.

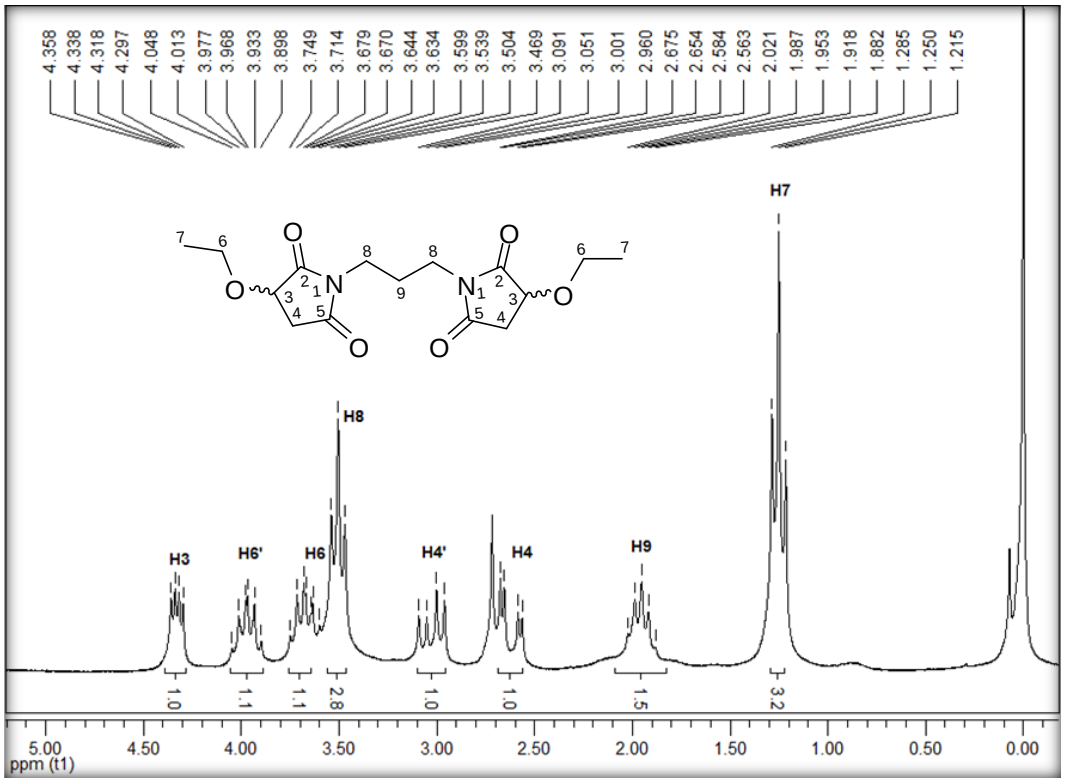

Figure S47. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **12**.

