Supporting Information

One-step Barton decarboxylation by micellar catalysis – application to the synthesis of maleimide derivatives

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General information

All products were purchased either from Acros or Sigma Aldrich depending on their availability and were used without further purification. All solvents were purchased from Carlo Erba. Reactions were performed with an ultrasonic probe (Bioblock Scientific, Vibra cell 75042) and monitored by TLC (Kieselgel 60F254 MERCK aluminium sheet) with detection by UV light and/or phosphomolybdic acid solution and by HPLC (Shimazu). Flash column chromatography was performed on an automatic Grace apparatus, using 40 g connected silica gel cartridges. ^{1}H and ^{13}C NMR spectra were recorded on a 400 MHz Bruker UltraShield 400 MHz/54 mm Ultra long hold. Chemical shifts (δ) are quoted in ppm and are referenced to TMS as an internal standard. Coupling constants (*J*) are quoted in Hz. IR spectra were recorded on a Jasco FT/IR-4100 ATR.

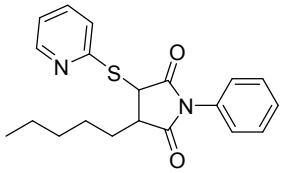
General procedure for the Barton decarboxylation

In a 100 mL Beaker containing a Brij®30 aqueous solution (300 mg in 30 mL), 2-mercaptopyridine-*N*-oxide (150 mg, 1.2 mmol), DCC (250 mg, 1.2 mmol), *N*-phenylmaleimide (268 mg, 1.5 mmol) and carboxylic acid (1.03 mmol) were introduced. The mixture was sonicated 20 min in an ice bath (the ultrasonic probe was placed at 1cm from the bottom of the beaker to ensure a homogenous dispersion). The crude mixture was lyophilized and the residue was purified on flash chromatography (Cyclohexane/EtOAc 1/0 to 7/3).

General procedure for maleimide formation

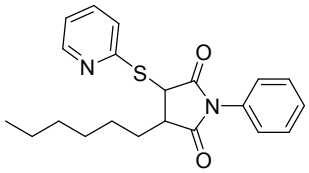
In a 50 mL round bottom flask containing 1-phenylpyridin-2-ylthiopyrrolidine-2,5-dione (1 mmol) product in CH_2Cl_2 (10 mL) was added m-CPBA (246 mg, 10 mmol) and the mixture is stirred for 10 min. at 0°C. Then saturated $NaHCO_3$ solution (30 mL) was added and the crude product extracted with CH_2Cl_2 (30 mL). After the evaporation of the solvent, toluene (20 mL) was added and the media was heated under reflux for 1h. Toluene was evaporated and the crude mixture was purified on flash chromatography (Cyclohexane/EtOAc 1/0 to 7/3).

3-Pentyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (1)



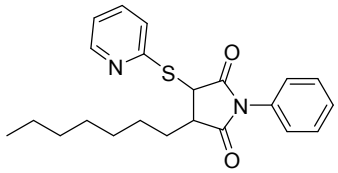
Following general procedure for Barton decarboxylation, white solid (218 mg, 60% yield).
mp: 77-79°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.92 (t, *J* = 7.1 Hz, 3H, CH_3), 1.34-1.37 (m, 4H, CH_2), 1.53-1.60 (m, 2H, CH_2), 1.78-1.87 (m, 1H, CH_2), 2.04-2.12 (m, 1H, CH_2), 3.23 (dt, *J* = 8.6 and 5.2 Hz, 1H, CHCO), 3.92 (d, *J* = 5.6 Hz, 1H, CHS), 7.02 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.22 (d, *J* = 8.1 Hz, 1H, CHπ), 7.34-7.42 (m, 3H, CH_{ar}), 7.46-7.55 (m, 3H, CH_{ar}), 8.29 (ddd, *J* = 5.0, 1.8 and 1.0 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.0 (CH_3), 22.4 (CH_2), 26.1 (CH_2), 30.7 (CH_2), 31.6 (CH_2), 46.9 (CH), 47.2 (CHS), 120.2 (CHπ), 122.1 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.6 (CHπ), 149.0 (CHπN), 156.1 (CπS), 174.4 (*C*OCHS), 177.4 (CO)
ν_{max}/cm^{-1}: 1707 (C=O), 1389 (CH), 1173 (C-N),
HRMS (ESI): found 355.1480; calculated 355.1480 for $C_{20}H_{23}N_2O_2S$

3-Hexyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (2)



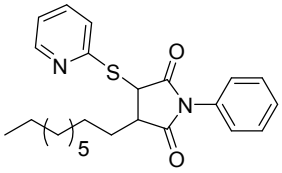
Following general procedure for Barton decarboxylation, colourless wax (235 mg, 62% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.89 (t, *J* = 7.0 Hz, 3H, CH_3), 1.26-1.39 (m, 6H, CH_2), 1.55 (quin, *J* = 7.5 Hz, 2H, CH_2), 1.77-1.87 (m, 1H, CH_2), 2.04-2.13 (m, 1H, CH_2), 3.24 (dt, *J* = 8.7 and 5.3 Hz, 1H, CHCO), 3.90 (d, *J* = 5.7 Hz, 1H, CHS), 7.01 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.21 (d, *J* = 8.1 Hz, 1H, CHπ), 7.34-7.42 (m, 3H, CH_{ar}), 7.46-7.54 (m, 3H, CH_{ar}), 8.29 (ddd, *J* = 4.9, 1.7 and 0.9 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.0 (CH_3), 22.5 (CH_2), 26.4 (CH_2), 29.1 (CH_2), 30.7 (CH_2), 31.5 (CH_2), 46.9 (CH), 47.2 (CHS), 120.2 (CHπ), 122.1 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.6 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1715 (C=O), 1381 (CH), 1183 (C-N),
HRMS (ESI): found 369.1635; calculated 369.1632 for $C_{21}H_{25}N_2O_2S$

3-Heptyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3)



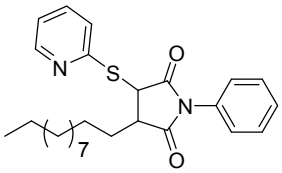
Following general procedure for Barton decarboxylation, colourless oil (228 mg, 58% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26-1.37 (m, 8H, CH_2), 1.55 (quin, *J* = 7.5 Hz, 2H, CH_2), 1.77-1.87 (m, 1H, CH_2), 2.04-2.12 (m, 1H, CH_2), 3.23 (dt, *J* = 8.7 and 5.2 Hz, 1H, CHCO), 3.93 (d, *J* = 5.6 Hz, 1H, CHS), 7.02 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.23 (d, *J* = 8.1 Hz, 1H, CHπ), 7.33-7.41 (m, 3H, CH_{ar}), 7.45-7.55 (m, 3H, CH_{ar}), 8.29 (ddd, *J* = 5.0, 1.7 and 1.0 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.0 (CH_3), 22.6 (CH_2), 26.5 (CH_2), 29.0 (CH_2), 29.4 (CH_2), 30.7 (CH_2), 31.7 (CH_2), 46.9 (CH), 47.1 (CHS), 120.3 (CHπ), 122.2 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.7 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1715 (C=O), 1380 (CH), 1184 (C-N),
HRMS (ESI): found 383.1788; calculated 383.1793 for $C_{22}H_{27}N_2O_2S$

3-Nonyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (4)



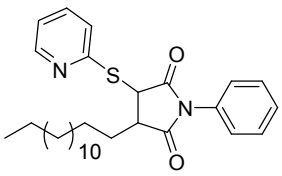
Following general procedure for Barton decarboxylation, white solid (235 mg, 54% yield).
mp: 56-57°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26-1.36 (m, 12H, CH_2), 1.56 (quin, *J* = 7.4 Hz, 2H, CH_2), 1.78-1.87 (m, 1H, CH_2), 2.04-2.12 (m, 1H, CH_2), 3.23 (dt, *J* = 8.8 and 5.1 Hz, 1H, CHCO), 3.92 (d, *J* = 5.6 Hz, 1H, CHS), 7.02 (dd, *J* = 6.7 and 5.0 Hz, 1H, CHπ), 7.23 (d, *J* = 8.1 Hz, 1H, CHπ), 7.35-7.41 (m, 3H, CH_{ar}), 7.46-7.55 (m, 3H, CH_{ar}), 8.29 (d, *J* = 4.5 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.6 (CH_2), 26.5 (CH_2), 29.2 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 30.8 (CH_2), 31.8 (CH_2), 46.9 (CH), 47.2 (CHS), 120.3 (CHπ), 122.2 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.7 (CHπ), 149.0 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1716 (C=O), 1386 (CH), 1180 (C-N),
HRMS (ESI): found 411.2102; calculated 411.2106 for $C_{24}H_{31}N_2O_2S$

1-Phenyl-3-(pyridin-2-ylthio)-4-undecylpyrrolidine-2,5-dione (5)



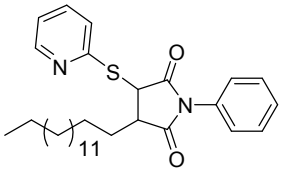
Following general procedure for Barton decarboxylation, white solid (258 mg, 57% yield).
mp: 62-65°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26 (bs, 16H, CH_2), 1.55 (quin, *J* = 7.5 Hz, 2H, CH_2), 1.77-1.87 (m, 1H, CH_2), 2.03-2.12 (m, 1H, CH_2), 3.23 (dt, *J* = 8.7 and 5.2 Hz, 1H, CHCO), 3.92 (d, *J* = 5.7 Hz, 1H, CHS), 7.01 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.22 (dt, *J* = 8.1 and 0.9 Hz, 1H, CHπ), 7.34-7.41 (m, 3H, CH_{ar}), 7.45-7.54 (m, 3H, CH_{ar}), 8.28 (ddd, *J* = 5.0, 1.8 and 1.0 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.6 (CH_2), 26.4 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.5 (2 CH_2), 30.7 (CH_2), 31.8 (CH_2), 46.9 (CH), 47.1 (CHS), 120.2 (CHπ), 122.1 (CHπ), 126.4 (2 CH_{Ph}), 128.4 (CH_{Ph}), 129.0 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.6 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.3 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1714 (C=O), 1388 (CH), 1182 (C-N),
HRMS (ESI): found 439.2418; calculated 439.2419 for $C_{26}H_{35}N_2O_2S$

1-Phenyl-3-(pyridin-2-ylthio)-4-tetradecylpyrrolidine-2,5-dione (6)



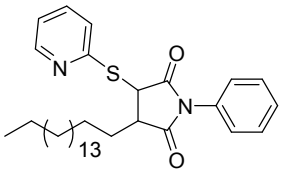
Following general procedure for Barton decarboxylation, white solid (301 mg, 61% yield).
mp: 72-74°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.8 Hz, 3H, CH_3), 1.26 (bs, 22H, CH_2), 1.55 (quin, *J* = 7.5 Hz, 2H, CH_2), 1.77-1.87 (m, 1H, CH_2), 2.04-2.12 (m, 1H, CH_2), 3.23 (dt, *J* = 8.7 and 5.1 Hz, 1H, CHCO), 3.91 (d, *J* = 5.6 Hz, 1H, CHS), 7.02 (dd, *J* = 6.7 and 5.6 Hz, 1H, CHπ), 7.22 (d, *J* = 8.1 Hz, 1H, CHπ), 7.35-7.41 (m, 3H, CH_{ar}), 7.46-7.55 (m, 3H, CH_{ar}), 8.29 (d, *J* = 4.9 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.7 (CH_2), 26.5 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.6 (2 CH_2), 29.7 (CH_2), 29.7 (CH_2), 30.8 (CH_2), 31.9 (CH_2), 46.9 (CH), 47.1 (CHS), 120.2 (CHπ), 122.1 (CHπ), 126.4 (2 CH_{Ph}), 128.4 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 (C_{Ph}N), 136.7 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1704 (C=O), 1391 (CH), 1174 (C-N),
HRMS (ESI): found 481.2891; calculated 481.2889 for $C_{29}H_{41}N_2O_2S$

3-Pentadecyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (7)



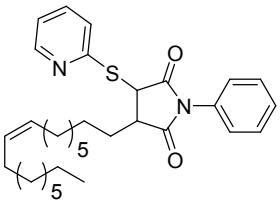
Following general procedure for Barton decarboxylation, white solid (336 mg, 66% yield).
mp: 68-71°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.80 (t, *J* = 6.9 Hz, 3H, CH_3), 1.18-1.36 (m, 24H, CH_2), 1.50 (quin, *J* = 7.4 Hz, 2H, CH_2), 1.70-1.77 (m, 1H, CH_2), 2.96-2.03 (m, 1H, CH_2), 3.13 (dt, *J* = 8.8 and 5.1 Hz, 1H, CHCO), 3.86 (d, *J* = 5.6 Hz, 1H, CHS), 6.95 (dd, *J* = 6.7 and 5.0 Hz, 1H, CHπ), 7.17 (d, *J* = 8.1 Hz, 1H, CHπ), 7.27-7.34 (m, 3H, CH_{ar}), 7.39-7.49 (m, 3H, CH_{ar}), 8.22 (d, *J* = 4.5 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.7 (CH_2), 26.5 (CH_2), 29.4 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.6 (CH_2), 29.6 (CH_2), 29.7 (CH_2), 30.7 (CH_2), 31.9 (CH_2), 46.9 (CH), 47.2 (CHS), 120.3 (CHπ), 122.2 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 ($C_{Ph}N$), 136.8 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1708 (C=O), 1387 (CH), 1173 (C-N),
HRMS (ESI): found 495.3047; calculated 495.3045 for $C_{30}H_{43}N_2O_2S$

3-Heptadecyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (8)



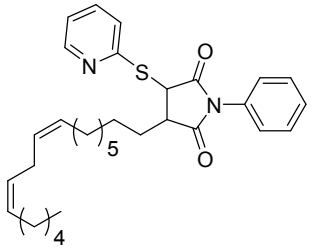
Following general procedure for Barton decarboxylation, white solid (296 mg, 55% yield).
mp: 85-86°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.9 Hz, 3H, CH_3), 1.26 (bs, 28H, CH_2), 1.56 (quin, *J* = 7.5 Hz, 2H, CH_2), 1.78-1.87 (m, 1H, CH_2), 2.04-2.13 (m, 1H, CH_2), 3.23 (dt, *J* = 8.7 and 5.2 Hz, 1H, CHCO), 3.95 (d, *J* = 5.6 Hz, 1H, CHS), 7.03 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.24 (dt, *J* = 8.1 and 0.9 Hz, 1H, CHπ), 7.34-7.42 (m, 3H, CH_{ar}), 7.46-7.56 (m, 3H, CH_{ar}), 8.30 (ddd, *J* = 5.0, 1.8 and 1.0 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.7 (CH_2), 26.5 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (2 CH_2), 29.7 (5 CH_2), 30.8 (CH_2), 31.9 (CH_2), 46.9 (CH), 47.2 (CHS), 120.3 (CHπ), 122.3 (CHπ), 126.4 (2 CH_{Ph}), 128.5 (CH_{Ph}), 129.1 (2 CH_{Ph}), 132.5 ($C_{Ph}N$), 136.8 (CHπ), 148.9 (CHπN), 156.0 (CπS), 174.4 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1708 (C=O), 1388 (CH), 1169 (C-N),
HRMS (ESI): found 523.3363; calculated 523.3359 for $C_{32}H_{47}N_2O_2S$ and found 545.3198; calculated 545.3177 for $C_{32}H_{46}N_2O_2SNa$

(*Z*)-3-(Heptadec-8-en-1-yl)-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (9)



