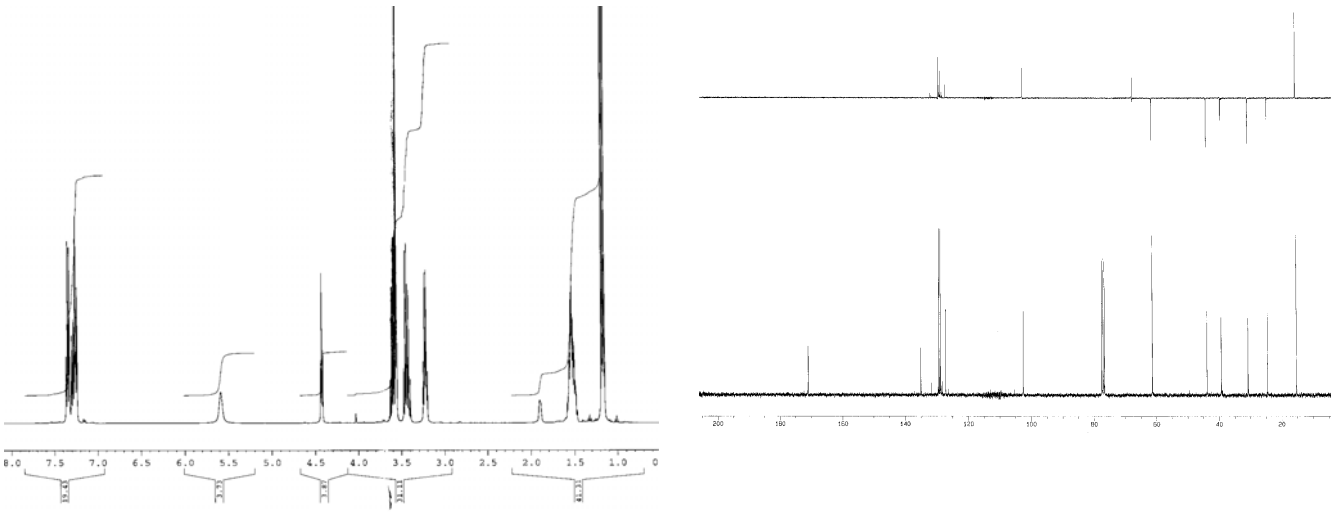
Supplementary information for :

An investigation into the electrophilic cyclisation of *N*-acyl -pyrrolidinium ions: a facile synthesis of pyrrolo-tetrahydroisoquinolones and pyrrolo-benzazepinones

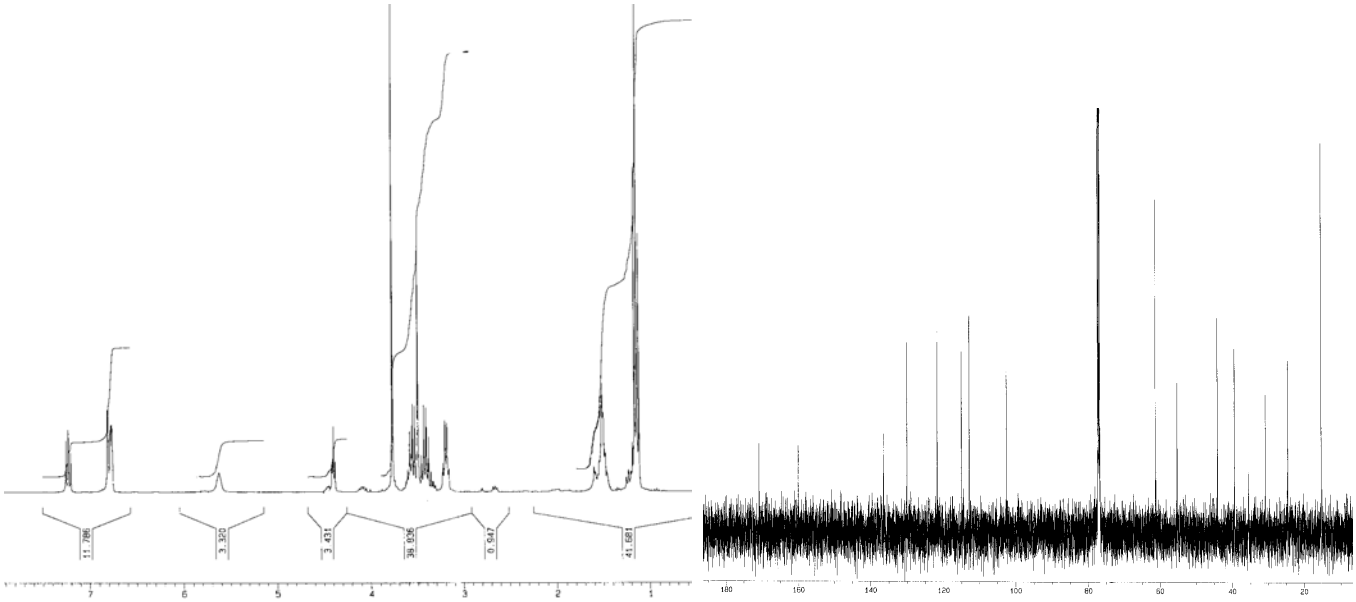
Frank D. King,* Abil A. Aliev, Stephen Caddick and Royston C. B. Copley

NMR of 2-(Phenyl)-N-(4,4-diethoxybutyl)acetamide (3):



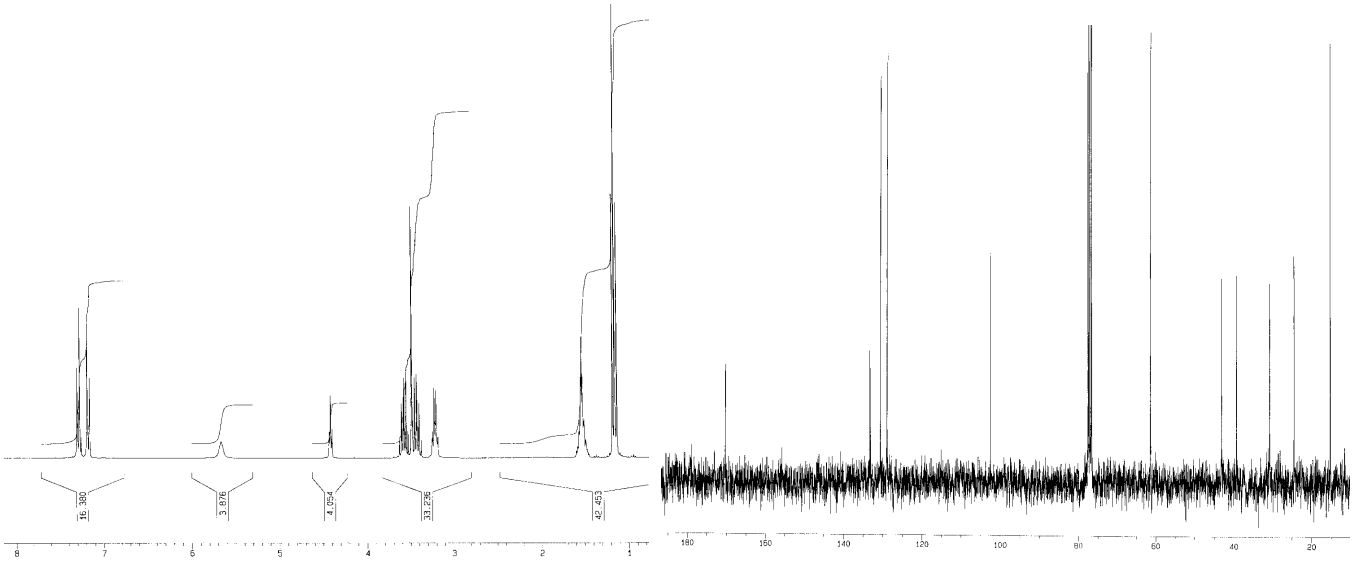
2-(3-Methoxyphenyl)-N-(4,4-diethoxybutyl)acetamide (3a):

Isolated as an oil (99% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, 6H), 1.56-1.65 (m, 4H), 3.20 (q, 2H, *J* = 7 Hz), 3.33–3.48 (m, 2H), 3.51 (s 2H), 3.53–3.60 (m, 2H), 3.78 (s, 3H), 4.41 (t, *J* = 5 Hz, 1H), 5.63 (brs, 1H), 6.74–6.81 (m, 2H), 7.23 (t, J = 7.5 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = δ = 15.3 (CH_3), 24.6 (CH_2), 30.8 (CH_2), 39.4 (CH_2), 44.1 (CH_2), 55.3 (CH_3), 61.3 (CH_2), 102.6 (CH), 112.9 (CH), 115.1 (CH), 121.8 (CH), 130.1 (CH), 136.5 (C), 160.1 (C), 170.9 (C);



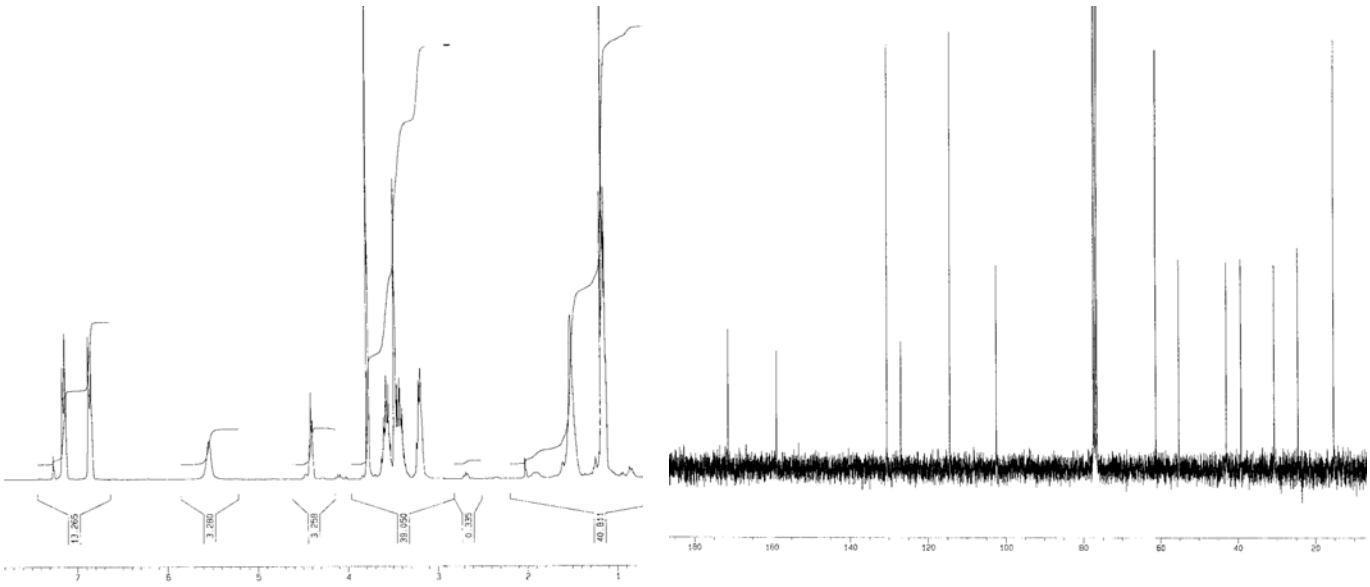
2-(4-Chlorophenyl)-N-(4,4-diethoxybutyl)acetamide (3c)

Isolated as a waxy solid (98% yield) m pt 62-4ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.48-1.60 (m, 4H), 3.24 (q, *J* = 7 Hz, 2H), 3.40 – 3.49 (m, 2H), 3.51 (s 2H), 3.54 – 3.65 (m, 2H), 4.43 (t, *J* = 5 Hz, 1H), 5.57 (brs, 1H), 7.20 (d, *J* = 8 Hz, 2H), 7.31 (d, *J* = 8 Hz, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.4 (CH_2), 30.8 (CH_2), 39.4 (CH_2), 43.1 (CH_2), 61.36 (CH_2), 102.5 (CH), 129.0 (CH), 130.7 (CH), 133.2 (C), 133.5 (C), 170.3 (C); ν_{max}/cm^{-1} 3293, 2977, 2929, 2870, 1638, 1548, 1491, 1152, 1126, 1110, 1088, 1054, 1016, 1000, 805, 728, 688.



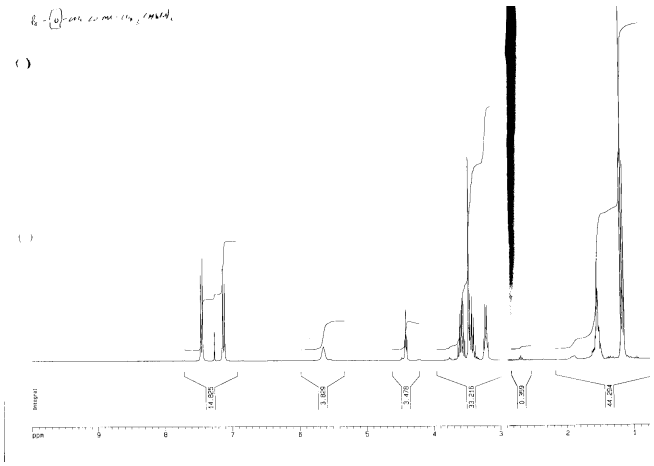
2-(4-Methoxyphenyl)-N-(4,4-diethoxybutyl)acetamide (3d):

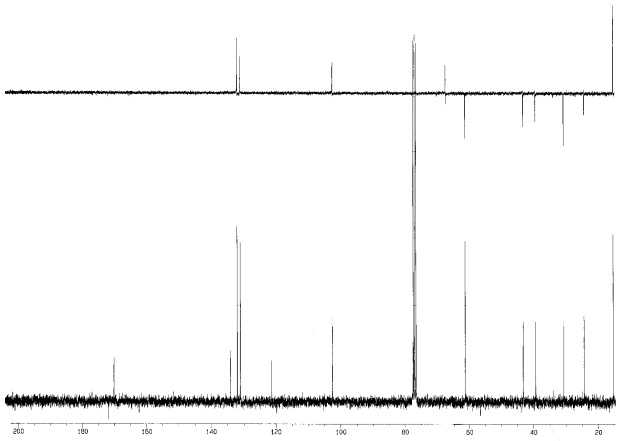
Isolated as a waxy solid (96% yield) mpt 32-34°C ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.56-1.65 (m, 4H), 3.24 (q, *J* = 7 Hz, 2H), 3.33–3.50 (m, 2H), 3.52 – 3.60 (m, 2H), 3.48 (s 2H), 3.79 (s, 3H), 4.41 (t, *J* = 5 Hz, 1H), 5.54 (brs, 1H), 6.86 (d, *J* = 7 Hz, 2H), 7.14 (d, *J* = 7 Hz, 2H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 43.0 (CH_2), 55.3 (CH_3), 61.3 (CH_2), 102.5 (CH), 114.4 (CH), 127.0 (C), 130.5 (CH), 158.8 (C), 171.4 (C), ν_{max}/cm^{-1} 2973, 2931, 1640, 1556, 1512, 1246, 1125, 1101, 1059, 1031, 1006, 817, 790, 720; HRMS: Theoretical Mass (M^+ - OEt): 264.15997, Measured Mass: 264.15999.