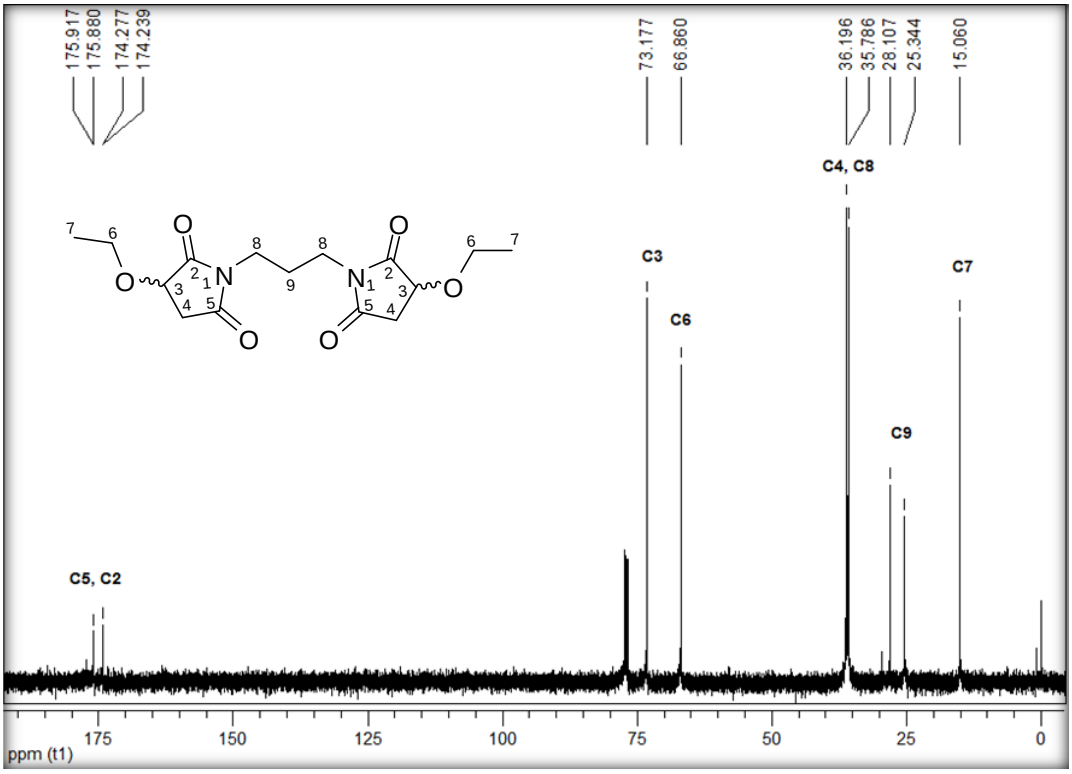

Figure S48. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **12**.

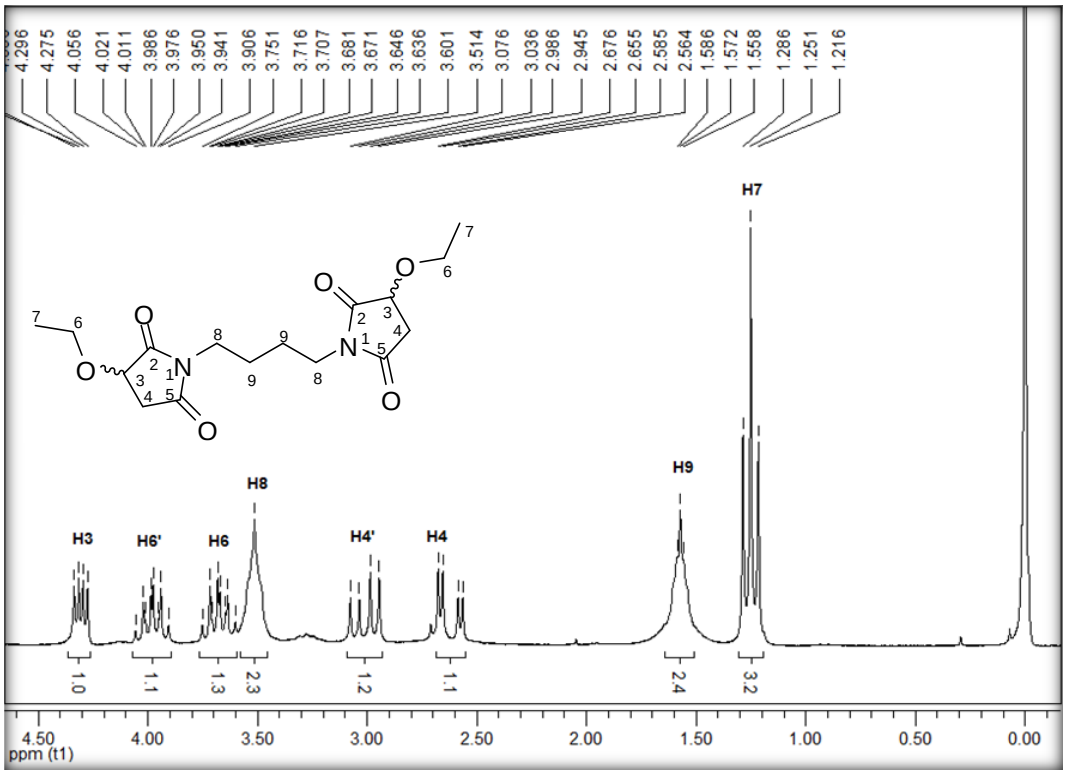

Figure S49. ^{1}H NMR (200 MHz, $CDCl_3$) spectrum of compound **13**.