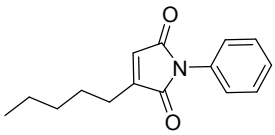
Following general procedure for Barton decarboxylation, colourless oil (321 mg, 60% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.88 (t, *J* = 6.9 Hz, 3H, CH_3), 1.27-1.31 (m, 22H, CH_2), 1.56 (quin, *J* = 7.4 Hz, 2H, CH_2), 1.77-1.87 (m, 1H, CH_2), 2.01-2.05 (m, 1H, CH_2), 3.23 (dt, *J* = 8.7 and 5.2 Hz, 1H, CHCO), 3.91 (d, *J* = 5.7 Hz, 1H, CHS), 5.35 (dt, *J* = 5.7 and 3.5 Hz, 2H, CH), 7.01 (ddd, *J* = 7.4, 5.0 and 1.0 Hz, 1H, CHπ), 7.23 (dt, *J* = 8.1 and 0.9 Hz, 1H, CHπ), 7.35-7.41 (m, 3H, CH_{ar}), 7.46-7.55 (m, 3H, CH_{ar}), 8.29 (ddd, *J* = 4.9, 1.8 and 0.9 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.1 (CH_3), 22.7 (CH_2), 26.5 (CH_2), 27.1 (CH_2), 27.2 (CH_2), 29.1 (CH_2), 29.2 (CH_2), 29.3 (2 CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.7 (CH_2), 29.7 (CH_2), 30.7 (CH_2), 31.9 (CH_2), 46.9 (CH), 47.1 (CHS), 120.2 (CHπ), 122.1 (CHπ), 126.4 (2 CH_{Ph}), 128.4 (CH_{Ph}), 129.1 (2 CH_{Ph}), 129.7 (CH), 130.0 (CH), 132.5 ($C_{Ph}N$), 136.6 (CHπ), 149.0 (CHπN), 156.0 (CπS), 174.3 (*C*OCHS), 177.3 (CO)
ν_{max}/cm^{-1}: 1717 (C=O), 1384 (CH), 1179 (C-N),
HRMS (ESI): found 521.3221; calculated 521.3202 for $C_{32}H_{45}N_2O_2S$

3-((8*Z*,11*Z*)-Heptadeca-8,11-dien-1-yl)-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (10)



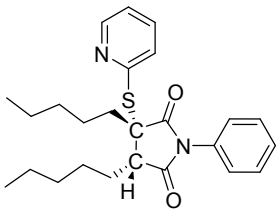
Following general procedure for Barton decarboxylation, colourless oil (327 mg, 61% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.89 (t, *J* = 7.0 Hz, 3H, CH_3), 1.29-1.37 (m, 14H, CH_2), 1.55 (quin, *J* = 7.2 Hz, 2H, CH_2), 1.76-1.86 (m, 1H, CH_2), 2.06 (q, *J* = 6.8 Hz, 5H, CH_2), 2.78 (t, *J* = 6.3 Hz, 2H, CH_2), 3.23 (dt, *J* = 8.7 and 5.2 Hz, 1H, CHCO), 3.89 (d, *J* = 5.7 Hz, 1H, CHS), 5.30-5.43 (m, 4H, CH), 6.98 (ddd, *J* = 7.3, 5.0 and 0.8 Hz, 1H, CHπ), 7.19 (d, *J* = 8.1 Hz, 1H, CHπ), 7.34-7.40 (m, 3H, CH_{ar}), 7.44-7.52 (m, 3H, CH_{ar}), 8.27 (ddd, *J* = 4.9, 1.7 and 1.0 Hz, 1H, CHπ)
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.0 (CH_3), 22.4 (CH_2), 25.5 (CH_2), 26.4 (CH_2), 27.1 (2 CH_2), 29.0 (CH_2), 29.1 (CH_2), 29.2 (CH_2), 29.3 (CH_2), 29.5 (CH_2), 30.6 (CH_2), 31.4 (CH_2), 46.8 (CH), 47.0 (CHS), 120.1 (CHπ), 121.9 (CHπ), 126.3 (2 CH_{Ph}), 127.8 (CH), 127.9 (CH), 128.3 (CH_{Ph}), 128.9 (2 CH_{Ph}), 129.9 (CH), 130.1 (CH), 132.5 (C_{Ph}N), 136.5 (CHπ), 148.9 (CHπN), 155.9 (CπS), 174.2 (*C*OCHS), 177.2 (CO)
ν_{max}/cm^{-1}: 1717 (C=O), 1382 (CH), 1179 (C-N),
HRMS (ESI): found 519.3052; calculated 519.3046 for $C_{32}H_{43}N_2O_2S$

3-Pentyl-1-phenyl-1*H*-pyrrole-2,5-dione (11)

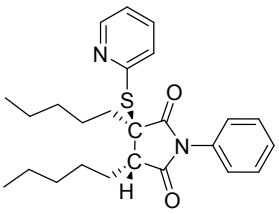


Following general procedure for maleimide formation, white solid, (242 mg, quantitative yield).
mp: 72-73°C
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.93 (t, *J* = 7.1 Hz, 3H, CH_3), 1.33-1.42 (m, 4H, CH_2), 1.63-1.70 (m, 2H, CH_2), 2.52 (dt, *J* = 7.7 and 1.7 Hz, 2H, CH_2), 6.43 (t, *J* = 1.2 Hz, 1H, CH), 7.33-7.36 (m, 3H, CH_{ar}), 7.43-7.48 (m, 2H, CH_{ar})
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 13.9 (CH_3), 22.3 (CH_2), 25.5 (CH_2), 26.8 (CH_2), 31.3 (CH_2), 125.9 (2 CH_{Ph}), 126.3 (*C*HCO), 127.6 (CH_{Ph}), 129.0 (2 CH_{Ph}), 131.6 (C_{Ph}N), 150.4 (CO*C*CH_2), 169.7 (CO), 170.4 (*C*OCCH_2)
ν_{max}/cm^{-1}: 1707 (C=O), 1388 (CH), 1179 (C-N)
HRMS (ESI): found 244.1340, calculated 244.1338 for $C_{15}H_{18}NO_2$

3,4-Dipentyl-1-phenyl-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione ((±)12a and (±)12b)



(±)12a



(±)12b

In a 100 mL Beaker containing a Brij®30 aqueous solution (300 mg in 30 mL), 2-mercaptopyridine-*N*-oxide (225 mg, 1.8 mmol), DCC (375 mg, 1.8 mmol), 11 (243 mg, 1 mmol) and caproic acid (174 mg,1.5 mmol) were introduced. The mixture was sonicated 20 min in an ice bath (the ultrasonic probe was placed at 1cm from the bottom of the beaker to ensure a homogenous dispersion). The crude mixture was lyophilized and the residue was purified on flash chromatography (Cyclohexane/EtOAc 1/0 to 7/3).

Pale yellow oil (200 mg, 47% yield).

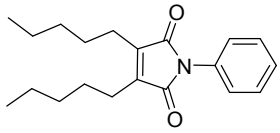
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.85-0.93 (m, 6H, CH_3), 1.27-1.38 (m, 9H, CH_2), 1.56-1.77 (m, 4H, CH_2), 1.82-2.16 (m, 3H, CH_2), 2.96 (dd, *J* = 7.1 and 5.7 Hz, 0.6H, CH), 3.65 (t, *J* = 6.8 Hz, 0.4H, CH), 6.99 (ddd, *J* = 7.4, 6.2, and 5.0 Hz, 1H, CHπ), 7.17-7.22 (m, 1H, CHπ), 7.26-7.29 (m, 1H, CHπ), 7.32-7.52 (m, 5H, CH_{Ph}), 8.19 (ddd, *J* = 5.0, 1.8 and 0.9 Hz, 0.6H, CHπ), 8.30 (ddd, *J* = 5.0, 1.8 and 0.9 Hz, 0.4H, CHπ)

^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 13.9 (CH_3), 14.0 (CH_3), 22.2 (CH_2), 22.3 (CH_2), 22.3 (CH_2), 22.3 (CH_2), 24.0 (CH_2), 24.3 (CH_2), 28.0 (CH_2), 28.1 (CH_2), 28.7 (CH_2), 31.7 (2 CH_2), 31.7 (CH_2), 31.8 (CH_2), 33.9 (*C*H_2CH), 38.4 (*C*H_2CS), 47.8 (*C*HCO), 48.1 (*C*HCO), 56.9 (*C*SCO), 59.0 (*C*SCO), 120.0 (CHπ), 120.2 (CHπ), 122.0 (CHπ), 122.1 (CHπ), 126.3 (2 CH_{Ph}), 126.4 (2 CH_{Ph}), 128.1 (CH_{Ph}), 128.3 (CH_{Ph}), 128.9 (2 CH_{Ph}), 129.0 (2 CH_{Ph}), 132.6 (C_{Ph}N), 136.4 (CHπ), 136.4 (CHπ), 148.7 (CHπN), 148.9 (CHπN), 156.9 (CπS), 157.4 (CπS), 176.2 (*C*OCS), 176.9 (*C*OCS), 177.1 (*C*OCH), 177.5 (*C*OCH)

ν_{max}/cm^{-1}: 1714 (C=O), 1387 (CH), 1182 (C-N),

HRMS (ESI): found 447.2086, calculated 447.2082 for $C_{25}H_{32}N_2O_2SNa$

3,4-Dipentyl-1-phenyl-1*H*-pyrrole-2,5-dione (13)



Following general procedure for maleimide formation, pale yellow oil (310 mg, quantitative yield).

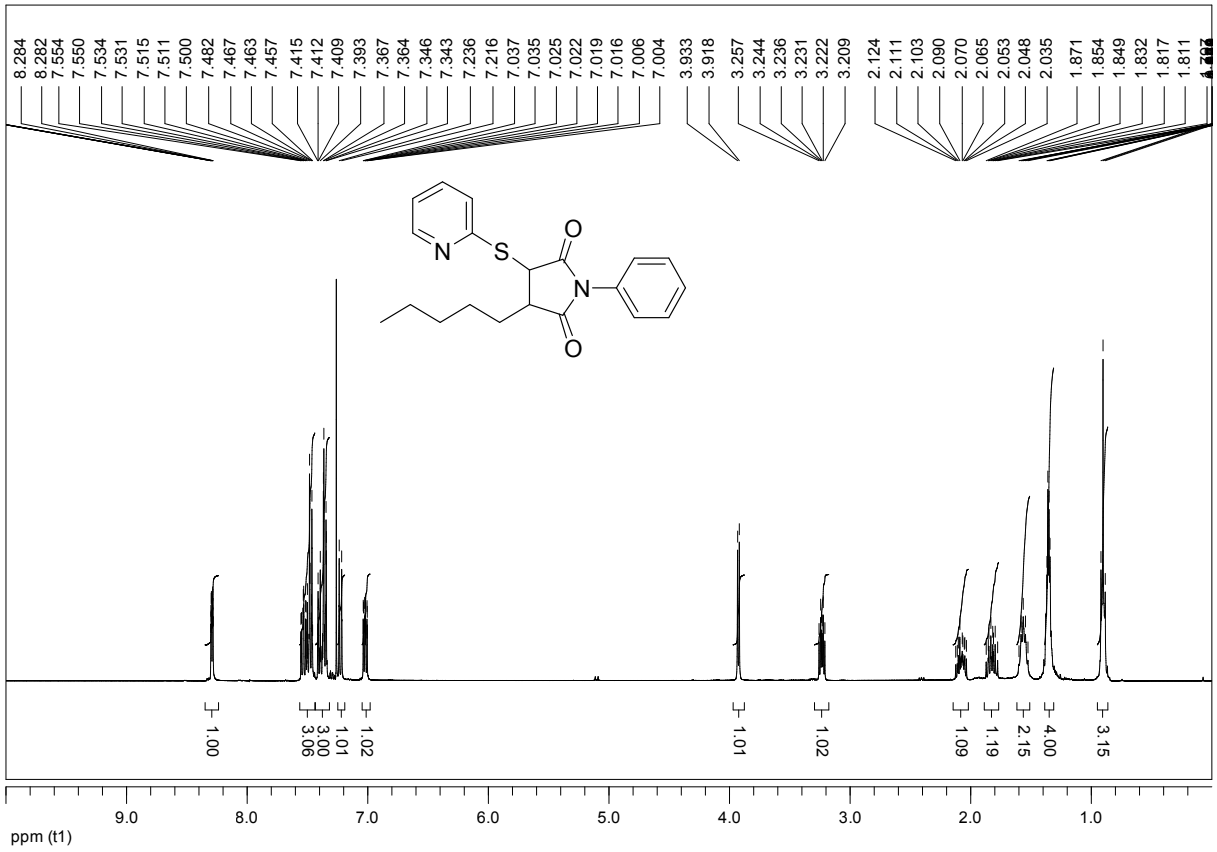
^{1}H NMR (400 MHz, $CDCl_3$) δ ppm: 0.92 (t, *J* = 7.1 Hz, 6H, CH_3), 1.34-1.39 (m, 8H, CH_2), 1.58-1.61 (m, 4H, CH_2), 2.46 (t, *J* = 7.7 Hz, 4H, CH_2), 7.31-7.38 (m, 3H, CH_{ar}), 7.42-7.46 (m, 2H, CH_{ar})

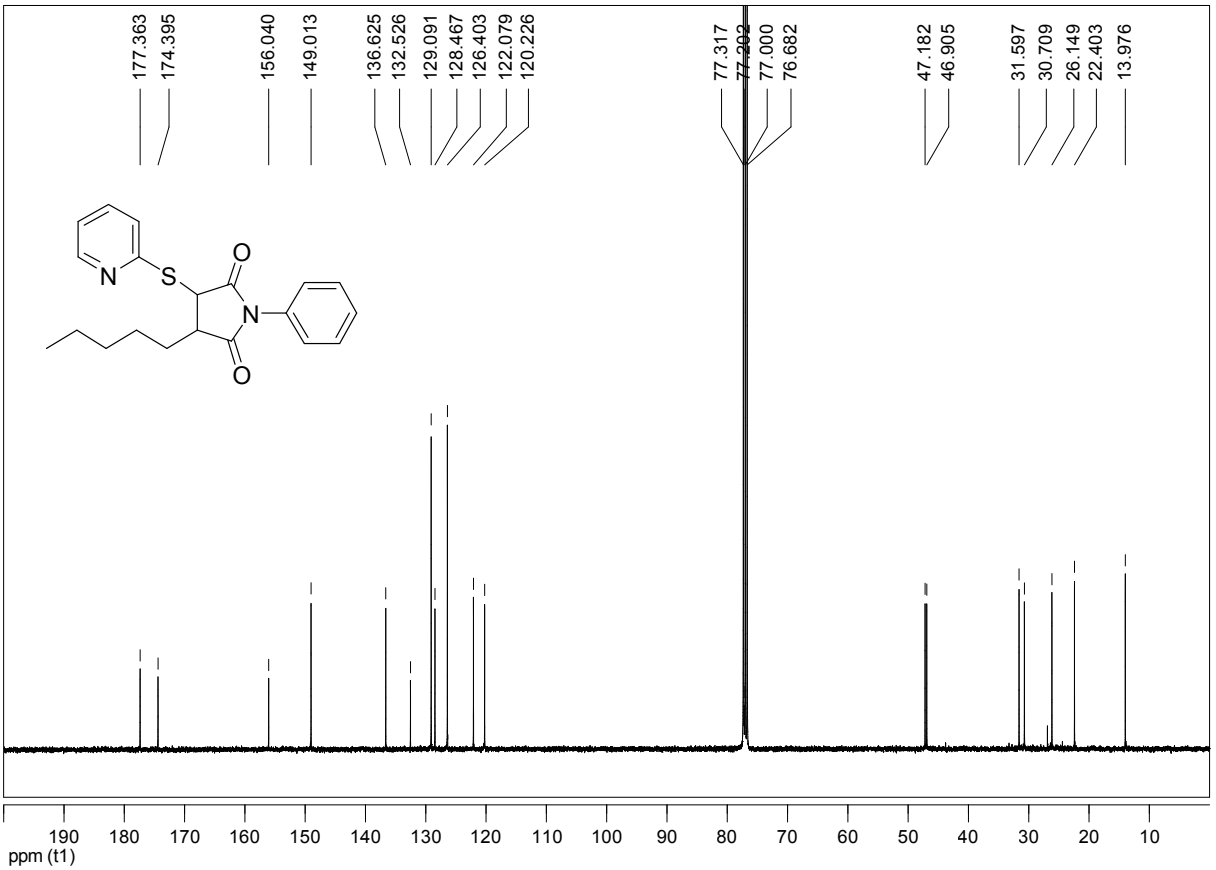
^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 13.9 (CH_3), 22.4 (CH_2), 23.8 (CH_2), 28.3 (CH_2), 31.8 (CH_2), 125.7 (2 CH_{Ph}), 127.2 (CH_{Ph}), 129.0 (2 CH_{Ph}), 132.0 (C_{Ph}N), 141.2 (C=C), 170.8 (*C*O)