2-(4-Bromophenyl)-N-(4,4-diethoxybutyl)acetamide (3e)

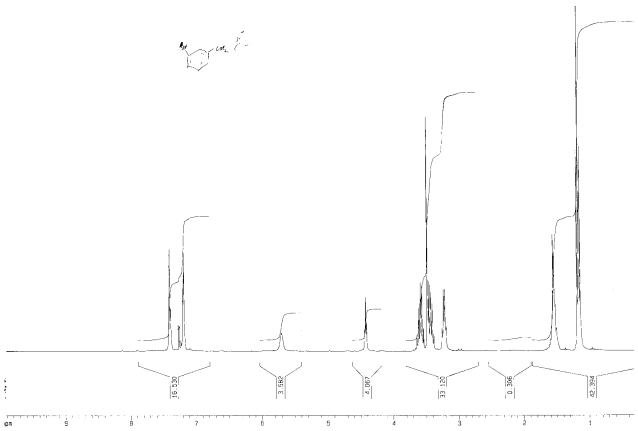
Isolated as a waxy solid (95% yield) mpt 66-67°C. NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.48-1.60 (m, 4H), 3.24 (q, *J* = 7 Hz, 2H), 3.40–3.49 (m, 2H), 3.47 (s 2H), 3.54–3.65 (m, 2H), 4.42 (t, *J* = 5 Hz, 1H), 5.64 (brs, 1H), 7.12 (d, *J* = 8 Hz, 2H), 7.44 (d, *J* = 8 Hz, 2H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.4 (CH_2), 30.8 (CH_2), 39.4 (CH_2), 43.2 (CH_2), 61.4 (CH_2), 102.5 (CH), 121.3 (C), 131.1 (CH), 132.0 (CH), 134.0 (C), 170.2 (C)

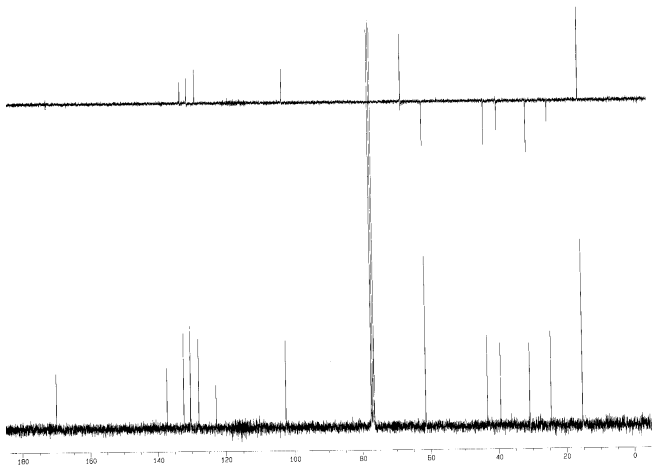




2-(3-Bromophenyl)-N-(4,4-diethoxybutyl)acetamide (3f)

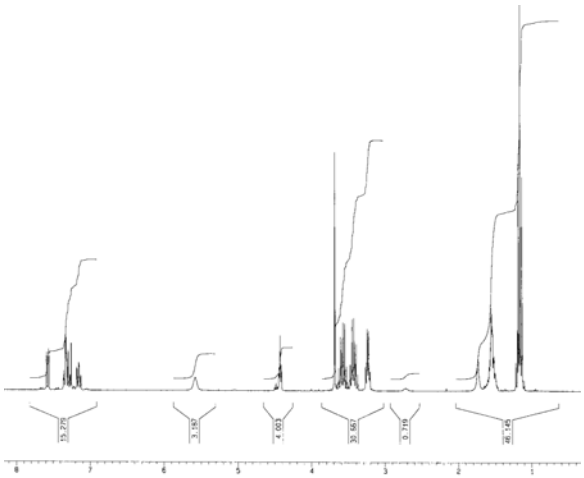
Isolated as a waxy solid (95% yield) m pt = 36-8°C: ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7Hz, 6H), 1.46-1.63 (m, 4H), 3.24 (q, *J* = 7 Hz, 2H), 3.30–3.43 (m, 2H), 3.47–3.59 (m, 2H), 3.68 (s 2H), 4.43 (t, *J* = 5 Hz, 1H), 5.73 (brs, 1H), 7.15-7.22 (m, 2H), 7.36–7.45 (m, 2H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.4 (CH_2), 30.8 (CH_2), 39.4 (CH_2), 43.3 (CH_2), 61.4 (CH_2), 102.5 (CH), 122.9 (C), 128.0 (CH), 130.4 (CH), 132.4 (CH), 137.3 (C), 170.0 (C).

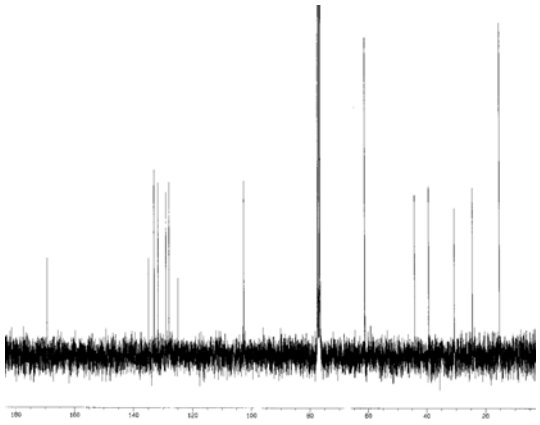




2-(2-Bromophenyl)-N-(4,4-diethoxybutyl)acetamide (3h)

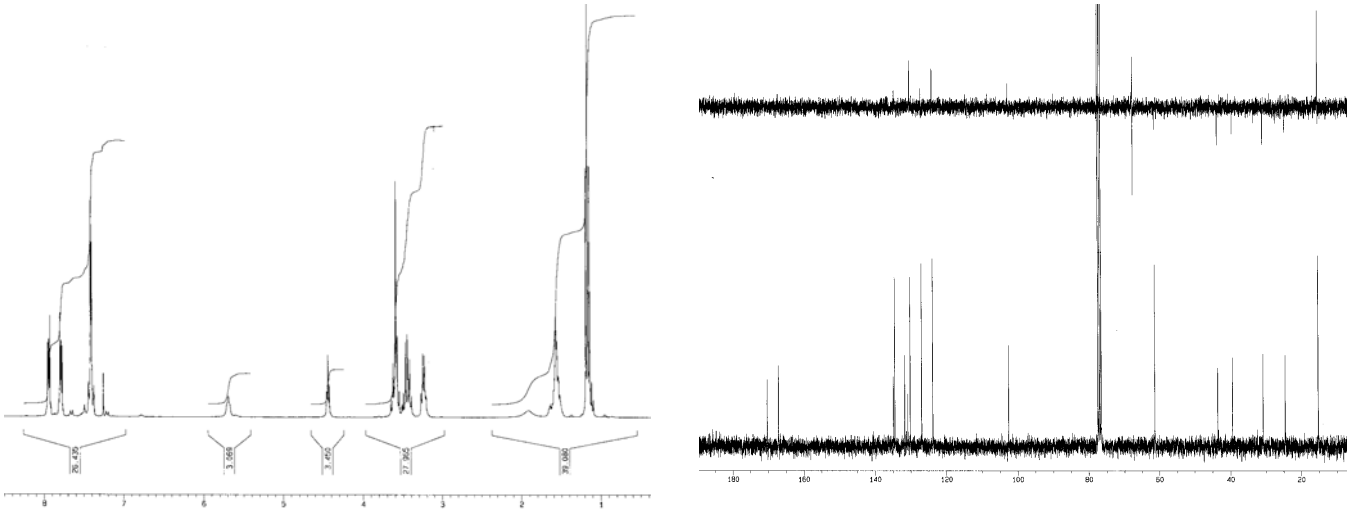
Isolated as white solid (100% yield), mpt 75-6°C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, J = 7 Hz, 6H), 1.46-1.65 (m, 4H), 3.22 (q, *J* = 7 Hz, 2H), 3.36–3.52 (m, 4H including 3.49 s, 2H), 3.53–3.66 (m, 2H), 4.43 (t, *J* = 5 Hz, 1H), 5.70 (brs, 1H), 7.15 (dt, *J* = 8, 2Hz, 1H), 7.25–7.36 (m, 2H), 7.58 (d, *J* = 8 Hz, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 44.1 (CH_2), 61.3 (CH_2), 102.5 (CH), 125.0 (C), 128.0 (CH), 129.1 (C), 131.7 (CH), 133.1 (CH), 135.0 (C), 169.4 (C).





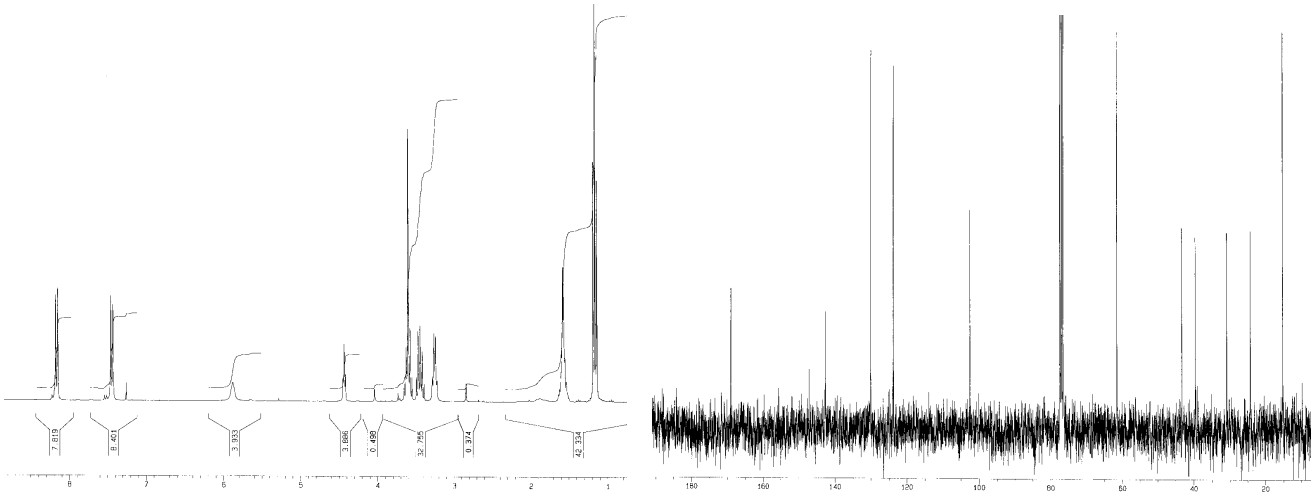
N-(4,4-Diethoxybutyl)-2-[4-(1,3-dioxo-1,3-dihydroisoindoly-2-yl)-phenyl]acetamide (3i)

Isolated as a solid (73% yield) m.pt. 169-72°C (Et_2O/petrol); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7 Hz, 6H), 1.46–1.70 (m, 4H), 3.24 (q, J = 6.5 Hz, 1H), 3.36–3.53 (m, 2H), 3.56–3.66 (m, 3H, including 3.58, s), 4.44 (t, *J* = 5 Hz, 1H), 5.69 (brs, 1H), 7.41 (d, *J* = 3.5 Hz, 2H), 7.44 (d, *J* = 3.5 Hz, 2H), 7.76-7.85 (m, 2H), 7.90-7.99 (m, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.6 (CH_2), 30.9 (CH_2), 39.5 (CH_2), 43.5 (CH_2), 61.3 (CH_2), 102.6 (CH), 123.8 (CH), 127.0 (CH), 130.2 (CH), 130.9 (C), 131.7 (C), 134.5 (C), 134.9 (C), 167.2 (C), 170.3 (C).



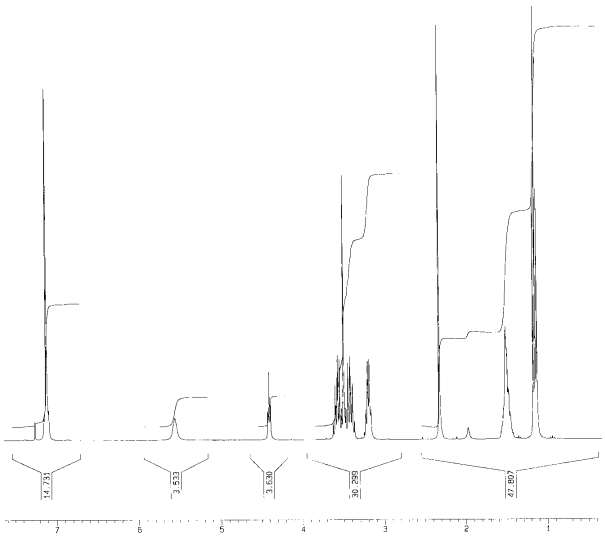
2-(4-Nitrophenyl)-N-(4,4-diethoxybutyl)acetamide (3j)

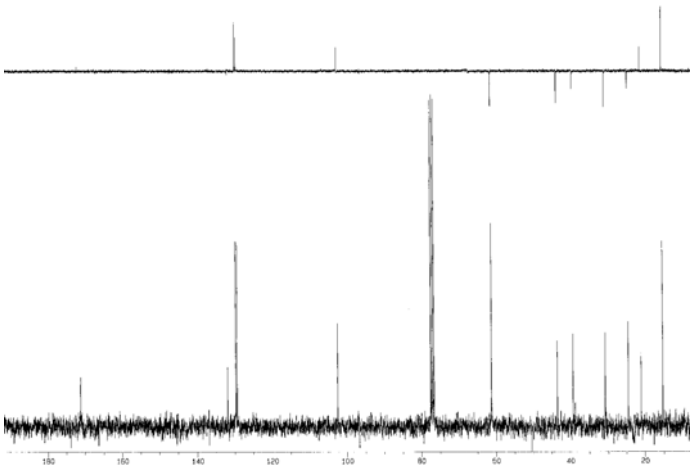
Isolated as a solid (98% yield) m.pt. 69-72°C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.50–1.65 (m, 4H), 3.27 (q, *J* = 6.8Hz, 2H), 3.40–3.52 (m, 2H), 3.55- 3.78 (m, 4H), 4.43 (t, *J* = 5 Hz, 1H), 5.64 (brs, 1H), 7.44 (d, *J* = 9 Hz, 2H), 8.21 (d, *J* = 9 Hz, 2H), ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.3 (CH_2), 30.9 (CH_2), 39.6 (CH_2), 43.3 (CH_2), 61.6 (CH_2), 102.6 (CH), 123.9 (CH), 130.2 (CH), 142.6 (C), 169.1 (C); ν_{max}/cm^{-1} 3291, 2935, 1639, 1605, 1558, 1510, 1344, 1132, 1111, 1049, 911, 856, 744, 720, 679, 666.