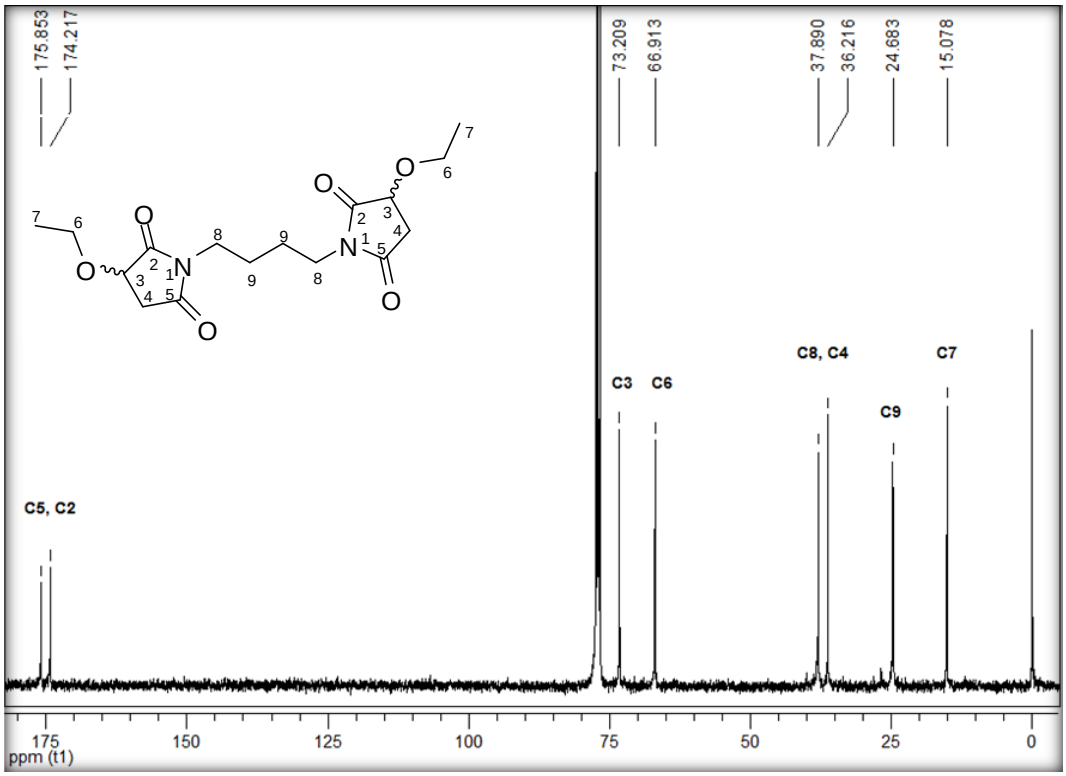

Figure S50. ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of compound **13**.

References

1. (a) Effenberger, F.; Maier, R.; Schönwälder, K. H.; Ziegler, T. *Chem. Ber.* **1982**, *115*, 2766. (b) Martins, M. A. P.; Cunico, W.; Pereira, C. M. P.; Sinhorin, A. P.; Flores, A. F. C.; Bonacorso, H. G.; Zanatta, N. *Curr. Org. Synth.* **2004**, *1*, 391.
2. Zanatta, N.; da Silva, F. M; da Rosa, L. S.; Jank, L.; Bonacorso, H. G., Martins, M. A. P. *Tetrahedron Lett.* **2007**, *48*, 6531.