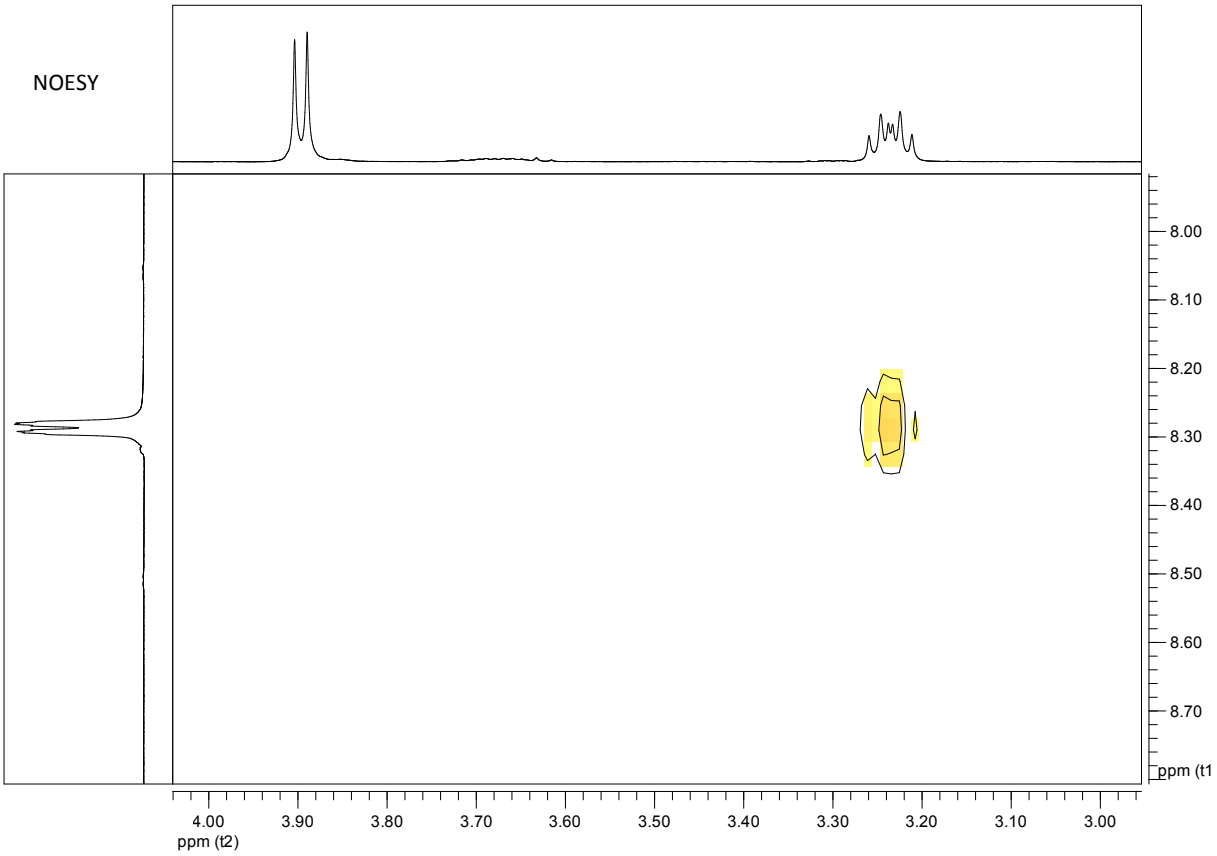
ν_{max}/cm^{-1}: 1709 (C=O), 1305 (CH), 1198 (C-N)

HRMS (ESI): found 314.2120, calculated 314.2106 for $C_{20}H_{28}NO_2$

3-pentyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (1)



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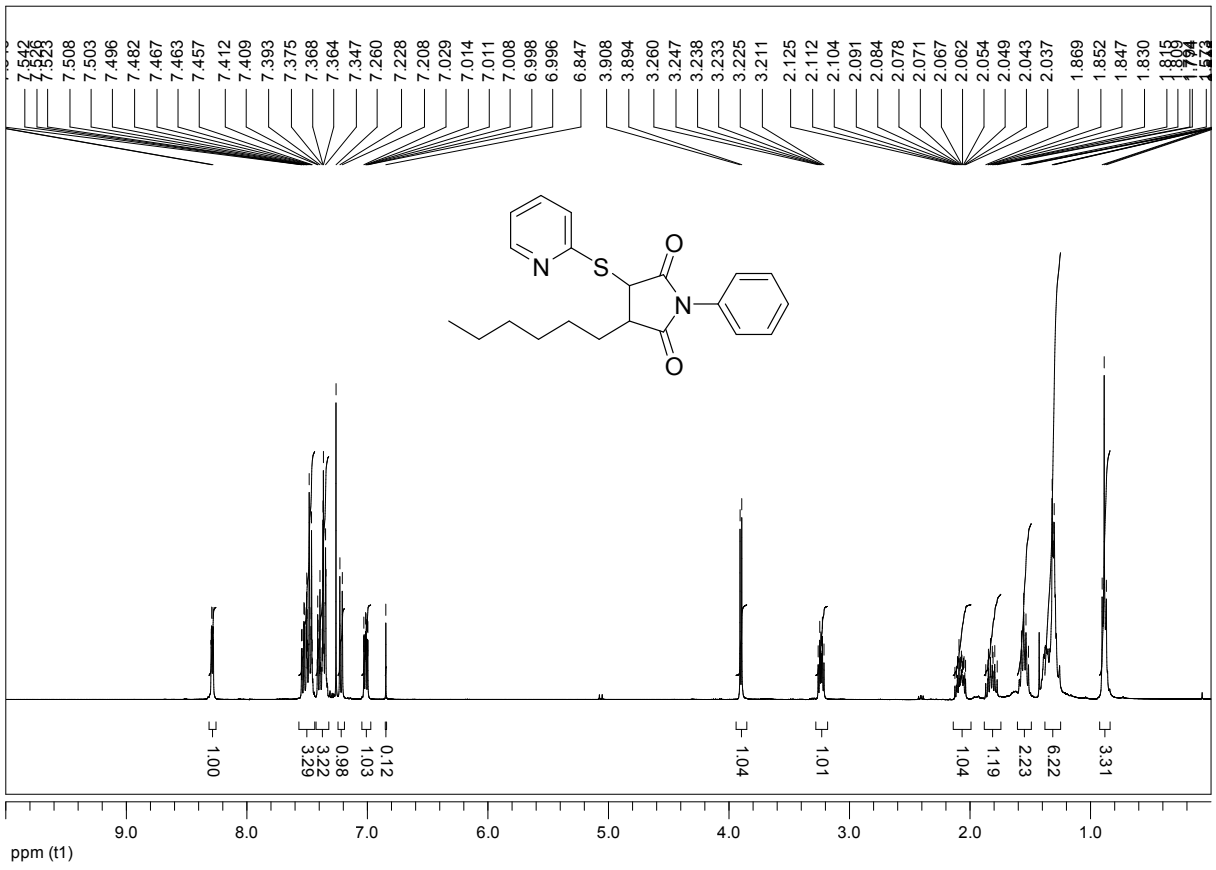
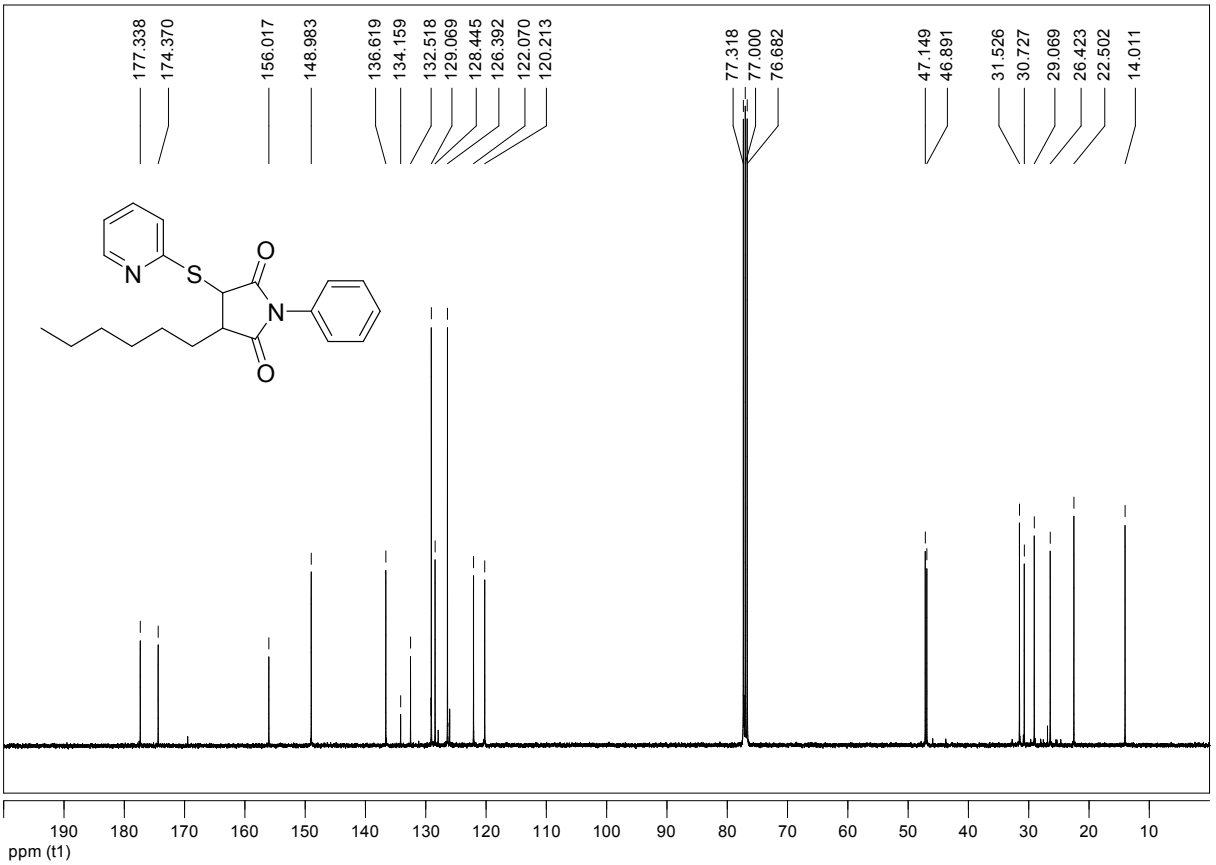
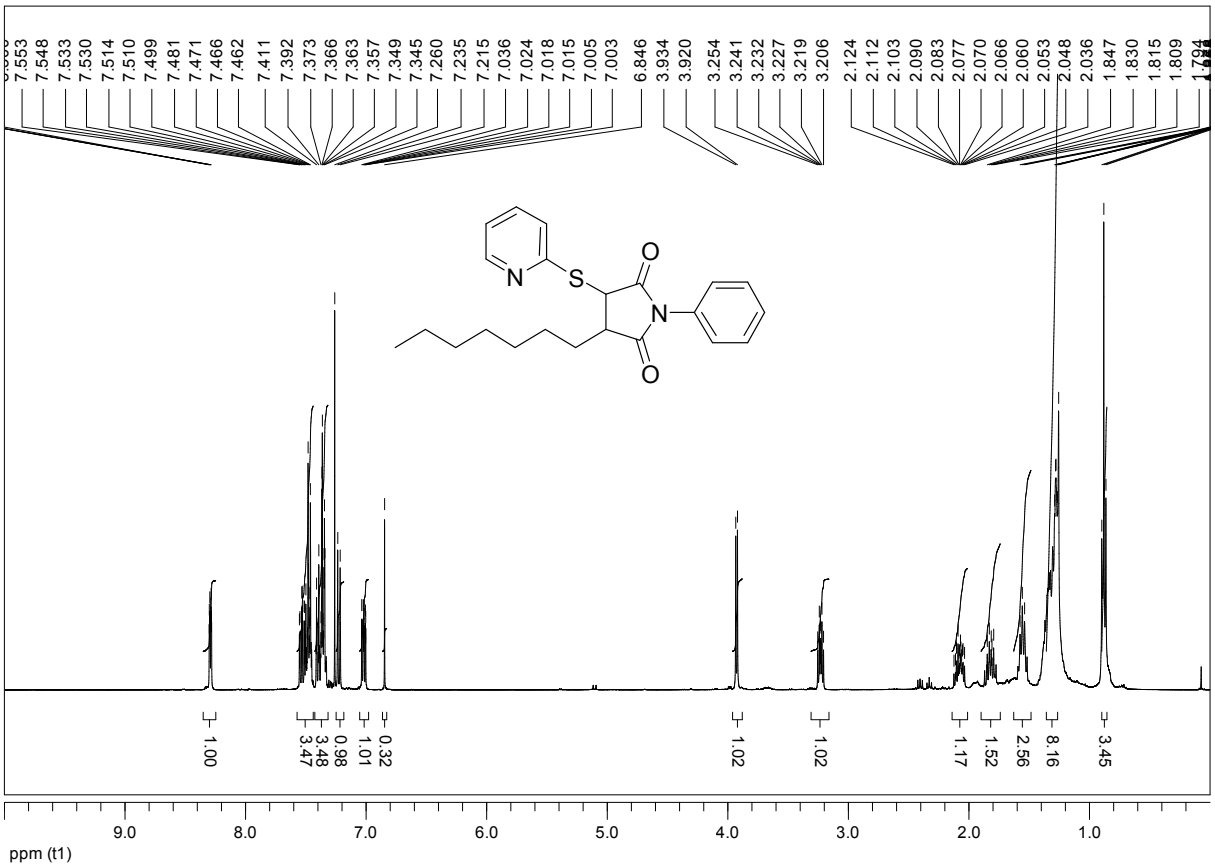
8.00
8.10
8.20
8.30
8.40
8.50
8.60
8.70
ppm (t1
4.00
3.90
3.80
3.70
3.60
3.50
3.40
3.30
3.20
3.10
3.00
ppm (t2)