2-(4-Methylphenyl)-N-(4,4-diethoxybutyl)acetamide (3k)

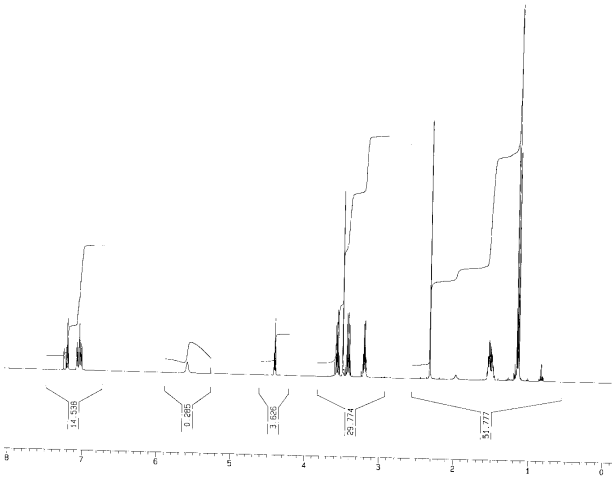
Isolated as white solid (98% yield) m pt 53-5°C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7 Hz, 6H), 1.40–1.58 (m, 4H), 2.32 (s, 3H), 3.19 (q, *J* = 6.8Hz, 2H), 3.34–3.62 (m, 6H including 3.50, s, 2H), 4.41 (t, *J* = 5 Hz, 1H), 5.56 (brs, 1H), 7.12 (s, 4H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 21.1 (CH_3), 24.5 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 43.5 (CH_2), 61.3 (CH_2), 102.5 (CH), 129.3 (CH), 129.7 (CH), 131.9 (C), 137.0 (C), 171.2 (C)

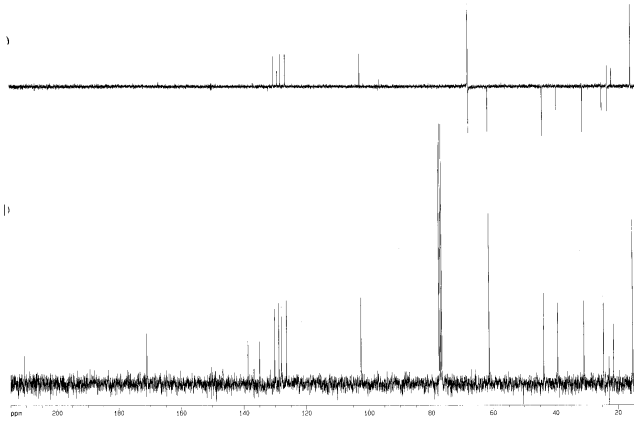




2-(3-Methylphenyl)-N-(4,4-diethoxybutyl)acetamide (3l)

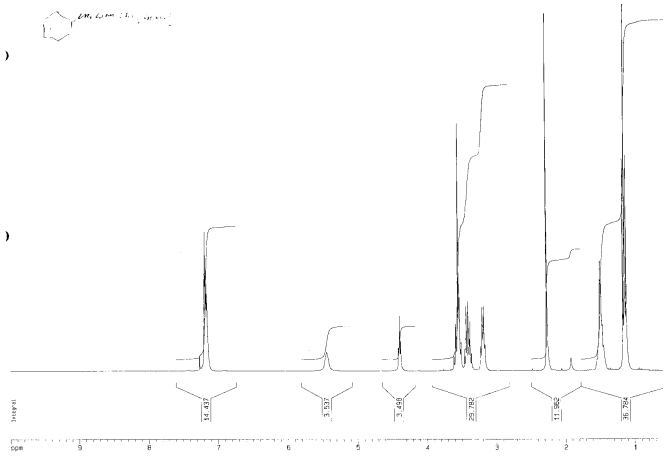
Isolated as an oil (97% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7 Hz, 3H), 1.45 – 1.57 (m, 4H), 2.33 (s, 3H), 3.20 (q, *J* = 7Hz, 2H), 3.38 – 3.48 (m, 2H), 3.50 (s, 2H), 3.55 – 3.65 (m, 2H), 4.41 (t, *J* = 5 Hz, 1H), 5.59 (brs, 1H), 7.00–7.10 (m, 3H), 7.21 (dt, *J* = 7.5, 3.5 Hz, 1H). ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 21.3 (CH_3), 24.5 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 43.8 (CH_2), 61.3 (CH_2), 102.5 (CH), 126.4 (CH), 128.0 (CH), 128.9 (CH), 130.2 (CH), 134.9 (C), 138.7(C), 171.0 (C); ν_{max}/cm^{-1} 2974, 2928, 2874, 1643, 1549, 1125, 1058, 993, 766, 691.

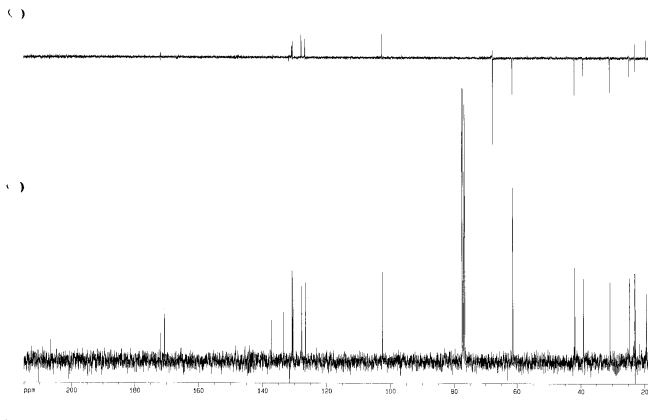




2-(2-Methylphenyl)-N-(4,4-diethoxybutyl)acetamide (3m)

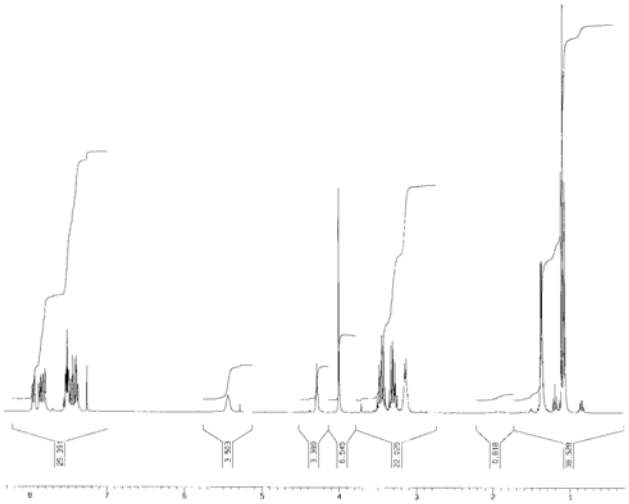
Isolated as a solid (97%) mpt 56-8°C (Et_2O/petrol). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7 Hz, 3H), 1.40–1.57 (m, 4H), 2.27 (s, 3H), 3.19 (q, *J* = 7Hz, 2H), 3.35–3.48 (m, 2H), 3.50–3.64 (m, 4H including 3.55, s), 4.39 (t, *J* = 5 Hz, 1H), 5.44 (brs, 1H), 7.10–7.22 (m, 4H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 19.5 (CH_3), 24.6 (CH_2), 30.7 (CH_2), 39.2 (CH_2), 41.9 (CH_2), 61.2 (CH_2), 102.5 (CH), 126.6 (CH), 127.8 (CH), 130.5 (CH), 130.8 (CH), 133.4 (C), 137.2(C), 170.6 (C); ν_{max}/cm^{-1} 2970, 1838, 1549, 1125, 1059, 1038, 997, 749, 730, 696.

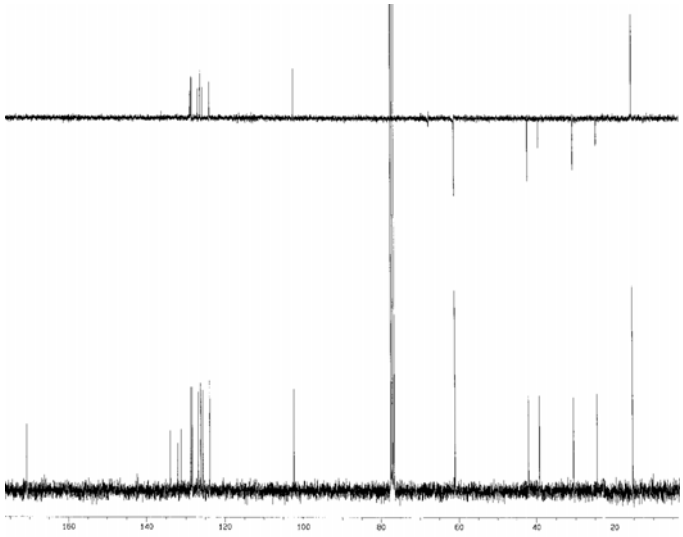




2-(1-Naphthyl)-N-(4,4-diethoxybutyl)acetamide (3n)

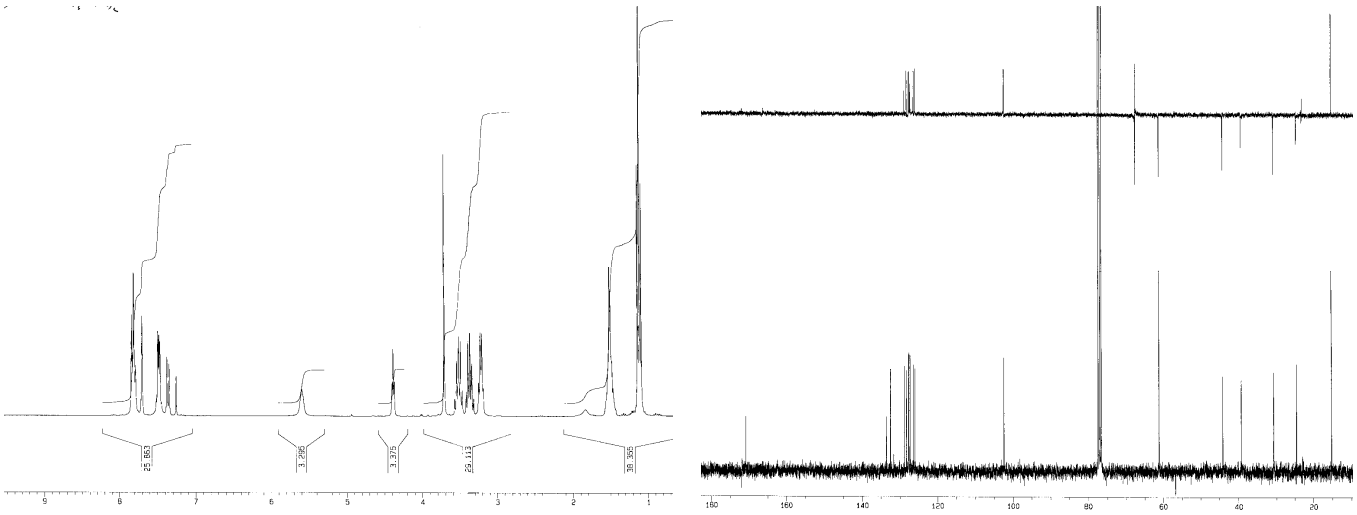
Isolated as a solid (100%), mpt 73-6°C (Et_2O/petrol). NMR (300 MHz, $CDCl_3$): δ = 1.10 (t, *J* = 7 Hz, 6H), 1.32–1.40 (m, 4H), 3.10-3.18 (m, 2H), 3.24–3.35 (m, 2H), 3.37–3.48 (m, 2H), 4.01 (s, 2H), 4.47 (t, *J* = 5 Hz, 1H), 5.44 (brs, 1H), 7.35–7.56 (m, 4H), 7.76–7.88 (m, 2H), 7.95 (d, *J* = 7 Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.2 (CH_3), 24.5 (CH_2), 30.5 (CH_2), 39.1 (CH_2), 41.9 (CH_2), 61.0 (CH_2), 102.3 (CH), 123.9 (CH), 125.6 (CH2), 126.2 (CH), 126.8 (CH), 128.4 (CH), 128.5 (CH), 128.8 (CH), 131.2 (C), 132.1 (C), 134.0 (C), 170.8 (C).





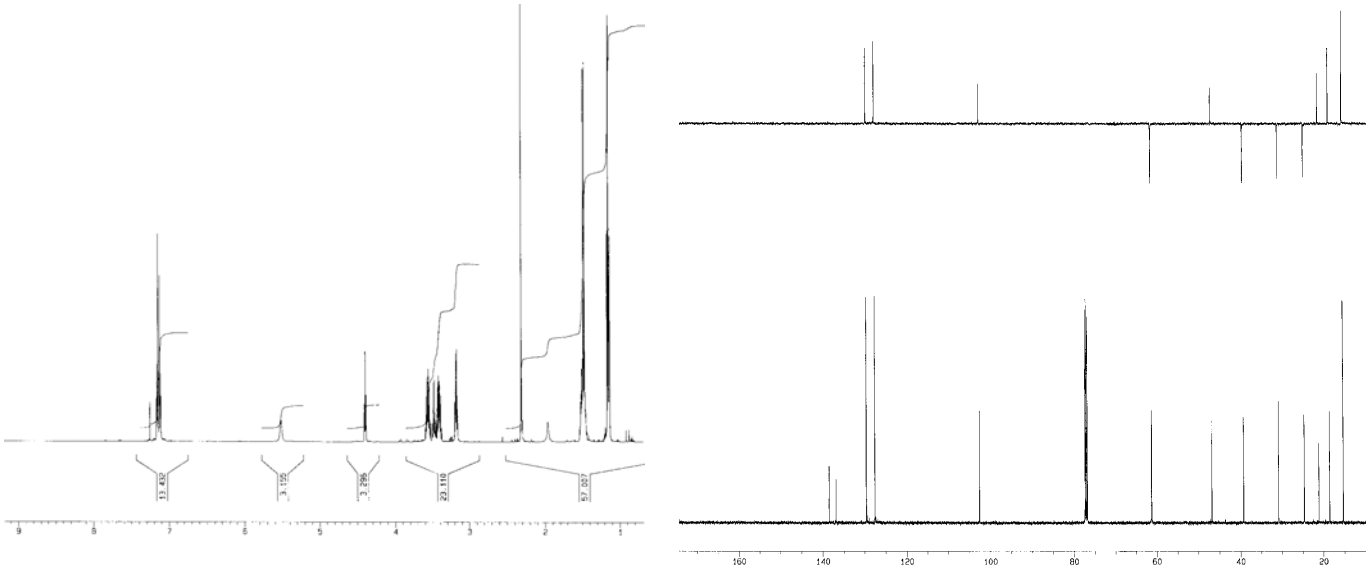
2-(2-Naphthyl)-N-(4,4-diethoxybutyl)acetamide (3o)

Isolated as an oil which gave a sticky solidified on standing (100% yield). NMR (300 MHz, $CDCl_3$): δ = 1.12 (t, *J* = 7 Hz, 6H), 1.41–1.58 (m, 4H), 3.18-3.25 (m, 2H), 3.30–3.42 (m, 2H), 3.45–3.58 (m, 2H), 3.72 (s, 2H), 4.39 (t, *J* = 5 Hz, 1H), 5.60 (brs, 1H), 7.36(d, *J* = 8.5Hz, 1H), 7.45–7.55 (m, 2H), 7.70 (s, 1H), 7.78-7.88 (m, 3H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 44.1 (CH_2), 61.2 (CH_2), 102.5 (CH), 126.0 (CH), 126.4 (CH), 127.3 (CH), 127.6 (CH), 127.7 (CH), 128.3 (CH), 128.8 (CH), 131.6 (C), 132.5 (C), 133.6 (C), 170.8 (C); ν_{max}/cm^{-1} 2972, 1642, 1616, 1545, 1124, 1062, 817, 737.