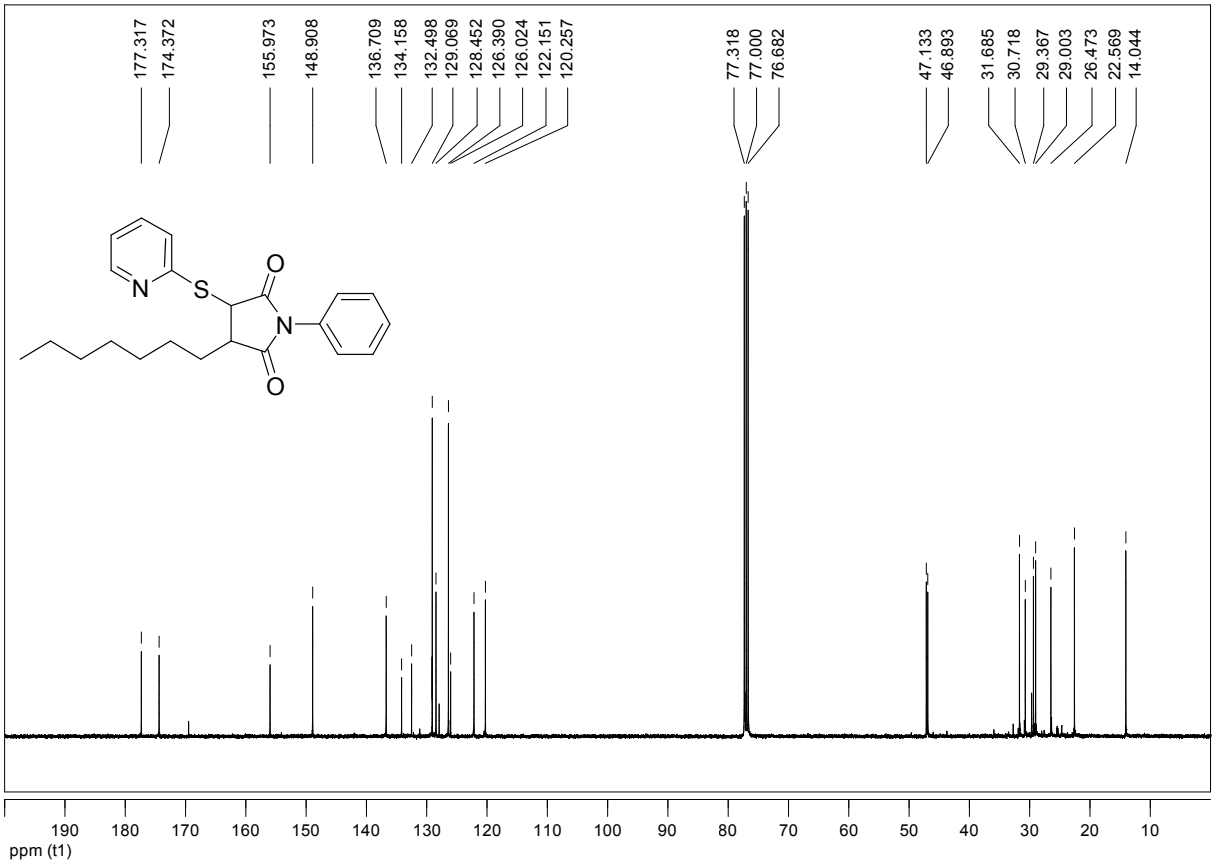
3-hexyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (2)



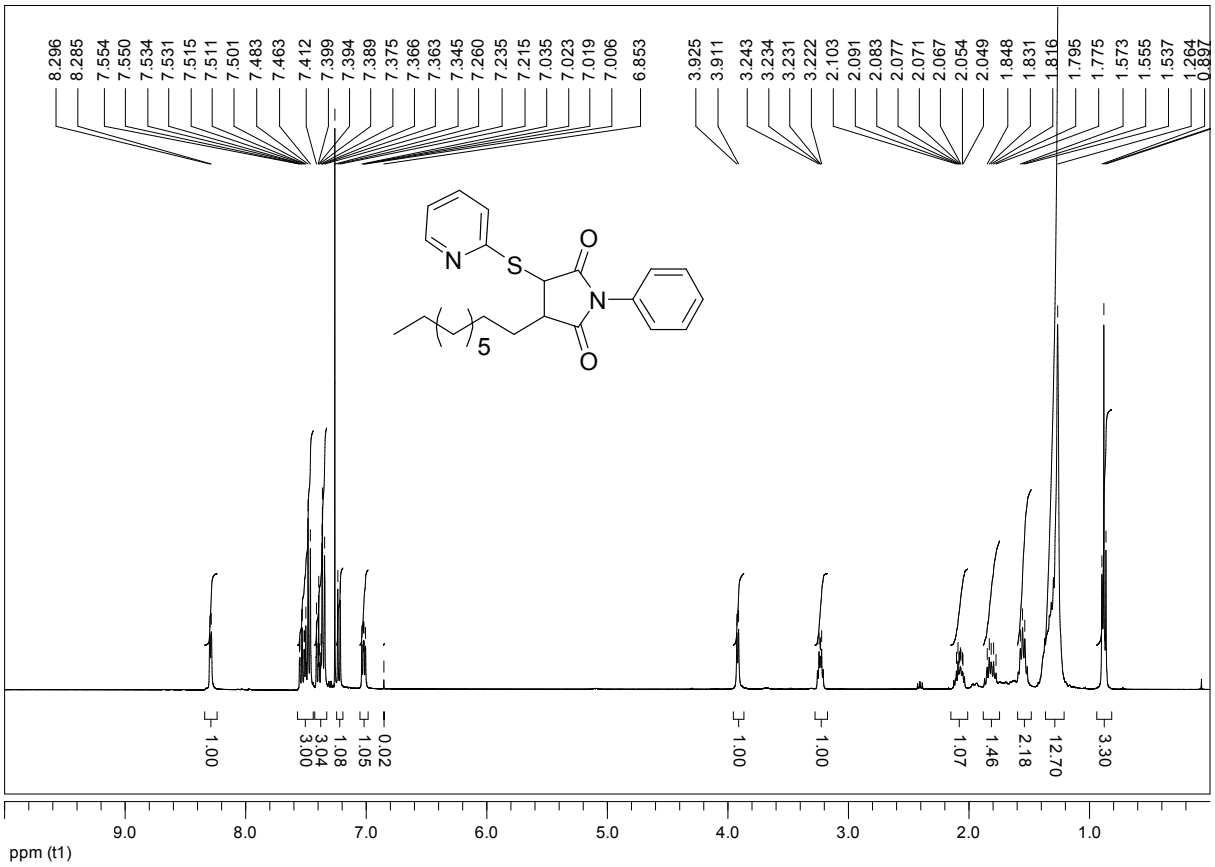


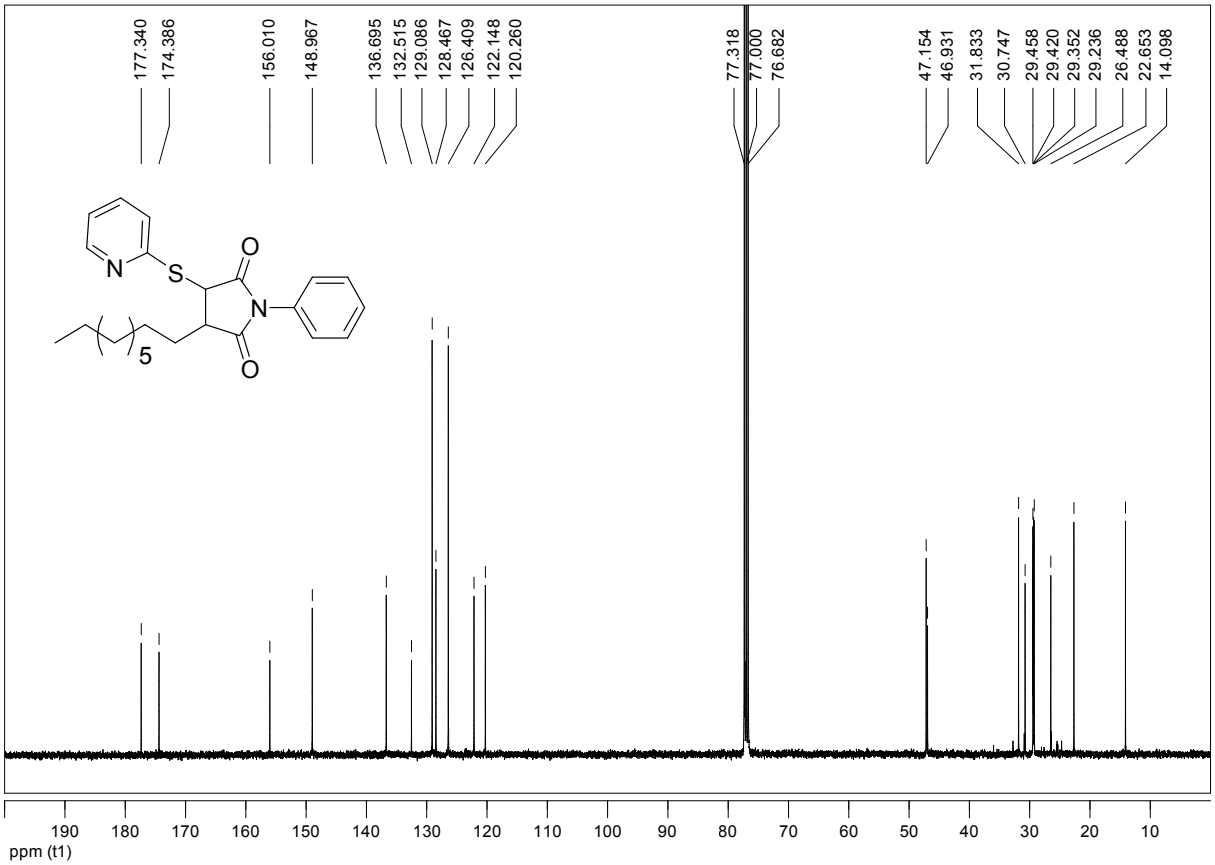
3-heptyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3)



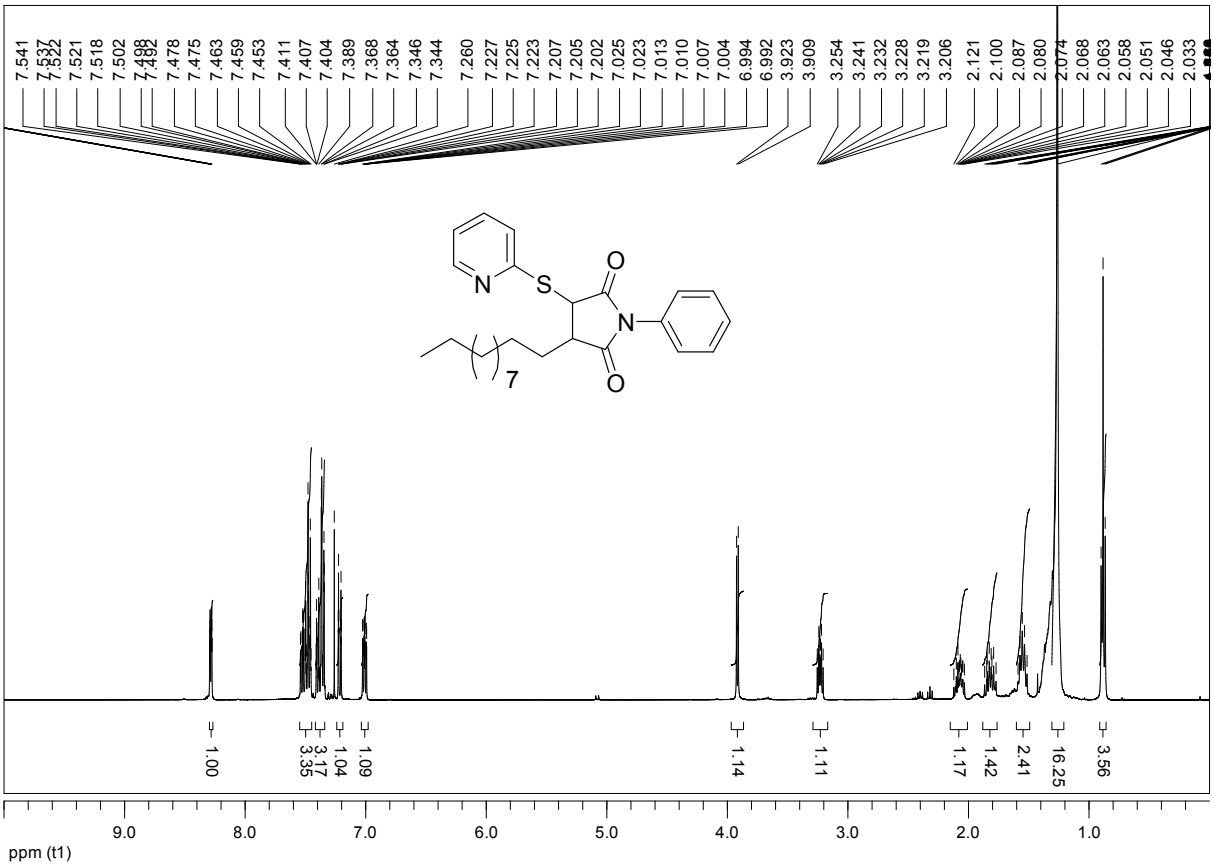


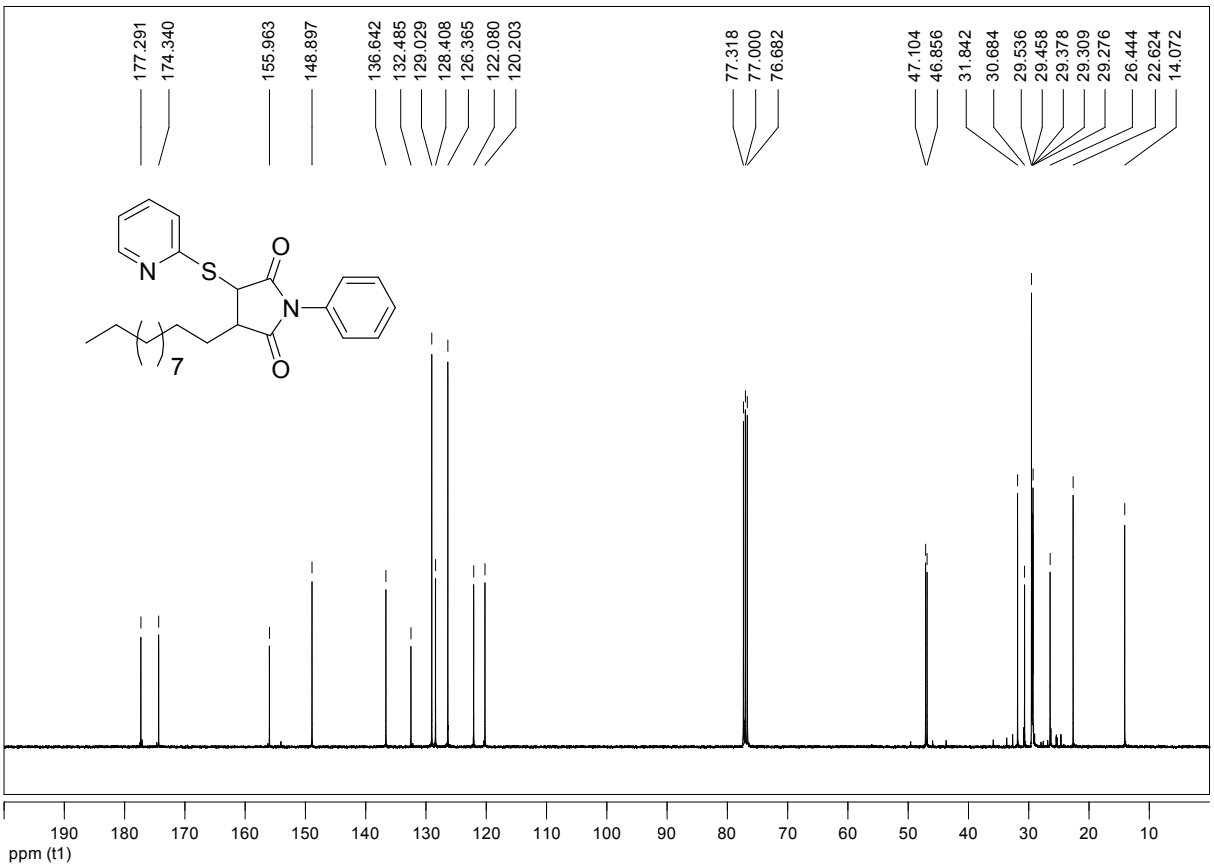
3-nonyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (4)