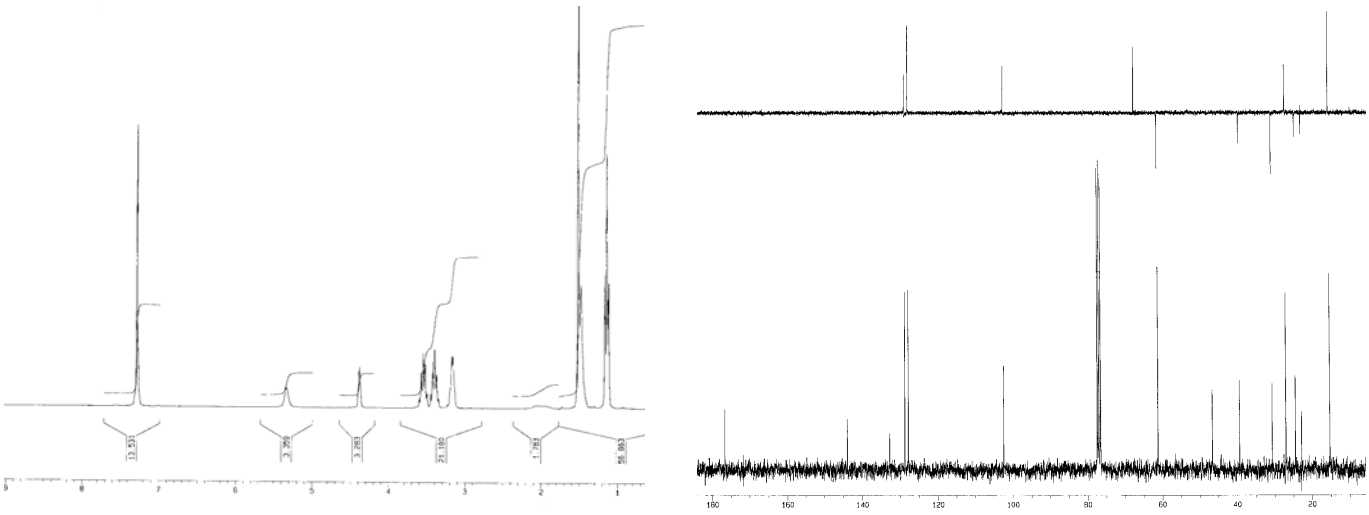
2-Methyl-2-(4-methylphenyl)-N-(4,4-diethoxybutyl)-acetamide (3p)

Ioslated as an oil (96% yield), ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7 Hz, 6H), 1.40–1.58 (m, 7H including 1.48, d, *J* = 7 Hz, 3H), 2.31 (s, 3H), 3.18 (q, *J* = 7Hz, 2H), 3.38–3.57 (m, 2H), 3.48 (q, *J* = 7 Hz, 1H), 3.54–3.62 (m, 2H), 4.40 (t, *J* = 5 Hz, 1H), 5.52 (brs, 1H), 7.12 (d, *J* = 9 Hz, 2H), 7.16 (d, *J* = 9 Hz, 2H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 15.4 (CH_3), 18.6 (CH_3), 21.1 (CH_3), 24.6 (CH_2), 30.8 (CH_2), 39.3 (CH_2), 46.8 (CH), 61.3 (CH_2), 102.6 (CH), 127.6 (CH), 129.6 (CH), 136.9 (C), 138.5 (C), 174.5 (C)



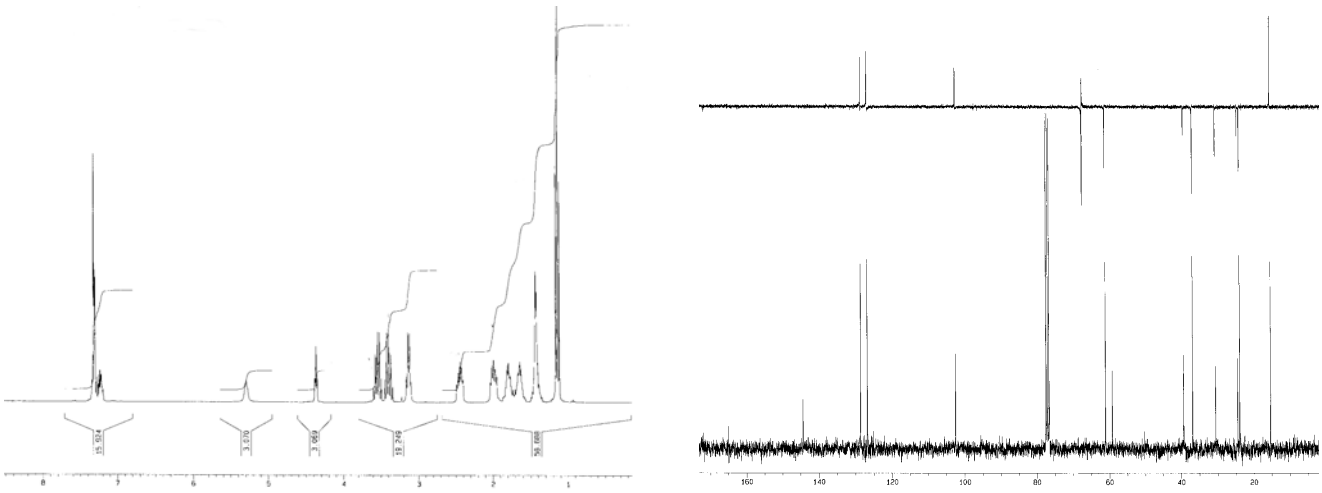
2-(4-Chlorophenyl)-2,2-dimethyl-N-(4,4-diethoxybutyl)-acetamide (3q)

Isolated as an oil (100% yield): ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7 Hz, 3H), 1.15 (t, *J* = 7 Hz, 3H), 1.38–1.56 (m, 10H including 1.51, s, 6H), 3.10–3.21 (m, 2H), 3.32–3.46 (m, 2H), 3.47–3.61 (m, 2H), 4.35–4.41 (m, 1H), 5.33 (brs, 1H), 7.28 (s, 4H).; ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 22.8 (CH_2), 24.5 (CH_2), 27.0 (CH_3), 30.7 (CH_2), 39.3 (CH_2), 46.6 (C), 61.2 (CH_2), 102.4 (CH), 127.8 (CH), 128.7 (CH), 132.8 (C), 143.9 (C), 176.7 (C).



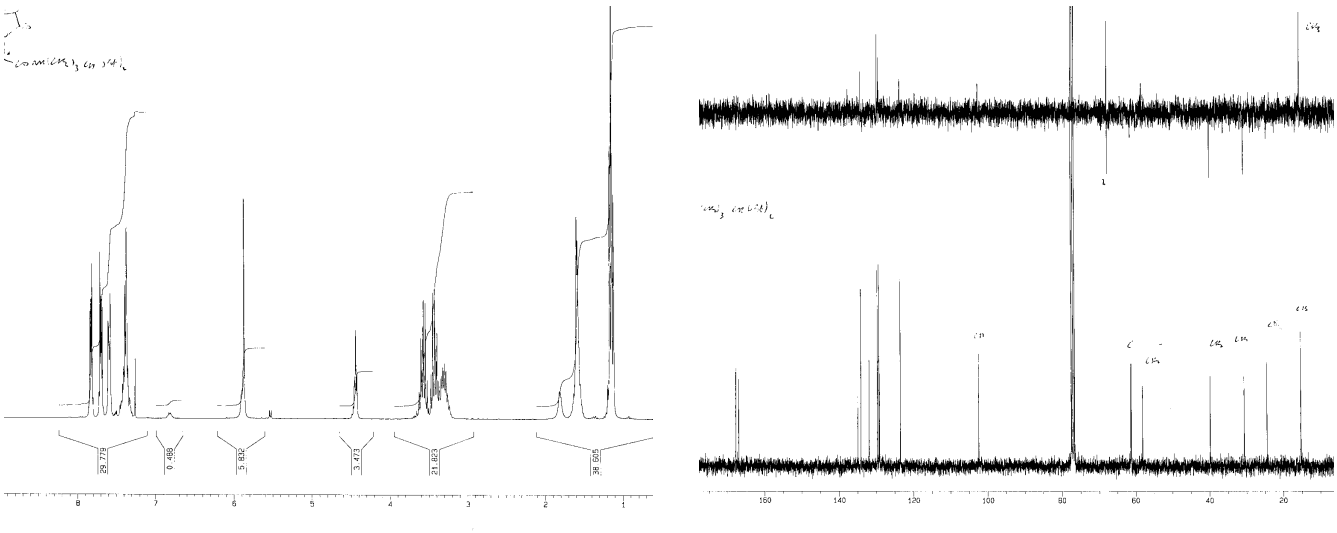
1-Phenyl-cyclopentanecarboxylic acid (4,4-diethoxy-butyl)-amide (3r)

Isolated as an oil (100% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = *7* Hz, 3H), 1.15 (t, *J* = 7 Hz, 3H), 1.37–1.50 (m, 4H), 1.56–1.88 (m, 4H), 1.91–2.07 (m, 2H), 2.37–2.50 (m, 2H), 3.13 (q, *J* = 6 Hz, 2H), 3.32–3.46 (m, 2H), 3.47–3.61 (m, 2H), 4.37 (t, *J* = 5 Hz, 1H), 5.45 (brs, 1H), 7.17–7.36 (m, 5H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.0 (CH_2), 24.5 (CH_2), 30.6 (CH_2), 36.8 (CH_2), 39.3 (CH_2), 59.3 (C), 61.1 (CH_2), 102.5 (CH), 126.8 (CH), 128.7 (CH), 144.4 (C), 176.4 (C).



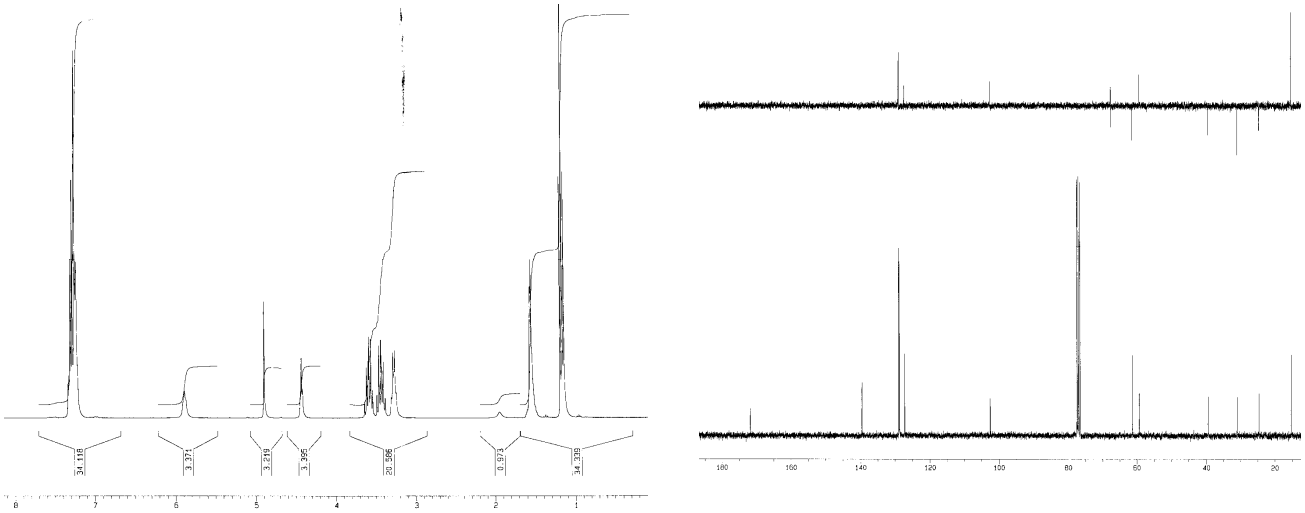
N-(4,4-Diethoxybutyl)-2-(4-(1,3-dioxo-1,3-dihydroisoindol-2-yl)-2-phenylacetamide (3s)

Isolated as a white solid (86%) mpt 78-80^{o}C (Et_2O/petrol). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7 Hz, 6H), 1.51-1.66 (m, 4H), 3.19-3.65 (m, 6H), 4.44 (t, *J* = 5 Hz, 1H), 5.87 (brs, 2H), 7.26-7.45 (m, 3H), 7.59 (dd, *J* = 7, 2Hz, 2H), 7.69 (dd, *J* = 6, 3 Hz, 2H), 7.83 (dd, *J* = 6, 3Hz, 2H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.7 (CH_2), 39.8 (CH_2), 58.1 (CH), 61.2 (CH_2), 61.3 (CH_2), 102.5 (CH), 123.6 (CH), 129.1 (CH), 129.3 (CH), 129.6 (CH), 131.9 (C), 134.1 (CH), 135.0 (C), 167.0 (C), 167.8 (C)



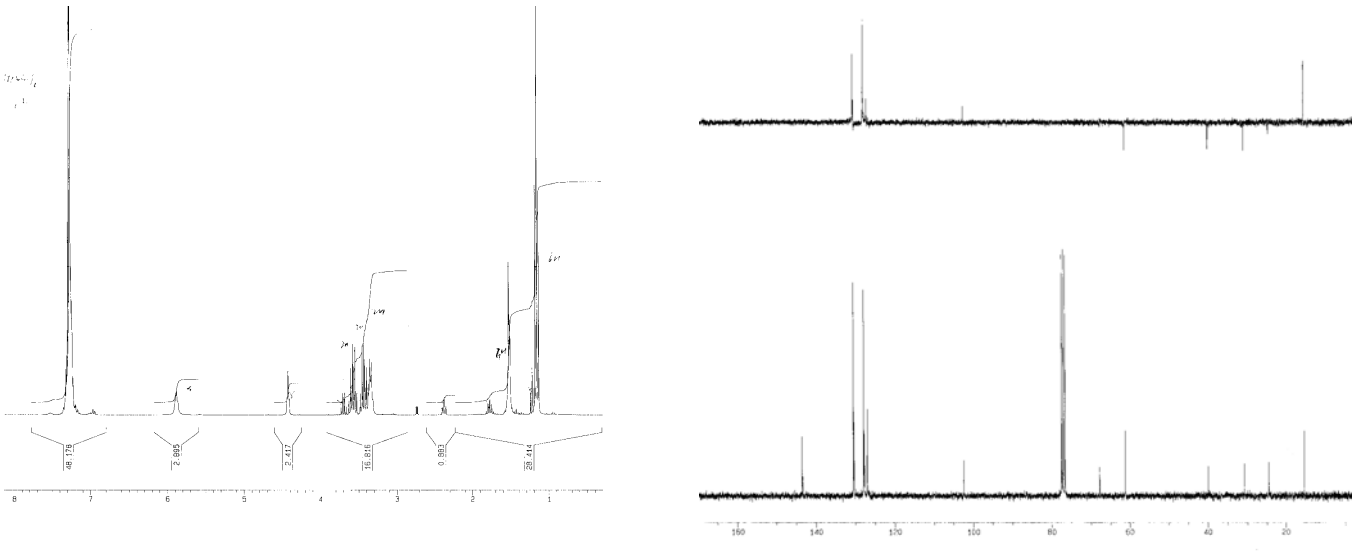
2,2-Diphenyl-N-(4,4-diethoxybutyl)acetamide (3t)

Isolated as a white solid (95%) mpt 70-2°C (Et_2O/petrol): ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7Hz, 6H), 1.48-1.60 (m, 4H), 3.27 (q, *J* = 7 Hz, 2H), 3.36–3.48 (m, 2H), 3.39–3.65 (m, 2H), 4.43 (t, *J* = 5 Hz, 1H), 4.90 (s, 1H), 5.89 (brs, 1H), 7.16–7.38 (m, 10H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.6 (CH_2), 30.8 (CH_2), 39.5 (CH_2), 59.2 (CH), 61.3 (CH_2), 102.5 (CH), 127.2 (CH), 128.7 (CH), 128.9 (CH), 139.6 (C), 171.8 (C), ν_{max}/cm^{-1} 3263, 2975, 2874, 1639, 1589, 1125, 1060, 1030, 1003, 743, 717, 696.



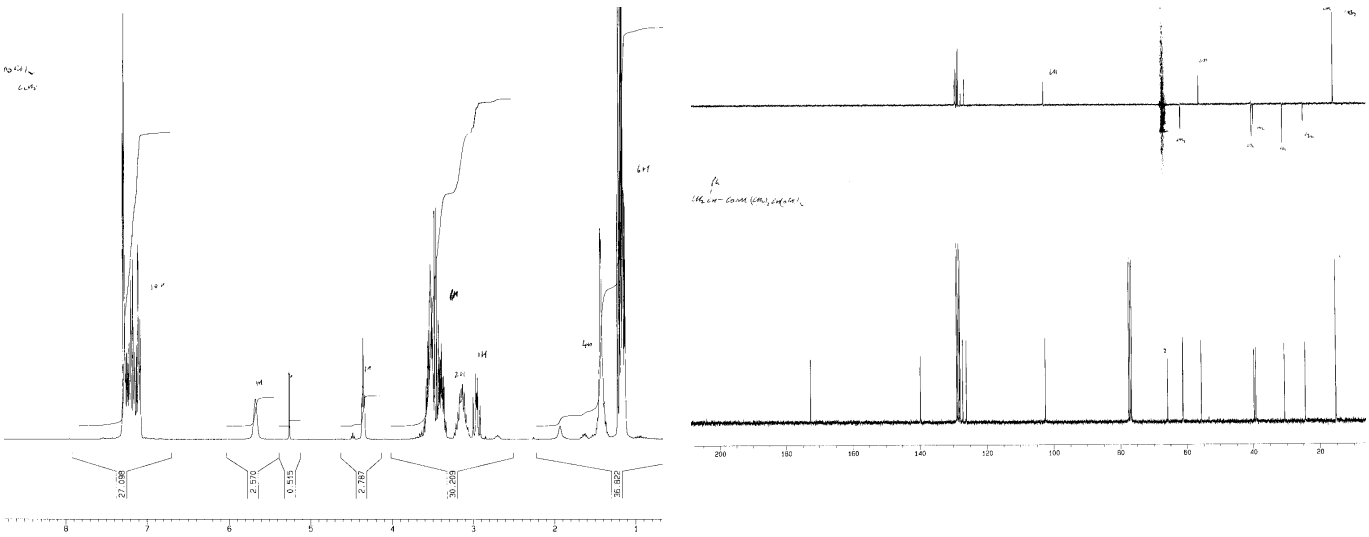
2,2,2-Triphenyl-N-(4,4-diethoxybutyl)acetamide (3u)

Isolated as white solid (100%)mpt 102-5°C: ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.50–1.56 (m, 4H), 3.30-3.64 (m, 6H), 4.42 (t, *J* = 5 Hz, 1H), 5.88 (brt, *J* = 7 Hz, 1H), 7.21-7.34 (m, 15H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.9 (CH_2), 39.5 (CH_2), 61.2 (CH_2), 67.8 (C), 102.5 (CH), 127.0 (C), 127.9 (CH), 130.5 (CH), 143.4 (C), 173.4 (C).



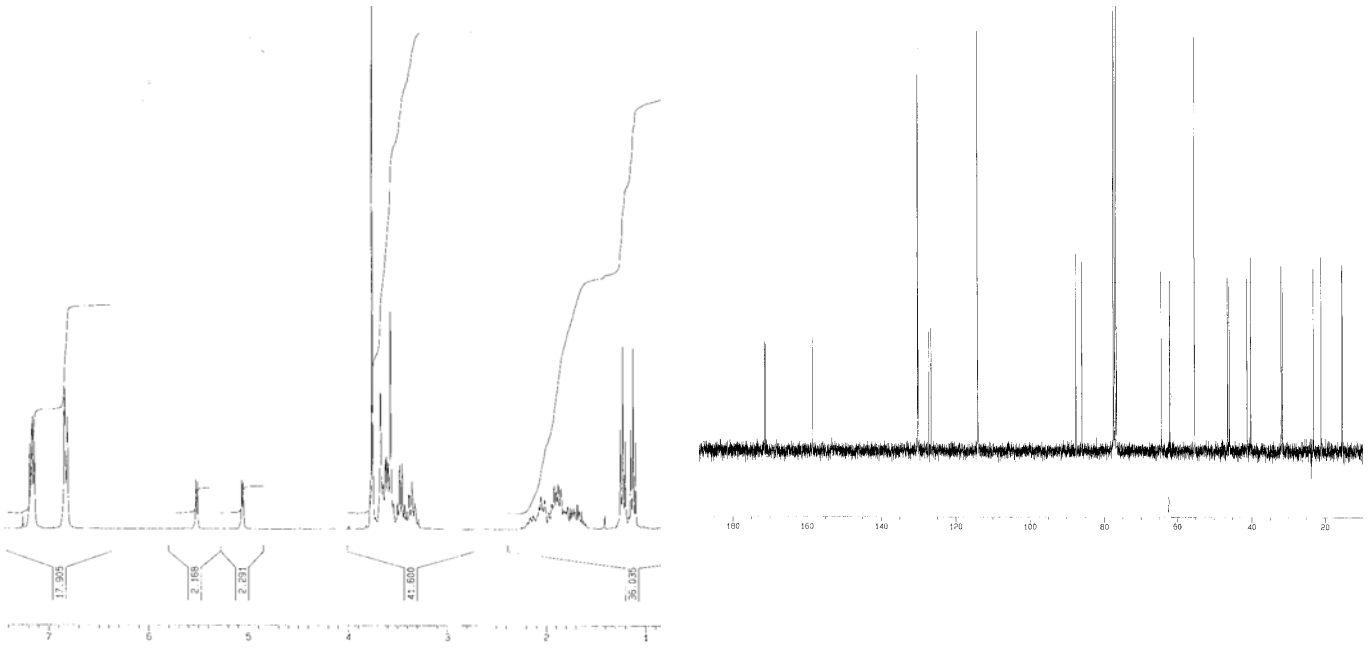
2-Benzyl -2-phenyl-N-(4,4-diethoxybutyl)-acetamide (3v)

Isolated as an oil which solidied on standing (97% yield), mpt 66-8°C (Et_2O/petrol). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 6 Hz, 6H), 1.42–1.57 (m, 4H), 2.88-3.01 (m, 1H), 3.03-3.23 (m, 2H), 3.30-3.60 (m, 6H), 4.35 (t, *J* = 5 Hz, 1H), 5.67 (brs, 1H), 7.06–7.32 (m, 10H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.7 (CH_2), 39.2 (CH_2), 39.7 (CH_2), 55.7 (CH), 61.2 (CH_2), 102.5 (CH), 126.2 (CH), 127.2 (CH), 128.0 (CH), 128.2 (CH), 128.7 (CH), 129.0 (CH), 139.8 (C), 139.9 (C), 172.6 (C); ν_{max}/cm^{-1} 1633, 1555, 1106, 1057, 755, 703.

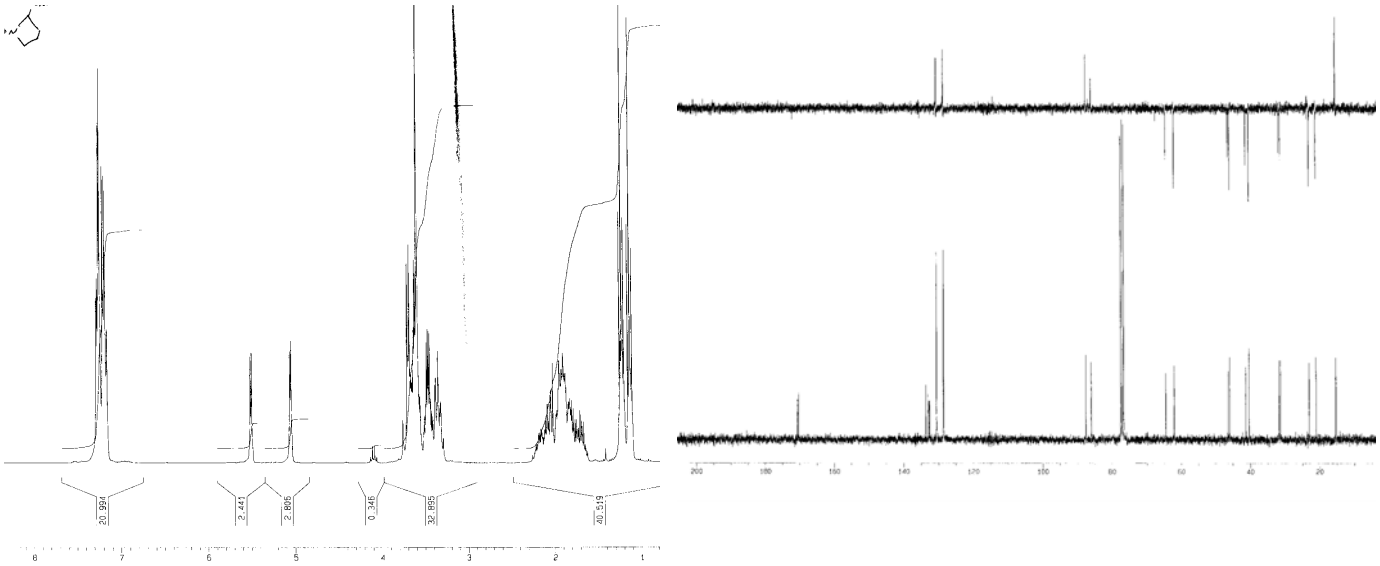


2-(4-Methoxyphenyl)-1-(2-ethoxypyrrolidin-1-yl)ethanone (5d)

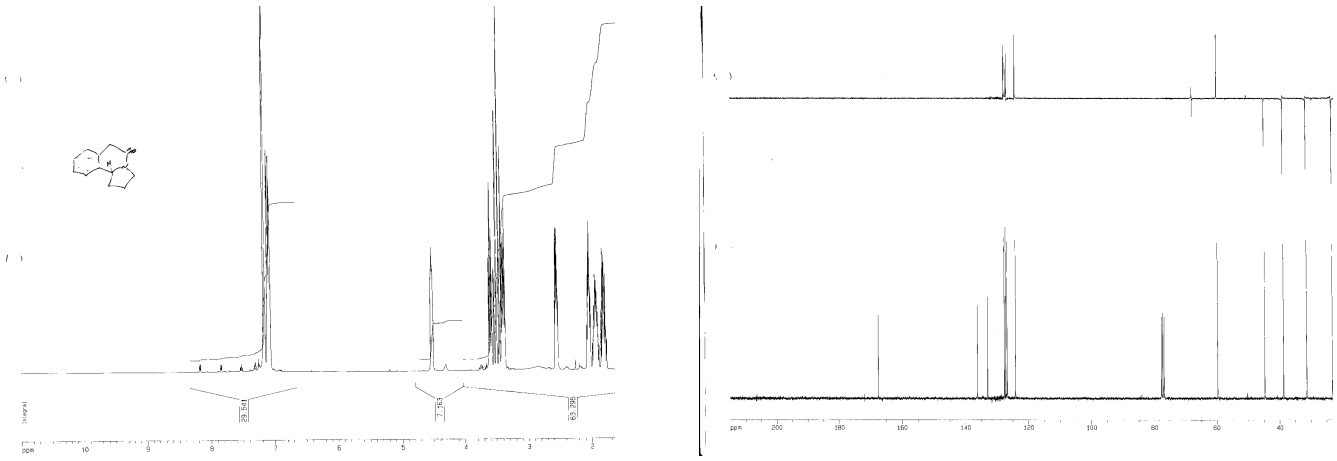
Isolated as an oil (86% yield). ^{1}H NMR (300 MHz, $CDCl_3$) as a mixture of rotomers : δ = 1.12 (t, *J* = 7Hz, 1.5H), 1.22 (t, *J* = 7Hz, 1.5H), 1.60–2.20 (m, 4H), 3.25–3.72 (m, 6H), 3.77 (s, 3H), 5.05 (d, *J* = 4 Hz, 0.5H), 5.51 (d, J = 4Hz, 0.5H), 6.82 (d, *J* = 8.5Hz, 1H), 6.83 (d, *J* = 8.5Hz, 1H), 7.15 (d, *J* = 8.5Hz, 1H), 7.17 (d, *J* = 8.5 Hz, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 15.4 (CH_3), 21.0 (CH_2), 23.1 (CH_2), 31.5 (CH_2), 31.9 (CH_2), 40.3 (CH_2), 41.2 (CH_2), 45.8 (CH_2), 46.3 (CH_2), 55.2 (CH_3), 62.1 (CH_2), 64.4 (CH_2), 85.9 (CH), 87.5 (CH), 114.0 (CH), 126.6 (C), 127.2 (C), 130.1 (CH), 130.2 (CH), 158.4 (C), 158.5 (C), 171.1 (C), 171.5 (C).