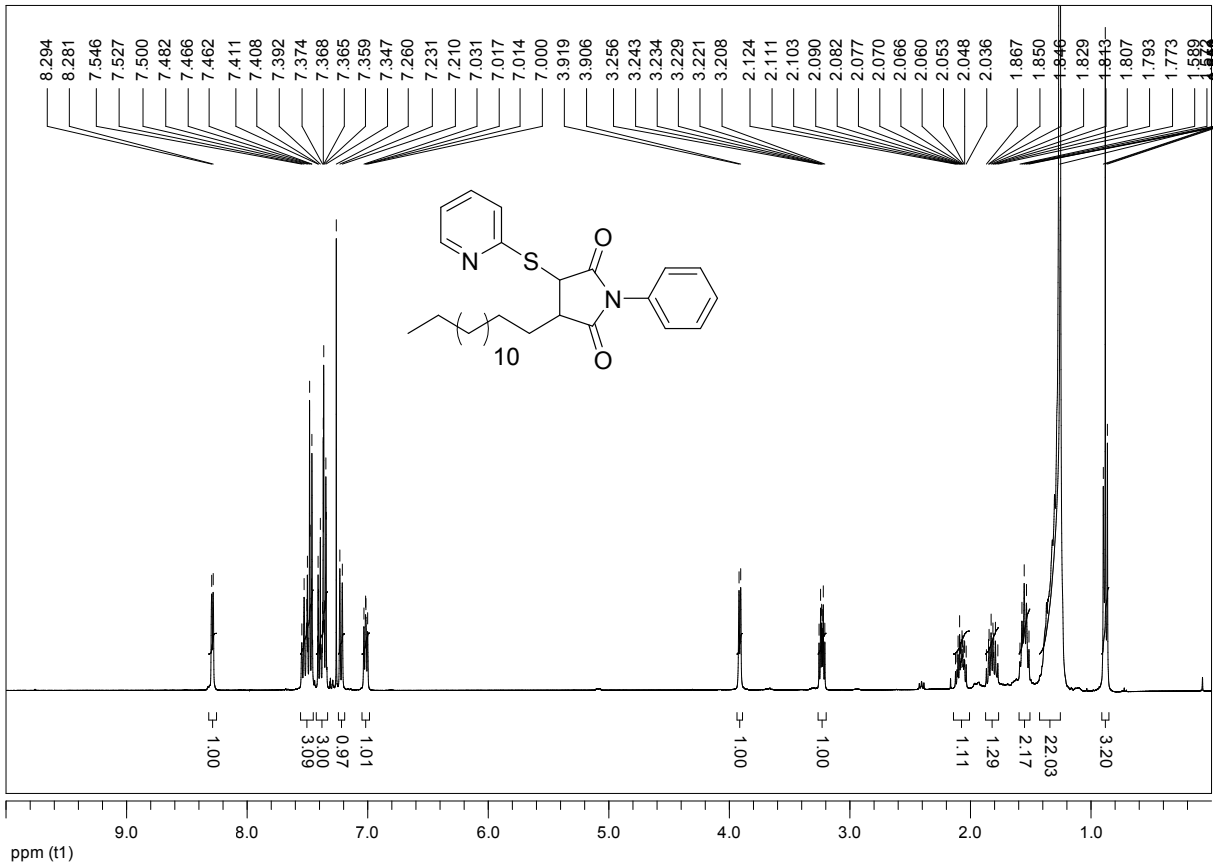


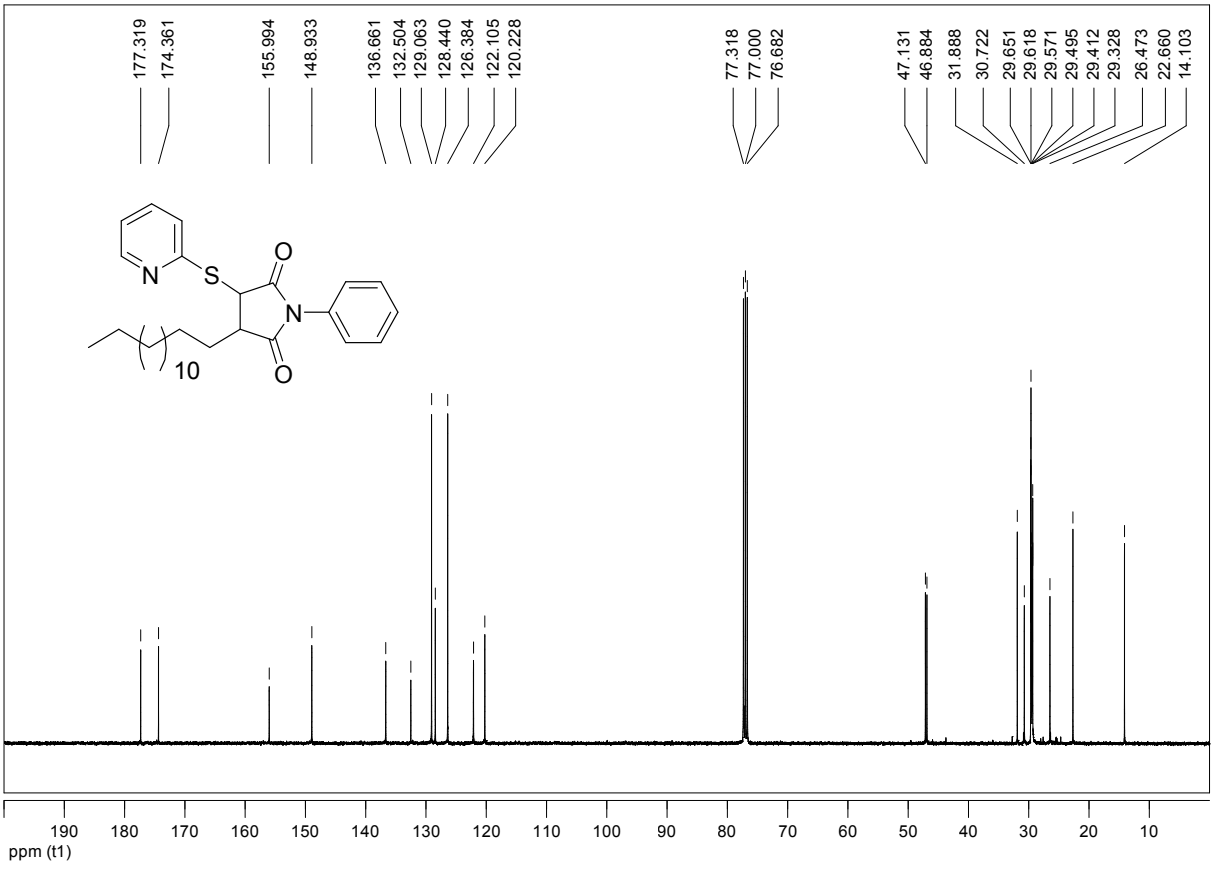
1-phenyl-3-(pyridin-2-ylthio)-4-undecylpyrrolidine-2,5-dione (5)



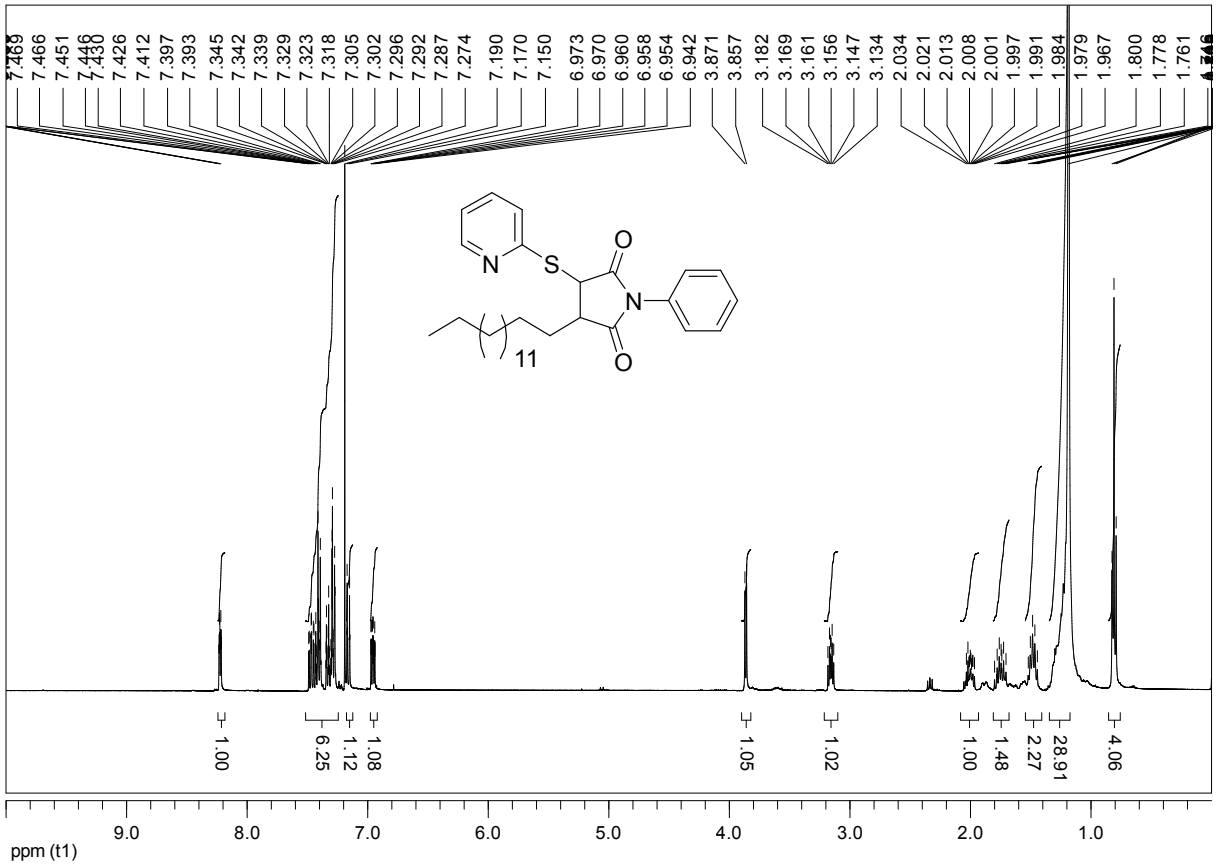


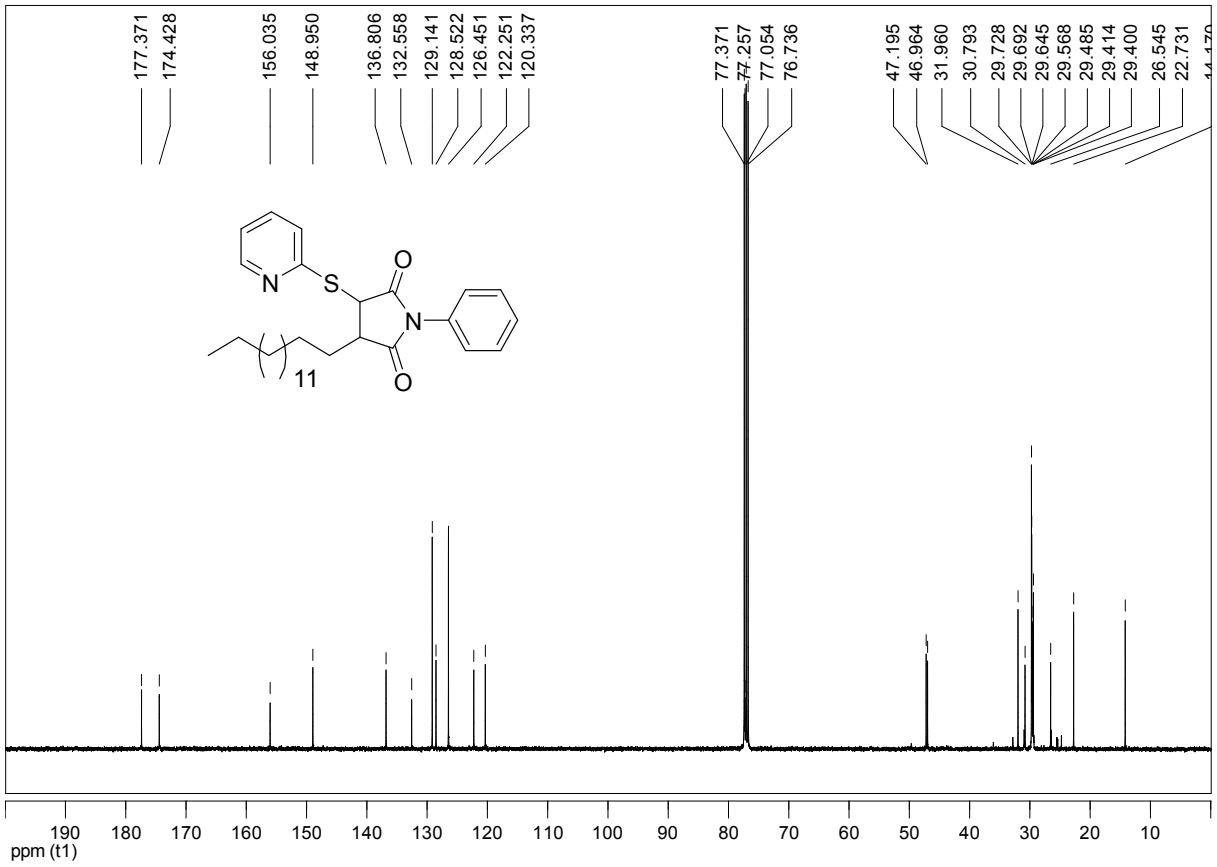
1-phenyl-3-(pyridin-2-ylthio)-4-tetradecylpyrrolidine-2,5-dione (6)



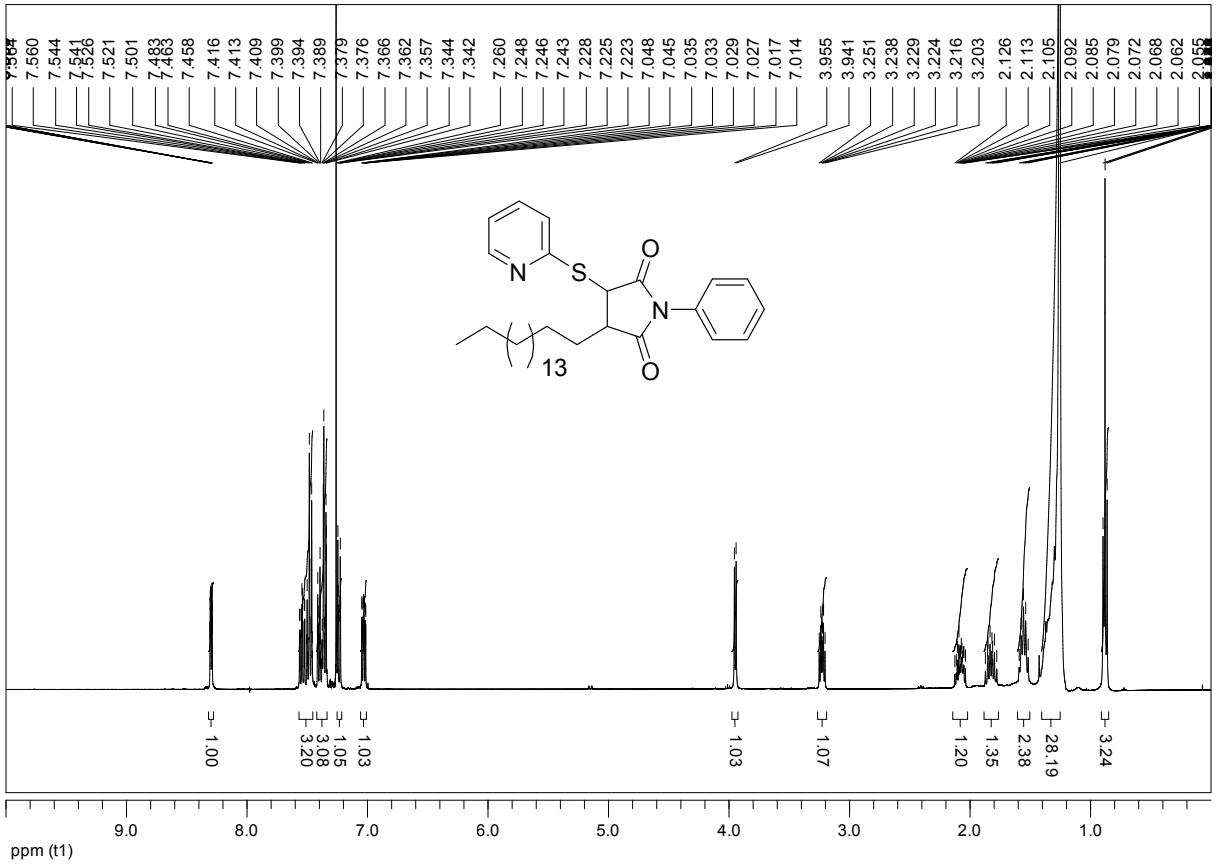

177.319
174.361
155.994
148.933
136.661
132.504
129.063
128.440
126.384
122.105
120.228
77.318
77.000
76.682
47.131
46.884
31.888
30.722
29.651
29.618
29.571
29.495
29.412
29.328
26.473
22.660
14.103
N
S
O
N
O
10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm (t1)