NMR of **2-(4-Chlorophenyl)-1-(2-ethoxypyrrolidin-1-yl)ethanone (5c**)

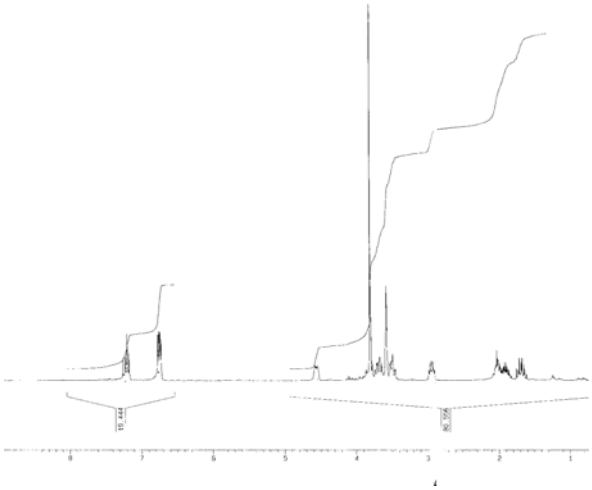


NMR of **2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquino-lin-5-one (7)**



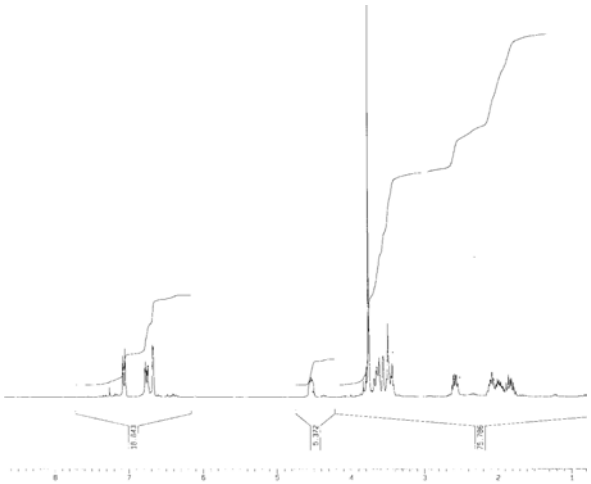
10-Methoxy-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquino-lin-5-one (7a)

Isolated as a solid (33% yield), mpt 133-6°C (lit. 134-6°C[12]) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.57 – 1.70 (m, 1H), 1.80 – 2.05 (m, 2H), 2.94 (dt, J = 6, 12 Hz, 1H), 3.40 – 3.80 (m, 4H), 3.81 (s, 3H), 4.52-4.59 (m, 1H),6.72 – 6.78 (m, 2H), 7.18 (t, J = 7Hz, 1H).



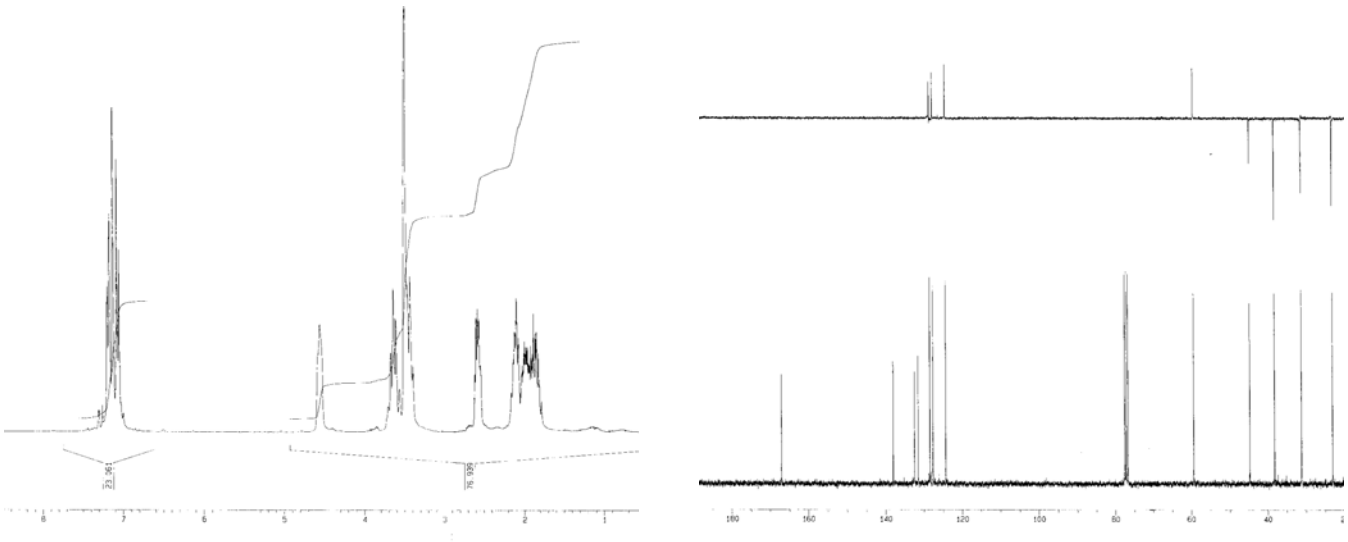
8-Methoxy-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquino-lin-5-one (7a')

Isolated as an oil (46% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.85 – 2.18 (m, 3H), 2.58 (dt, *J* = 6, 12Hz, 1H), 3.35 – 3.65 (m, 4H), 3.79 (s, 3H), 4.51-4.5 (m, 1H), 6.68 (d, *J* = 2 Hz, 1H), 6.75 (dd, *J* = 8.5, 2Hz, 1H), 7.06 (d, J = 8.5 Hz, 1H).



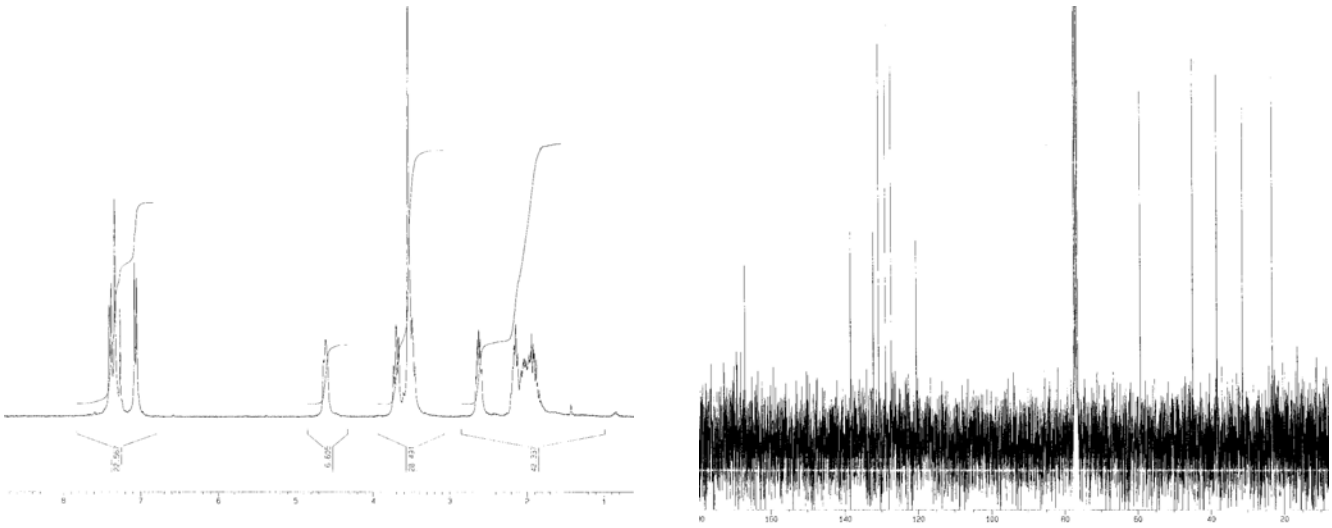
9-Chloro-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquino-lin-5-one (7c)

Isolated as a pale yellow solid (43% yield) m pt 76-9°C (Et_2O/petrol): HRMS: Theoretical Mass: 221.0602, Measured Mass: 221.0595. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.78–2.07 (m, 3H), 2.58 (quin, *J* = 5.5 Hz, 1H), 3.32-3.80 (m, 4H), 4.55–4.60 (m, 1H), 7.06 (d, *J* = 8 Hz, 1H), 7.13 (s, 1H), 7.18 (d, *J* = 8 Hz, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 23.1 (CH_2), 31.2 (CH_2), 38.2 (CH_2), 44.8 (CH_2), 59.3 (CH), 124.3 (CH), 127.7 (CH), 128.5 (CH), 131.6 (C), 132.5 (C), 138.0 (C), 167.1 (C); ν_{max}/cm^{-1} 1647, 1624, 1489, 1445, 1413, 811, 746, 657.



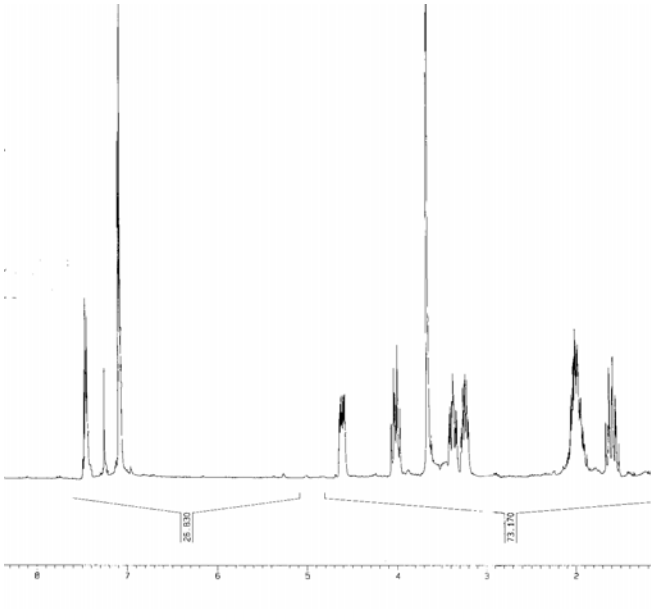
9-Bromo-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquino-lin-5-one (7e)

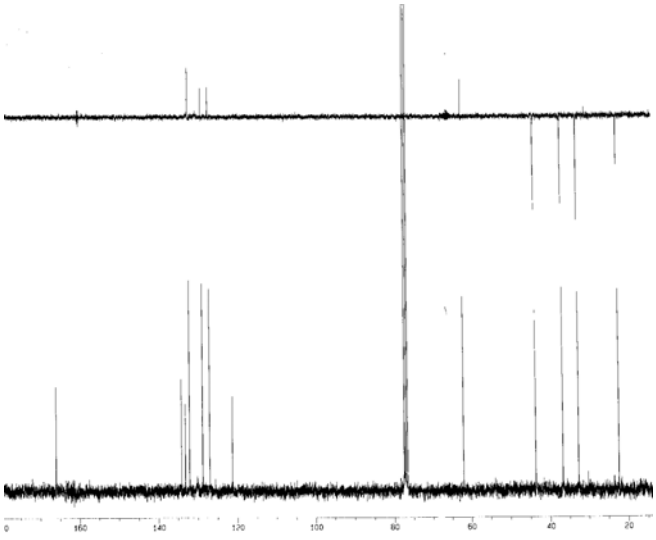
Isolated as an oil (55% yield). Theoretical Mass: 265.0097, Measured Mass: 265.0100. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.78-2.20 (m, 3H), 2.60 (quin, *J* = 5.5 Hz, 1H), 3.35 – 3.60 (m, 3H), 3.70 (t, *J* = 9 Hz, 1H), 4.50 – 4.60 (m, 1H), 7.06 (d, *J* = 7Hz, 1H), 7.30 – 7.45 (m, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 23.2 (CH_2), 31.3 (CH_2), 38.4 (CH_2), 44.8 (CH_2), 59.3 (CH), 120.5 (C), 127.3 (CH), 128.9 (CH), 130.7 (CH), 132.1 (C), 138.4 (C), 167.1 (C).



10-Bromo-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7f')

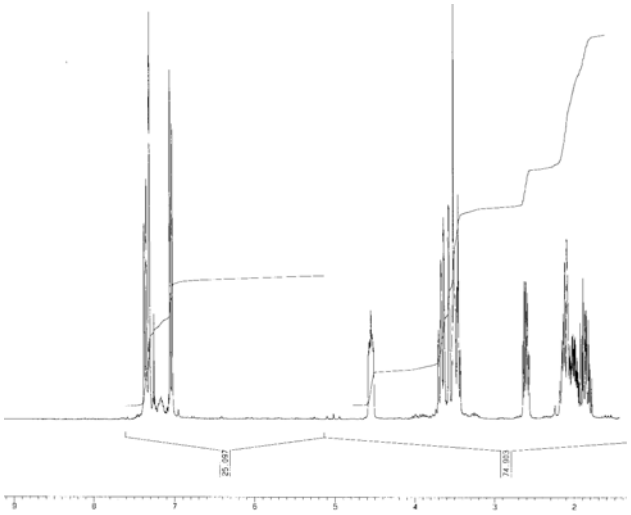
Isolated as a pale yellow solid (26% yield) mpt 96-9^{o}C (Et_2O/petrol), HRMS Theoretical Mass: 265.0097, Measured Mass: 265.0100. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.50–1.68 (m, 1H), 1.85–2.10 (m 2H), 3.18–3.28 (m, 1H), 3.19–3.41 (m, 1H), 3.66 (s, 2H), 3.95–4.07 (m, 1H), 4.53–4.65 (m 1H), 7.09 (d, *J* = 4 Hz, 1H), 7.09 (d, *J* = 5 Hz, 1H), 7.47 (dd, *J* =4, 5 Hz, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.4 (CH_2), 32.7 (CH_2), 36.8 (CH_2), 43.7 (CH_2), 62.0 (CH), 121.2 (C), 127.0 (CH), 128.8 (CH), 132.1 (CH), 133.2 (C), 134.1 (C), 166.1 (C); ν_{max}/cm^{-1} 1642, 1435, 1402, 882, 79, 719.

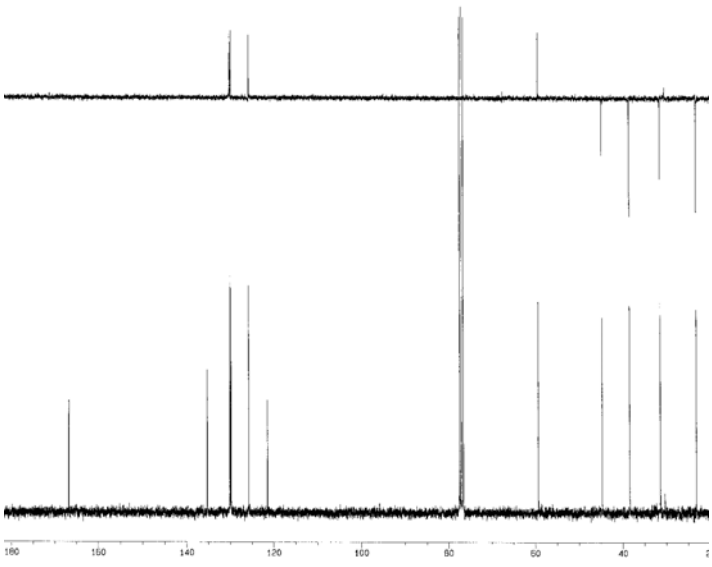




8-Bromo-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7f)

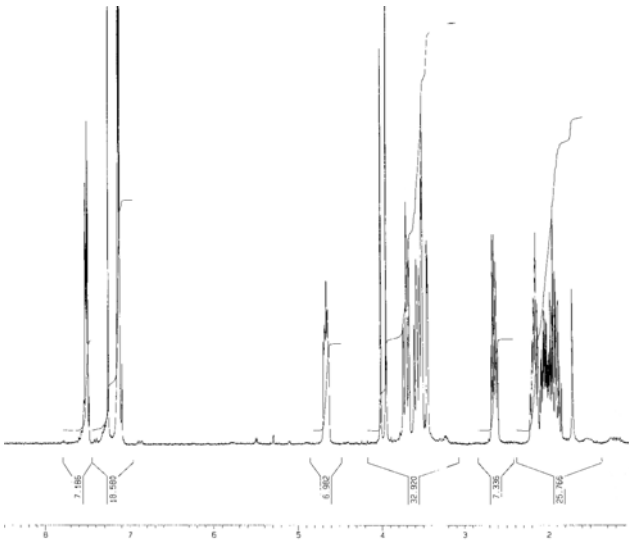
Isolated as a pale yellow solid (45% yield) m pt 118-20°C (Et_2O/petrol): HRMS Theoretical Mass: 265.0097, Measured Mass: 265.0101. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.77-2.19 (m, 3H), 2.61 (dt, *J* = 6, 12Hz, 1H), 3.41-3.71 (m, 4H), 4.50–4.59 (m, 1H), 7.04 (d, *J* = 8Hz, 1H), 7.31 (s, 1H), 7.36 (d, *J* = 8 Hz, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 23.2 (CH_2), 31.4 (CH_2), 38.4 (CH_2), 44.8 (CH_2), 59.3 (CH), 121.4 (C), 125.8 (CH), 129.9 (CH), 130.1 (CH), 135.3 (C), 135.3 (C), 166.8 (C).

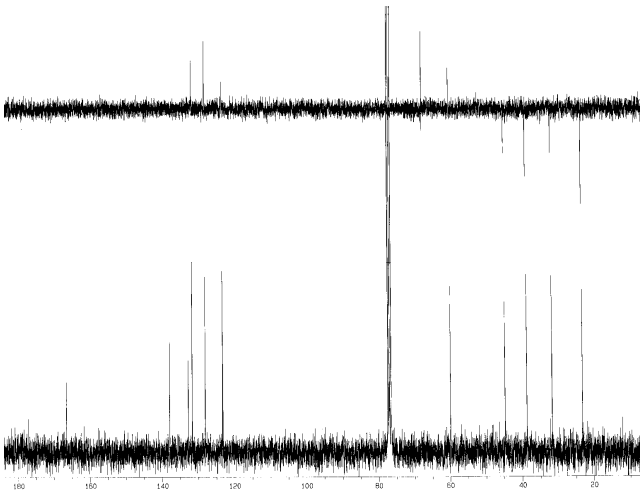




7-Bromo-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquinol-in-5-one (7h)

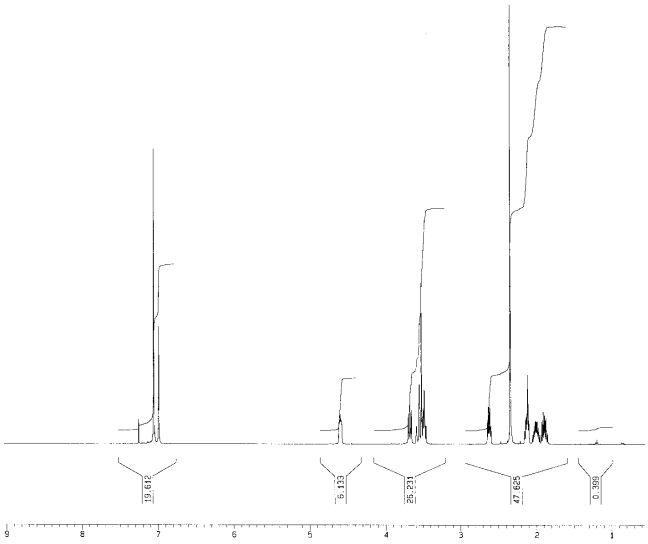
Isolated as pale yellow solid (20% yield), m pt 144-8°C; Theoretical Mass: 265.0097, Measured Mass: 265.0100. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.82-2.25 (m, 3H), 2.64 (dt, *J* = 12, 6Hz, 1H), 3.48 (dd, *J* = 19, 5 Hz, 1H), 3.58 (t, *J* = 8 Hz, 1H), 3.71 (dt, *J* = 9, 2 Hz, 1H), 3.98 (d, *J* = 19 Hz, 1H), 4.60–4.70 (m, 1H), 7.08–7.20 (m, 2H), 7.47–7.60 (m, 1H): ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 23.2 (CH_2), 31.7 (CH_2), 38.7 (CH_2), 44.7 (CH_2), 59.9 (CH), 123.3 (CH), 123.4 (C), 128.1 (CH), 131.7 (CH), 132.8 (C), 137.9 (C), 166.6 (C), ν_{max}/cm^{-1} 1646, 1438, 793, 750, 718, 661..

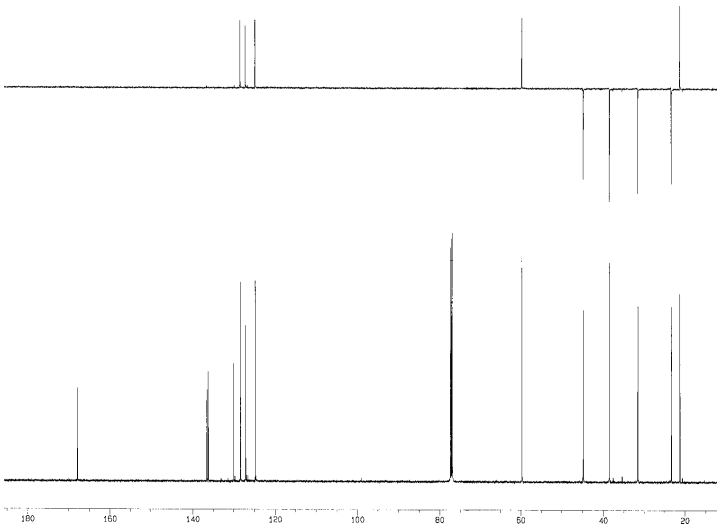




9-Methyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7k)

Isolated as a pale yellow solid (75%), mpt 120-4°C (Et_2O/petrol), HRMS Theory 201.1148, Found 201.1155. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.87–2.08 (m, 2H), 2.10–2.18 (m, 1H), 2.34 (s, 3H), 2.61–2.68 (m, 1H), 3.46–3.52 (m, 3H), 3.68 (t, *J* = 10 Hz, 1H), 4.56–4.62 (m, 1H), 7.02 (s, 1H), 7.07 (s, 2H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 21.2 (CH_3), 23.3 (CH_2), 31.5 (CH_2), 38.4 (CH_2), 44.8 (CH_2), 59.7 (CH), 124.8 (CH), 127.2 (CH), 128.4 (CH), 130.0 (C), 136.2 (C), 136.5 (C), 167.9 (C).

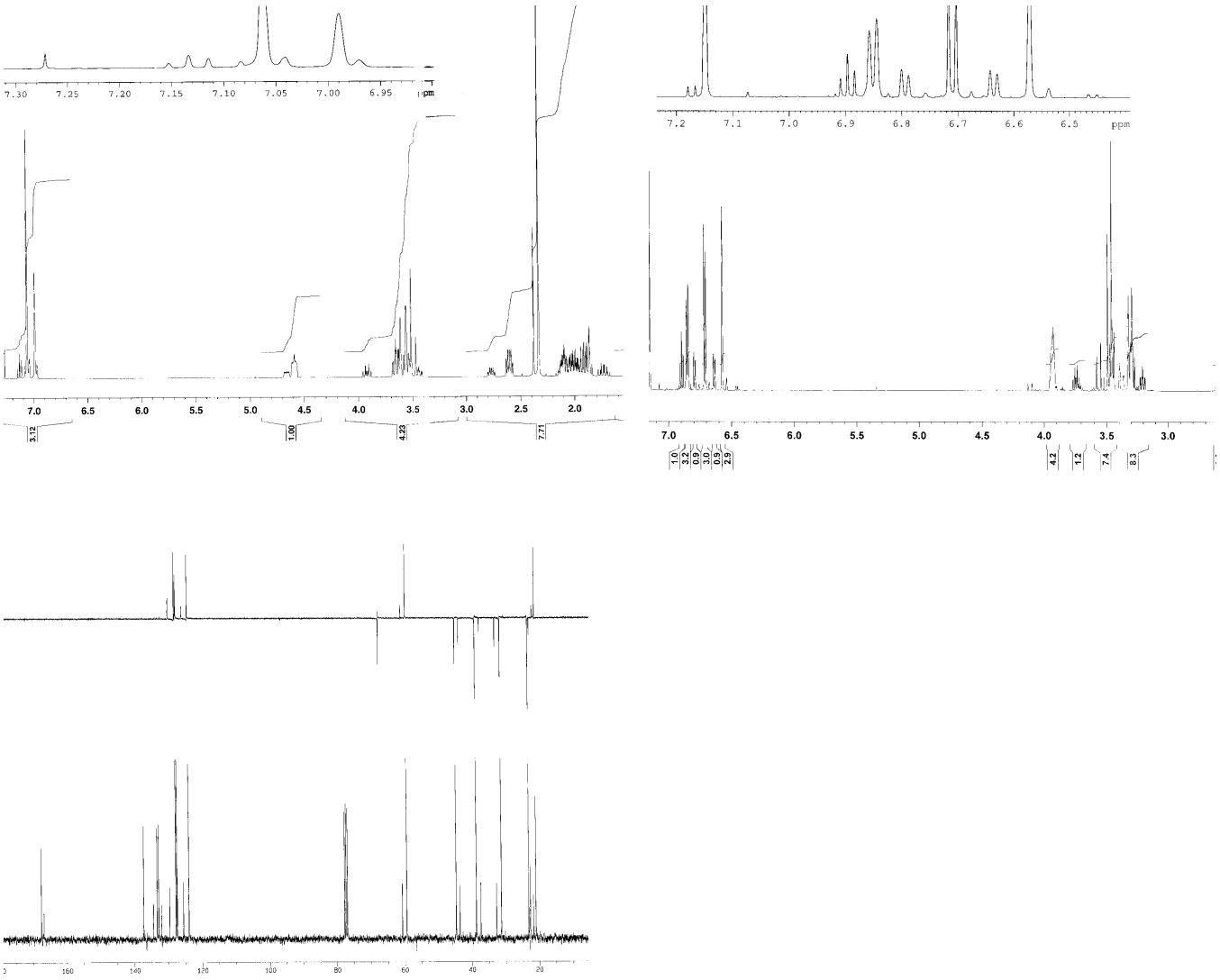




Mixture of 8-Methyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7l) and 10-Methyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7l')

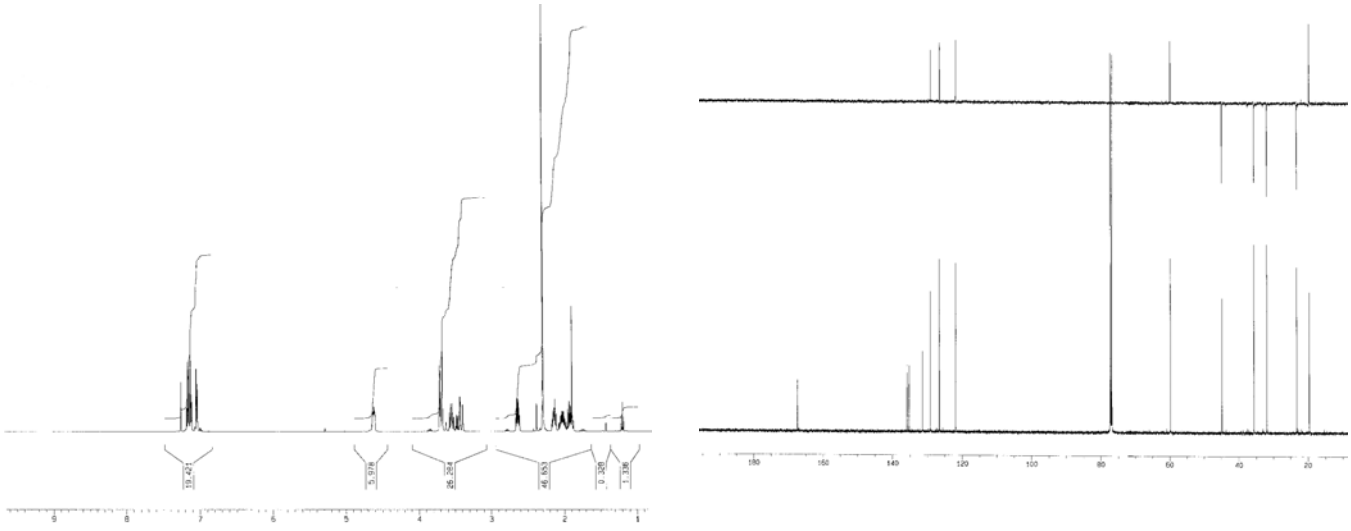
Isolated as an oil as an inseparable mixture of 3:1 ratio of the 8 and 10 isomers respectively (78% combined yield), HRMS Theory 201.1148, Found 201.1152. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.70-2.20 (m, 3H), 2.34 (s, 2.25H), 2.40 (s, 0.25H), 2.65 (tt, *J* = 4, 8 Hz, 0.75H), 2.82 (tt, *J* = 4, 8Hz, 0.25H), 3.40 – 3.70 (m, 4H), 4.58 – 4.64 (m, 0.75H), 4.66-4.72 (m, 0.25H), 7.01 (brs, 1H), 7.06-7.11 (m, 1.75H), 7.17 (t, *J* = 8Hz, 0.25H), ^{1}H NMR (600 MHz, C_6D_6): δ = 6.57 (s, 0.75H), 6.64 (d, *J* = 7.5 Hz, 0.25H), 6.71 (d, *J* = 7.5 Hz, 0.75H), 6.79 (d, *J* = 7.5 Hz, 0.25H), 6.85 (d, *J* = 7.5 Hz, 0.75H), 8.90 (t, J = 7.5 Hz, 0.25H), ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 21.0 (CH_3), *21.7 (CH_3), 22.7 (CH_2),* 23.2 (CH_2), 31.4 (CH_2), *32.9 (CH_2), 37.3 (CH_2),* 38.7 (CH_2), *43.5 (CH_2),* 44.6 (CH_2), 59.4 (CH), *60.7 (CH),* 123.9 (CH), *125.5 (CH), 127.3 (CH),* 127.4 (CH), 127.7 (CH), *129.6 (CH), 132.0 (C), 132.8 (C)*, 132.9 (C), 133.3 (C), *134.4 (C),* 137.3 (C), *166.8 (C),* 167.5 (C), Italics – minor (10-) isomer.