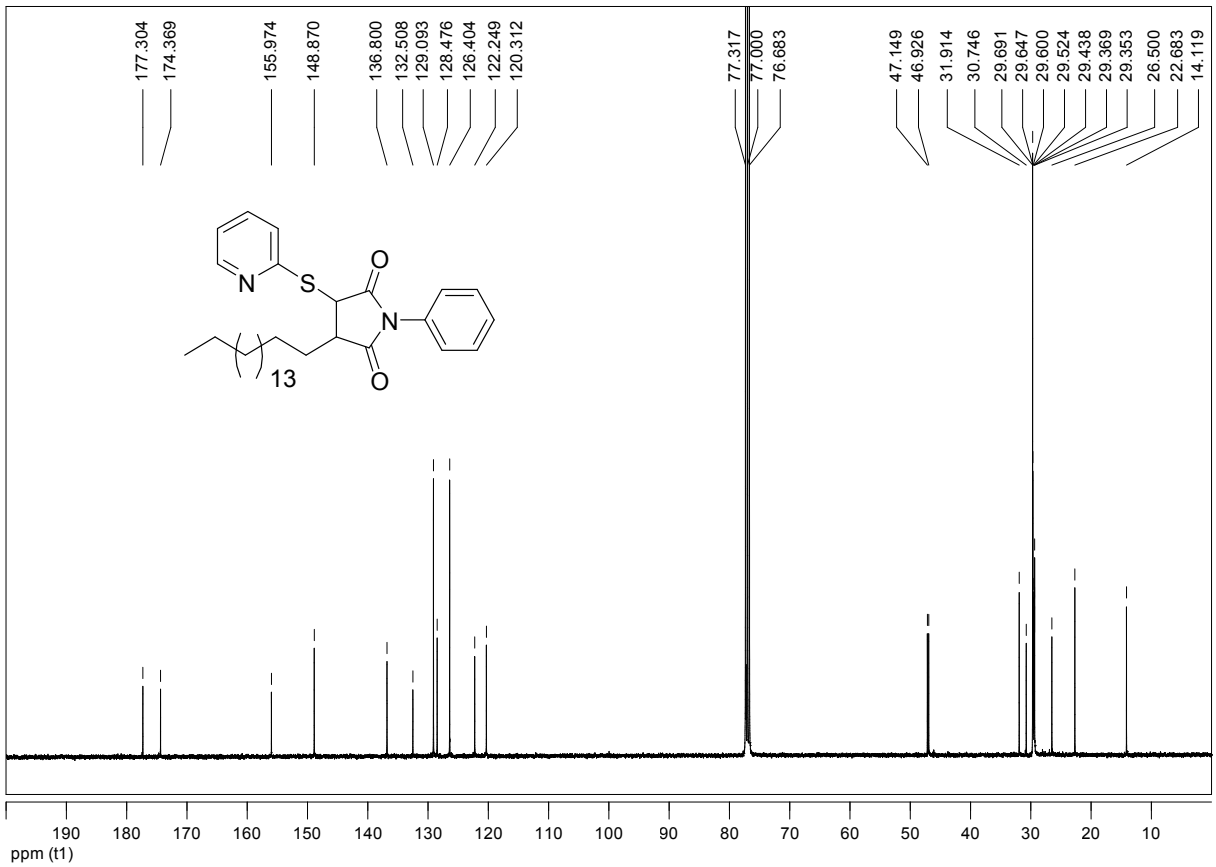
3-pentadecyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (7)



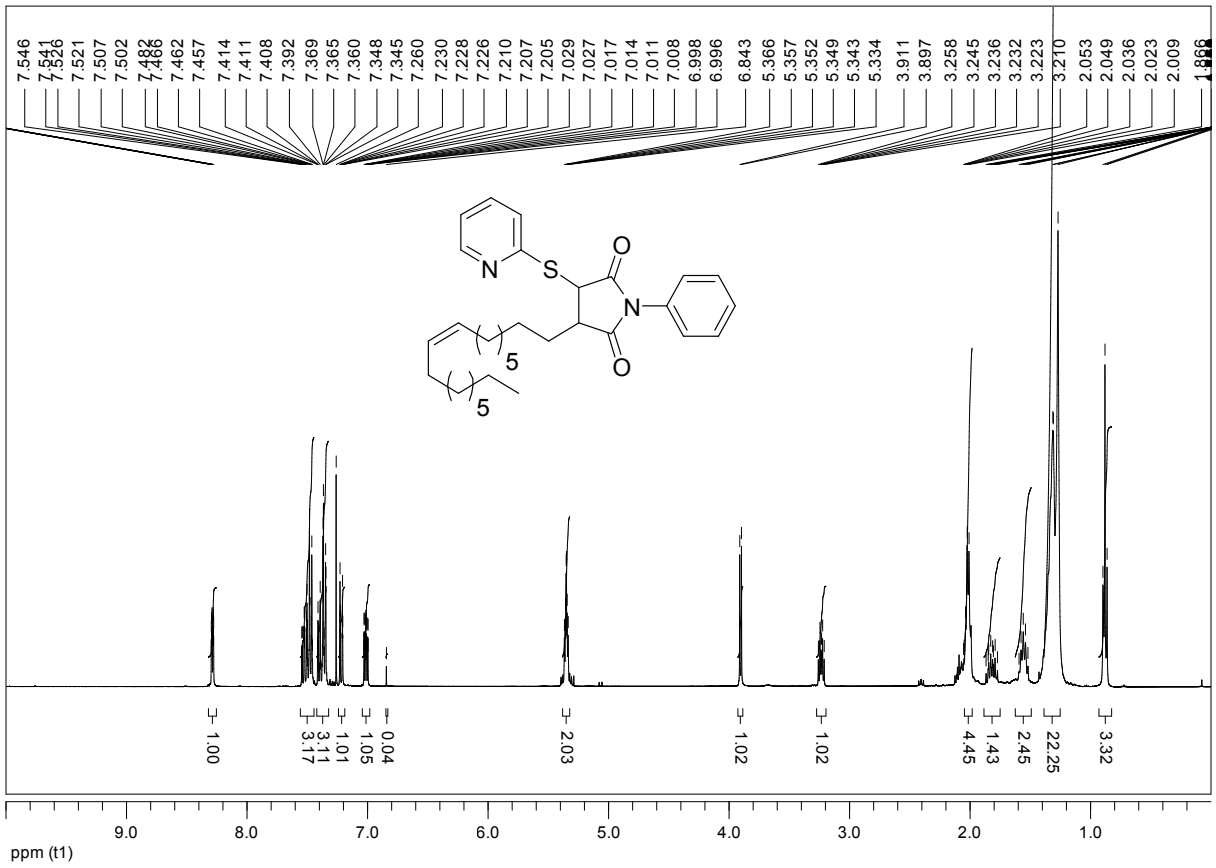


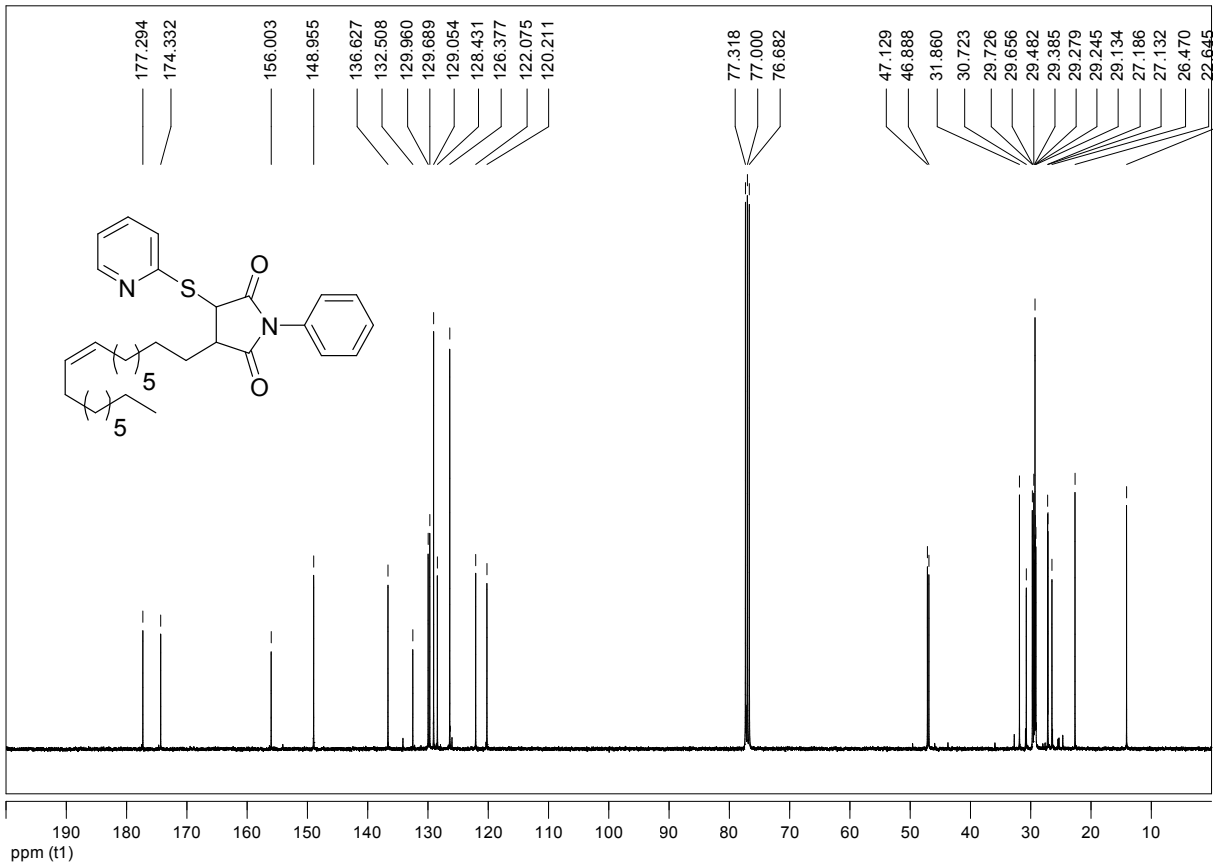
3-heptadecyl-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (8)



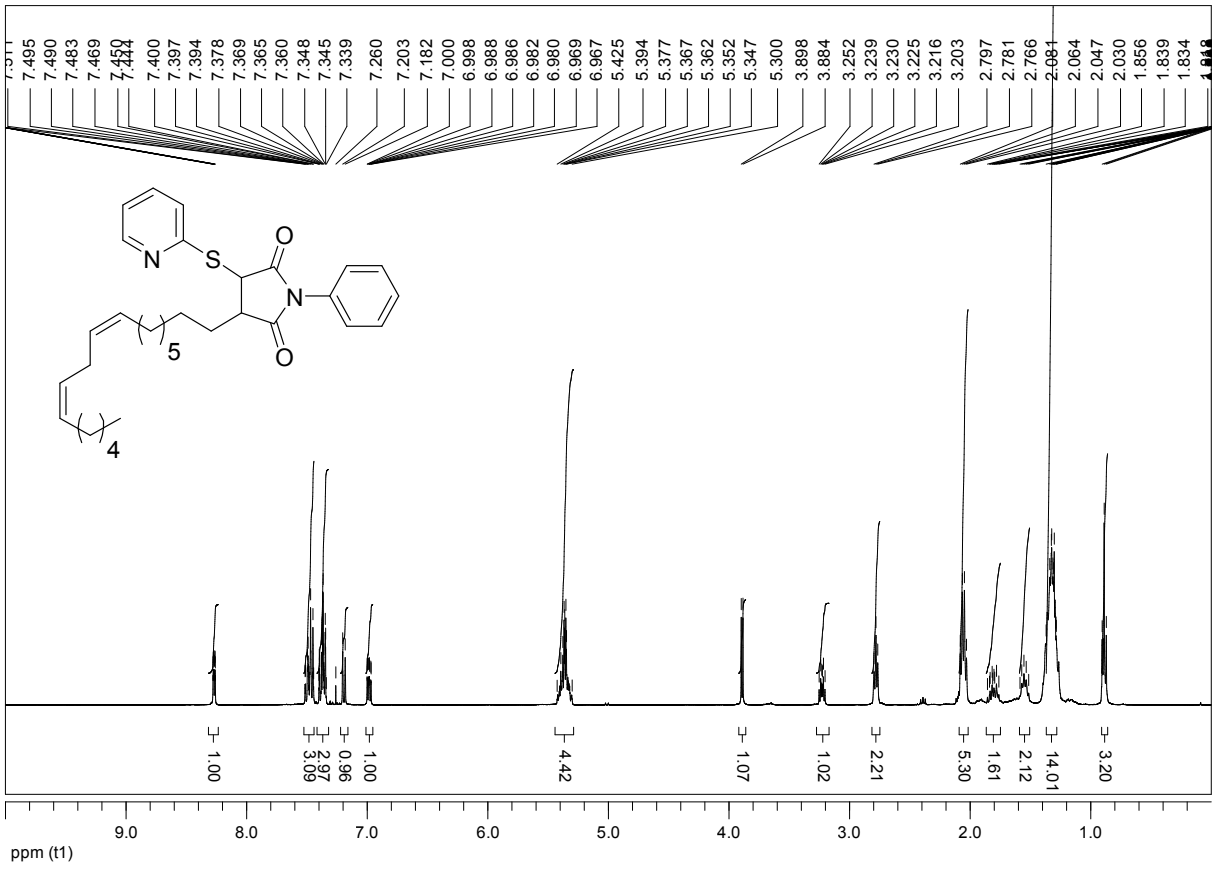


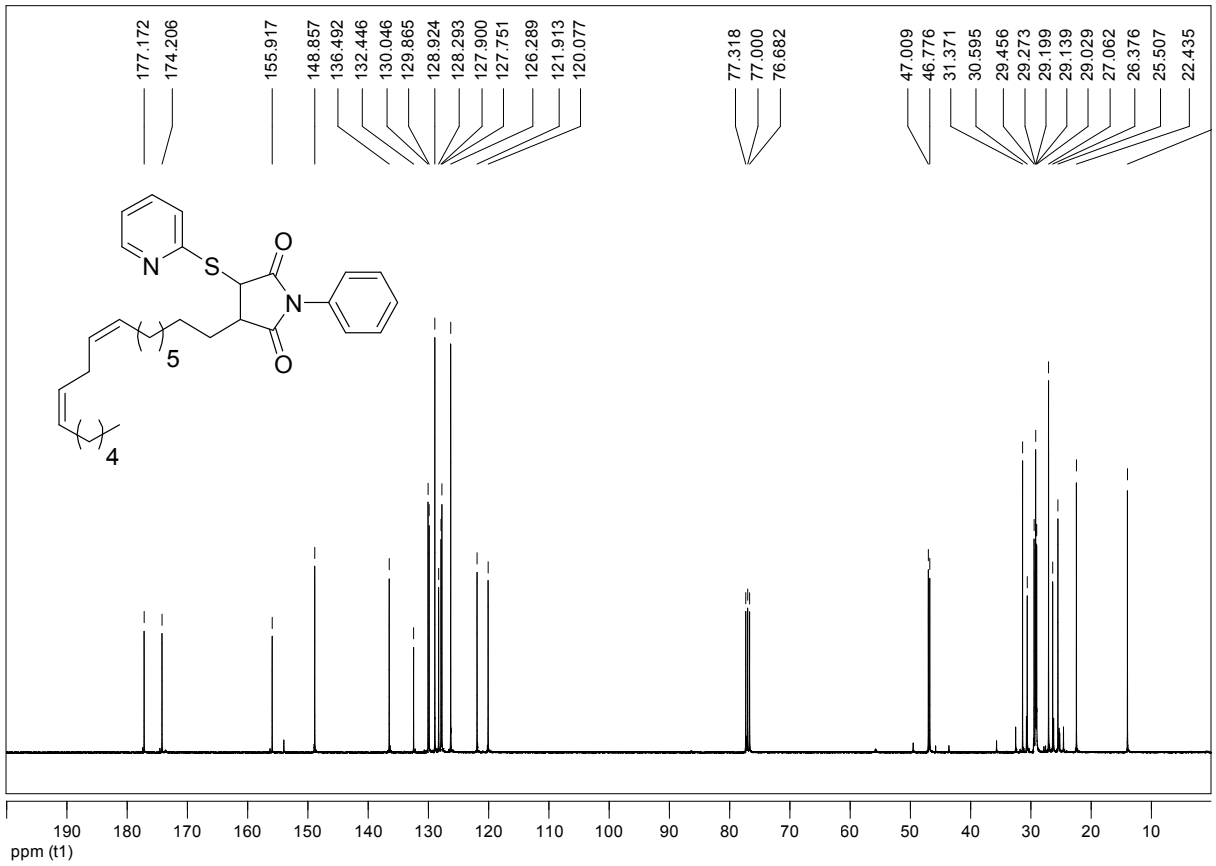
(Z)-3-(heptadec-8-en-1-yl)-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (9)



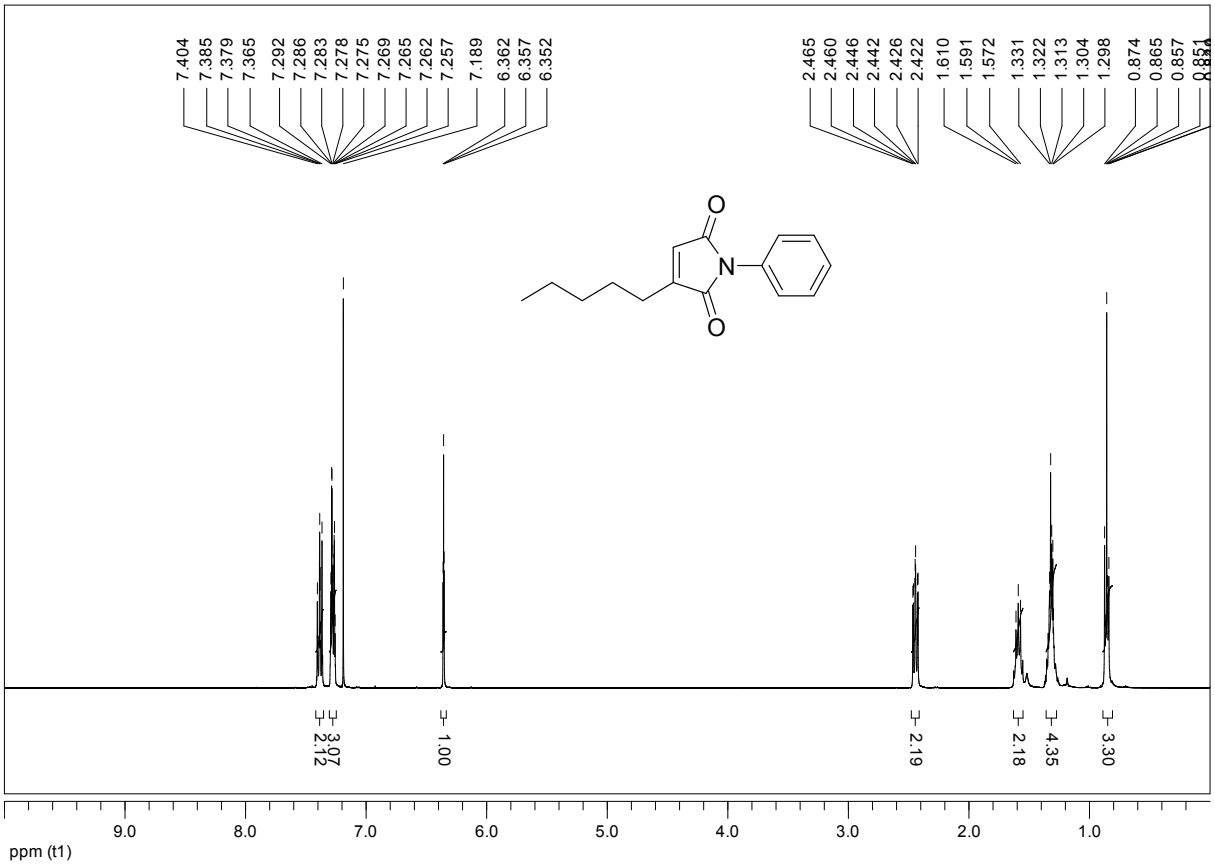


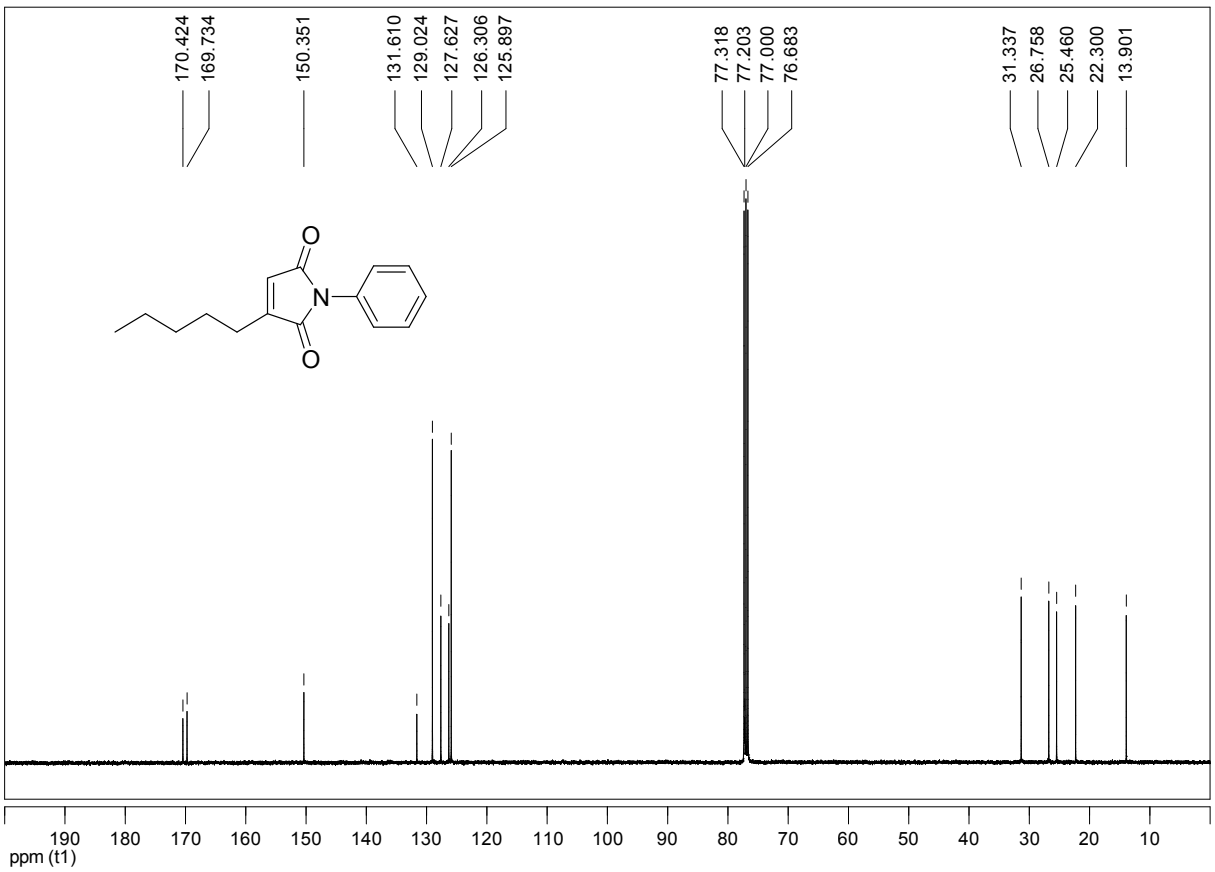
3-((8Z,11Z)-heptadeca-8,11-dien-1-yl)-1-phenyl-4-(pyridin-2-ylthio)pyrrolidine-2,5-dione (10)



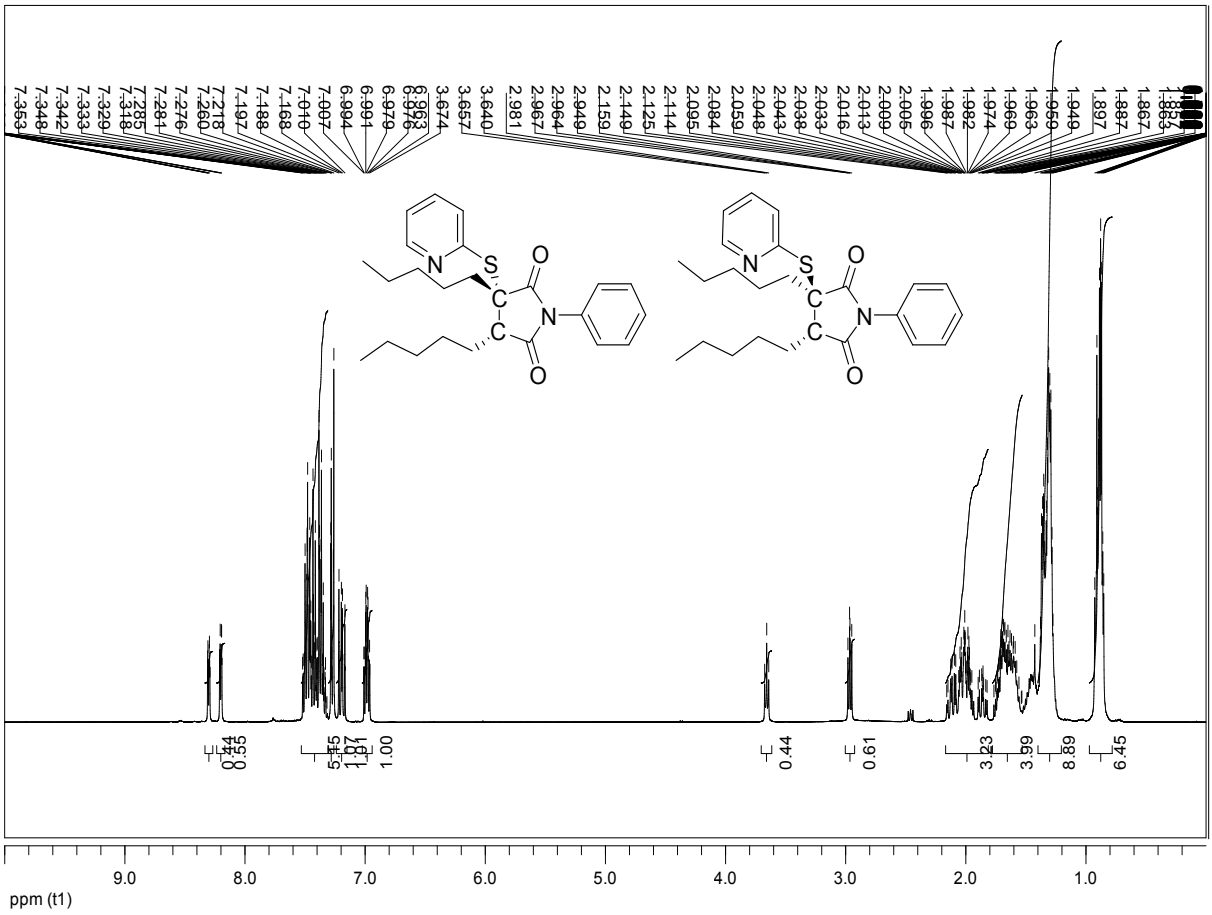


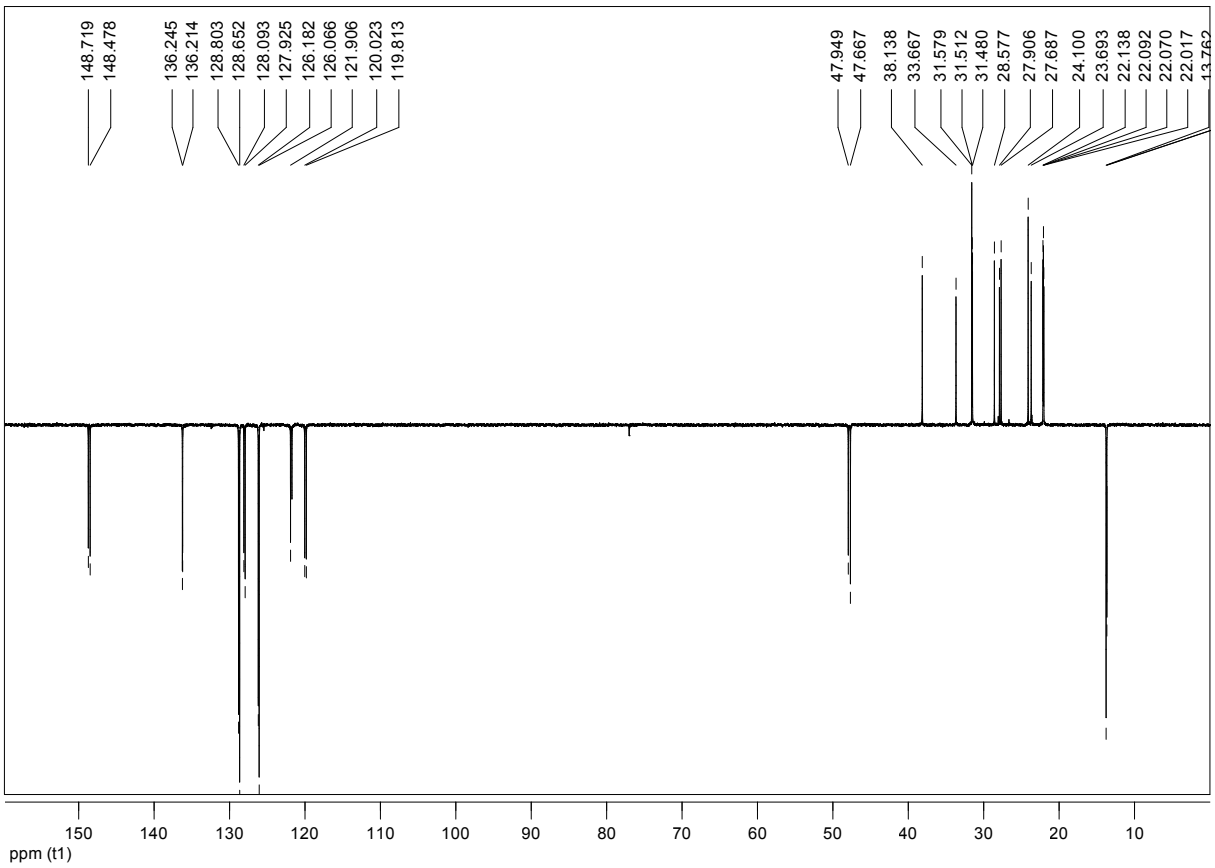
3-pentyl-1-phenyl-1*H*-pyrrole-2,5-dione (11)

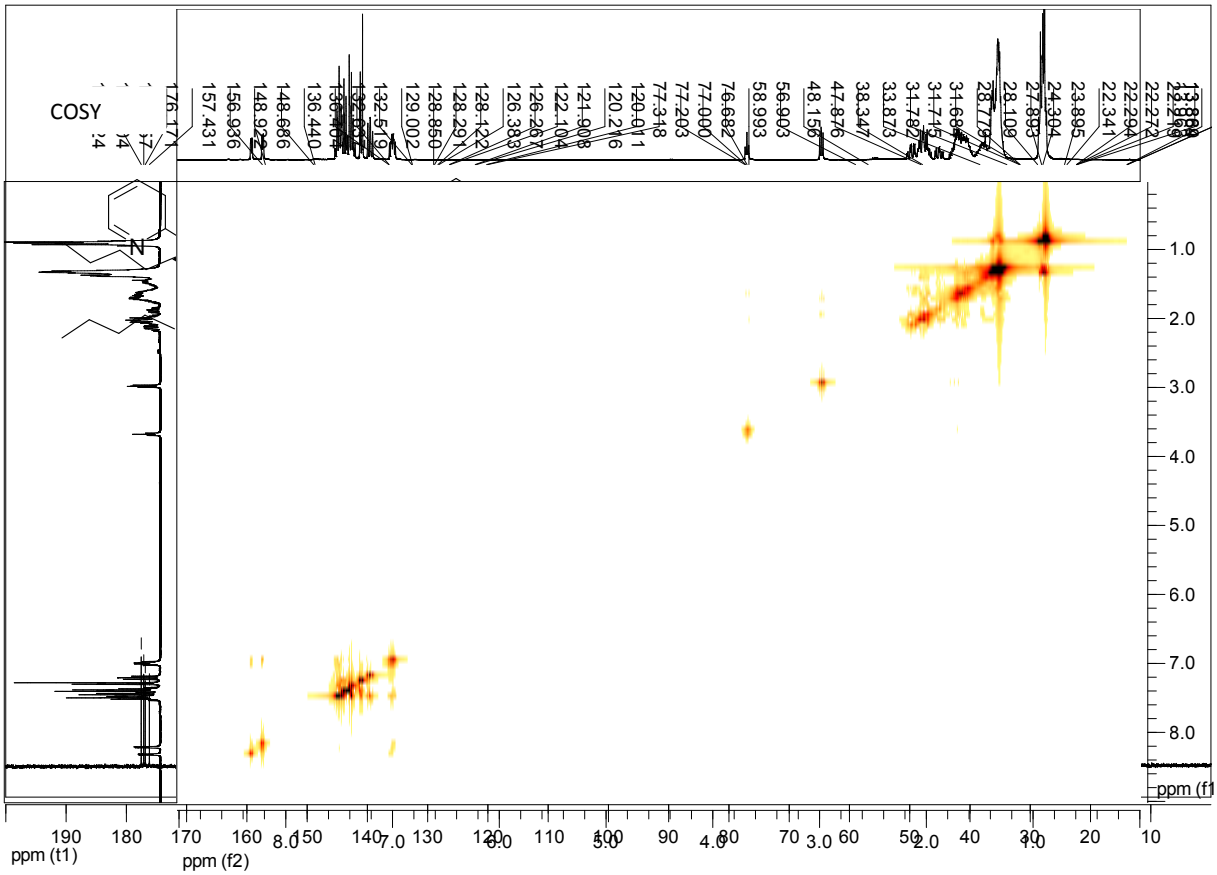




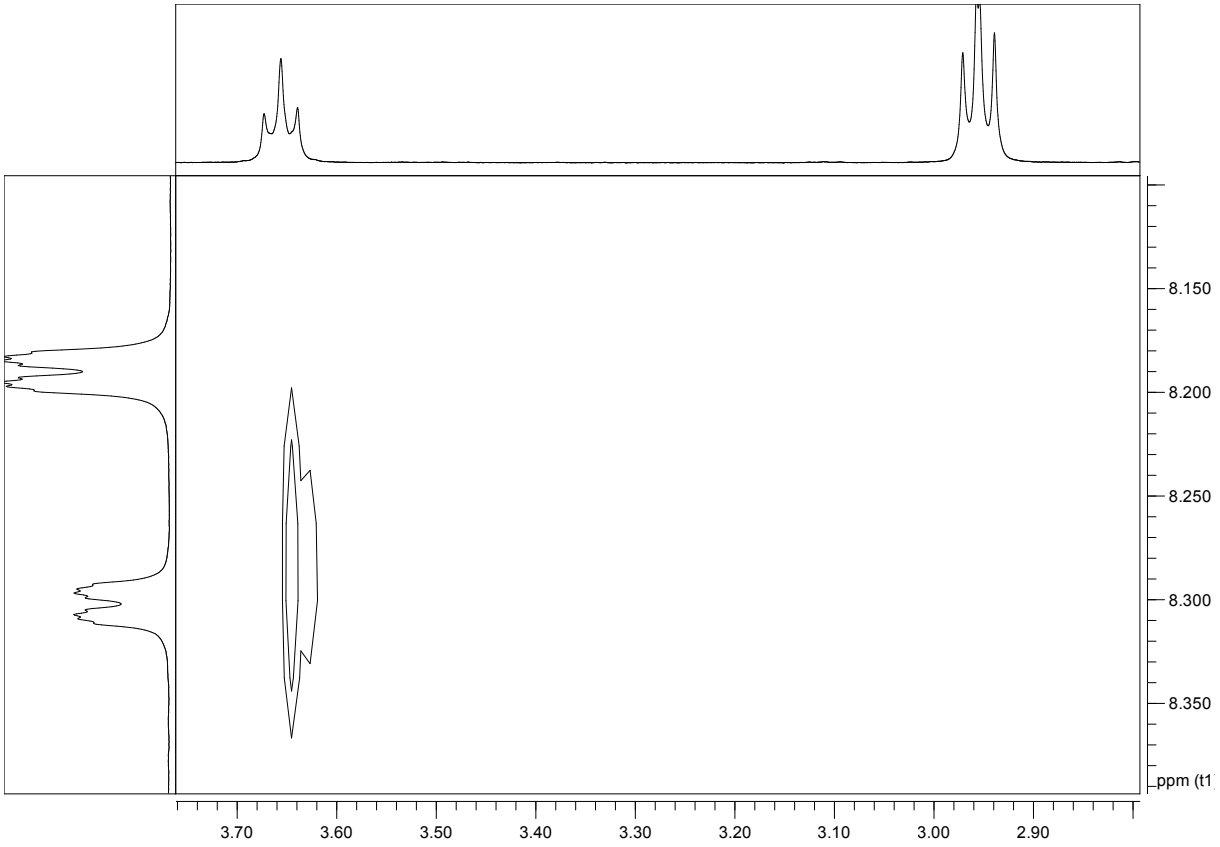
3,4-dipentyl-1-phenyl-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione (12a and 12b)



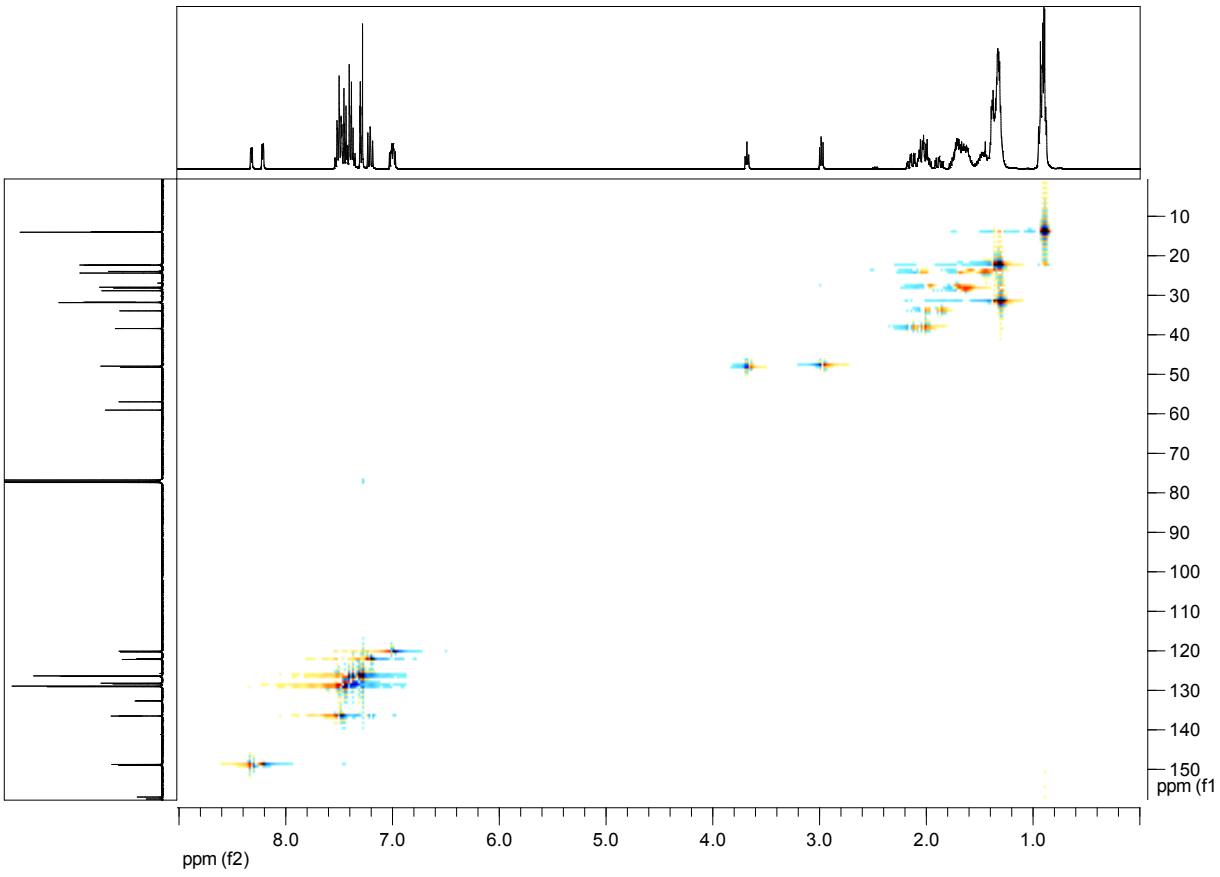

148.719
148.478
136.245
136.214
128.803
128.652
128.093
127.925
126.182
126.066
121.906
120.023
119.813
47.949
47.667
38.138
33.667
31.579
31.512
31.480
28.577
27.906
27.687
24.100
23.693
22.138
22.092
22.070
22.017
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm (t1)

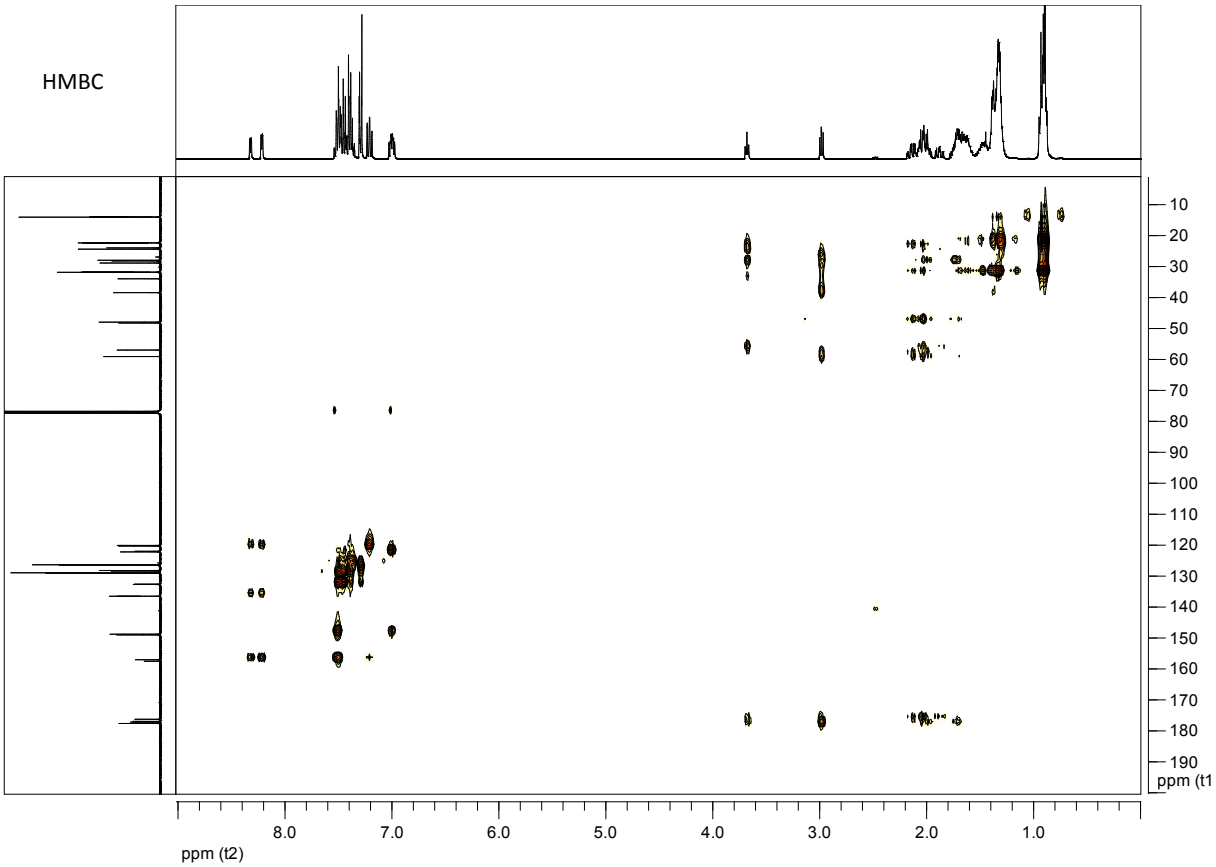

COSY
176.171
157.431
156.936
148.922
148.686
136.440
132.519
129.002
128.850
128.291
128.122
126.383
126.261
122.104
121.908
120.216
120.011
77.318
77.203
77.000
76.682
58.993
56.903
48.155
47.876
38.347
33.873
31.782
31.745
28.779
28.109
27.893
24.304
23.895
22.341
22.294
22.272
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
ppm (f1
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm (t1)
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
ppm (f2)

NOESY


8.150
8.200
8.250
8.300
8.350
ppm (t1
3.70
3.60
3.50
3.40
3.30
3.20
3.10
3.00
2.90

HSQC


10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
ppm (f1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f2)


HMBC
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
ppm (t1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t2)

3,4-dipentyl-1-phenyl-1*H*-pyrrole-2,5-dione (13)