In $CDCl_3$ In C_6D_6



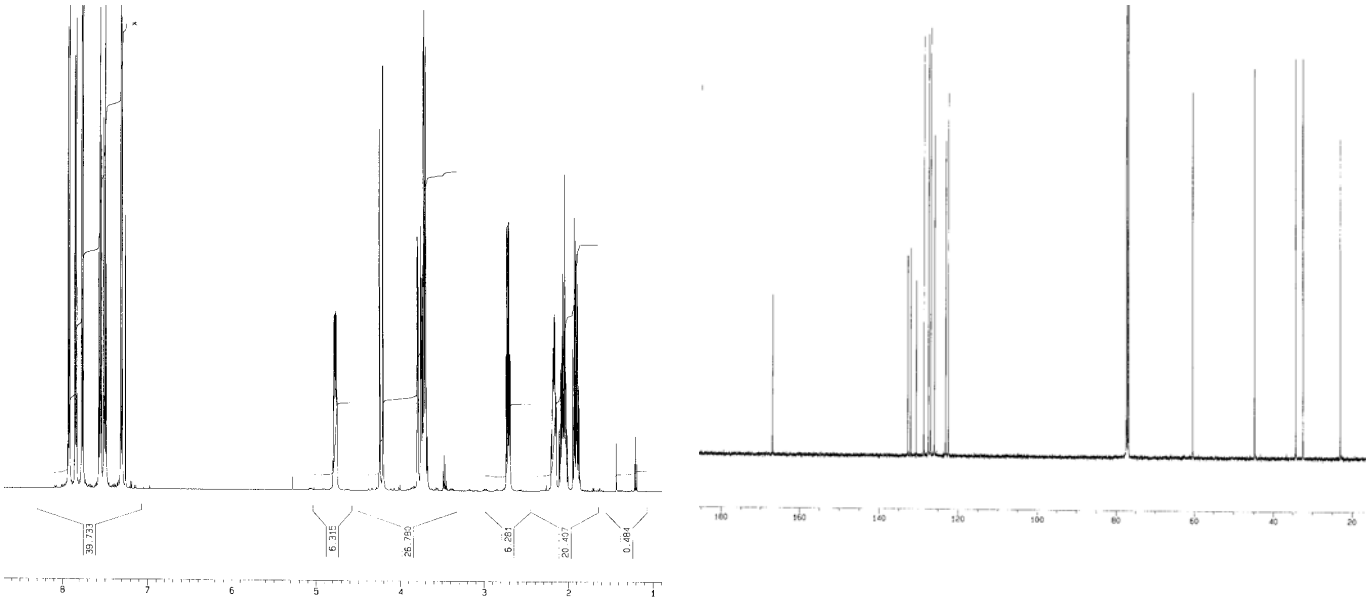
7-Methyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7m)

Isolated as a white solid (96% yield), mpt 111-3°C (Et_2O/petrol), HRMS Theory 201.1148, Found 201.1139. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.87–2.08 (m, 2H), 2.10–2.18 (m, 1H), 2.30 (s, 3H), 2.61–2.68 (m, 1H), 3.41 (dd, *J* = 19, 2.5 Hz, 1H), 3.50–3.60 (m, 1H), 3.66–3.76 (m, 2H), 4.60–4.67 (m, 1H), 7.02 (d, *J* = 7.5 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.18 (t, *J* = 7.5 Hz, 1H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 19.6 (CH_3), 23.2 (CH_2), 31.9 (CH_2), 35.7 (CH_2), 43.9 (CH_2), 59.8 (CH), 122.0 (CH), 126.6 (CH), 129.2 (CH), 132.1 (C), 135.3 (C), 135.9 (C), 167.5 (C); ν_{max}/cm^{-1} 1649, 1469, 1455, 1443, 1406, 795, 734.



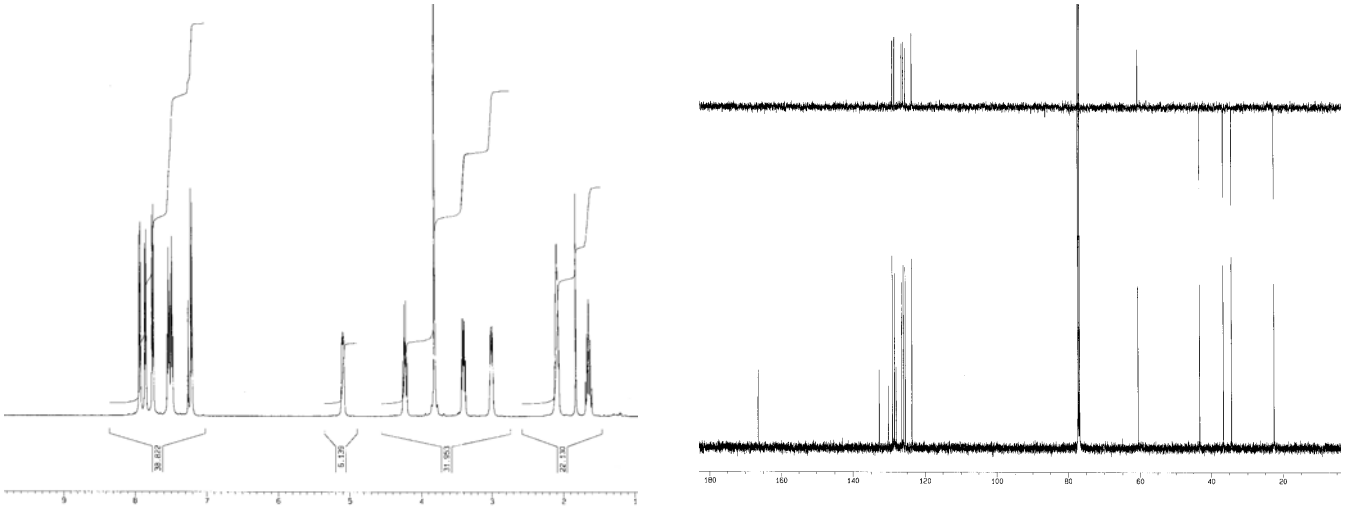
8,9,10,10a-tetrahydro-5H-benzo[f]pyrrolo[2.1-a]isoquinolin-6-one (7n)

Isolated as a pale yellow solid (79% yield), mpt 110-2°C (petrol). HRMS Theory 237.1148. Found 237.1155. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.91 (dq, *J* = 7.3, 11.6 Hz, 1H), 2.00–2.12 (m, 1H), 2,13–2.23 (m, 1H), 2.71 (dt, *J* = 5.5, 11.8 Hz, 1H), 3.65–3.73 (m, 2H), 3.75 (dd, *J* = 19.8, 4.4 Hz, 1H), 4.23 (dd, *J* = 19.6, 1.7 Hz, 1H), 4.77 (dt, *J* = 4.2, 11.2 Hz, 1H), 7.30 (d, *J* = 8.5 Hz, 1H), 7.50 (dt, *J* = 6.9, 1.5 Hz, 1H), 7.56 (dt, *J* = 6.9, 1.5 Hz, 1H), 7.76 (d, *J* = 8.5 Hz, 1H), 7.84 (d, *J* = 7.8 Hz, 1H), 7.92 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 23.0 (CH_2), 32.4 (CH_2), 34.2 (CH_2), 44.8 (CH_2), 60.4 (CH), 122.5 (CH), 123.2 (CH), 126.0 (CH), 127.0 (CH), 127.5 (C), 127.5 (CH), 128.7 (C), 130.6 (CH), 132.1 (C), 132.8 (C), 166.9 (C)



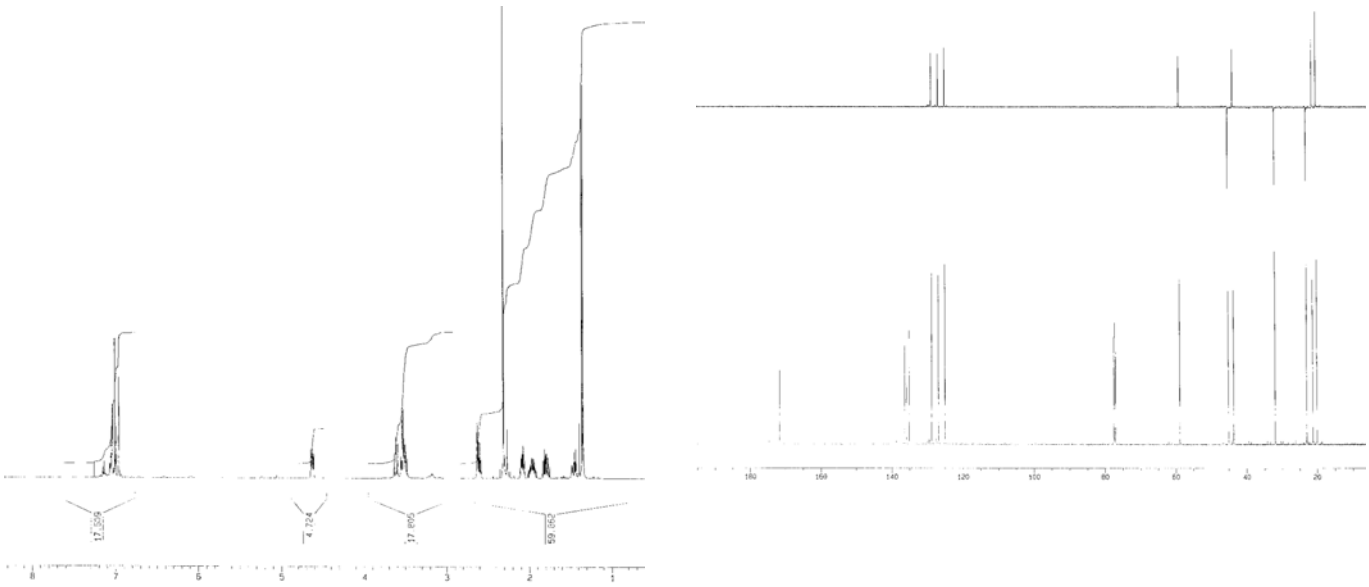
2,3,5,11c-Tetrahydro-1H-3a-aza-cyclopenta[c]phen-anthren-4-one (7o)

Isolated as an oil (71% yield), HRMS Theory 237.1148, Found 237.1149. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.64 (quintet, *J* = 11.2 Hz, 1H), 2.04–2.16 (m, 2H),2.95–3.06 (m, 1H), 3.37–3.47 (m, 1H), 3.81 (s, 2H), 4.23 (dt, *J* = 12.1, 8.2 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 7.49 (t, *J* = 7.1 Hz, 1H), 7.54 (t, *J* = 7.1 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 7.1 Hz, 1H), 7.93 (d, *J* = 7.1 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.5 (CH_2), 34.4 (CH_2), 36.6 (CH_2), 43.2 (CH_2), 60.6 (CH), 123.6 (CH), 125.5 (CH), 126.0 (CH), 126.5 (CH), 128.0 (C), 128.5 (CH), 128.5 (C), 129.0 (CH), 130.2 (C), 132.7 (C), 166.5 (C)



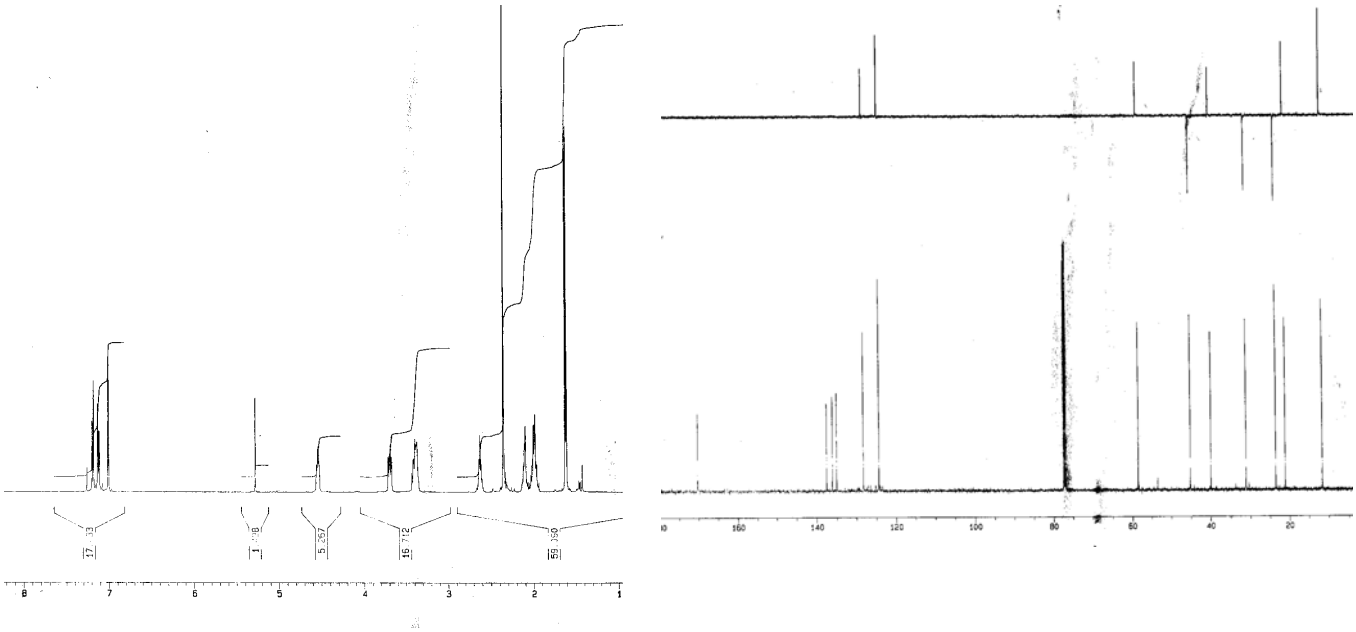
(6R, 10bR, 6S, 10bS)-6,9-Dimethyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7p)

Isolated as an oil (40% yield), HRMS Theory 215.1305, Found 215.1309. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.35 (d, *J* = 7.5 Hz, 3H), 1.73–1.84 (m, 1H), 1.90–2.01 (m, 1H), 2.04–2.13 (m, 1H), 2.31 (s, 3H), 2.61 (quartet, *J* = 6 Hz, 1H), 3.46–3.56 (m, 2H), 3.61 (dd, *J* = 4, 1.9 Hz, 1H), 4.63 (dd, *J* = 10.9, 5.6 Hz, 1H), 6.94 (s, 1H), 7.03 (d, *J* = 7.9 Hz, 1H), 7.06 (d, *J* = 7.9 Hz, 1H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 19.9 (CH_3), 21.1 (CH_3), 23.0 (CH), 32.0 (CH_2), 43.6 (CH_2), 45.1 (CH_2), 58.9 (CH_2), 125.0 (CH), 126.9 (CH), 128.7 (CH), 135.1 (C), 135.9 (C), 136.6 (C), 171.5 (C)



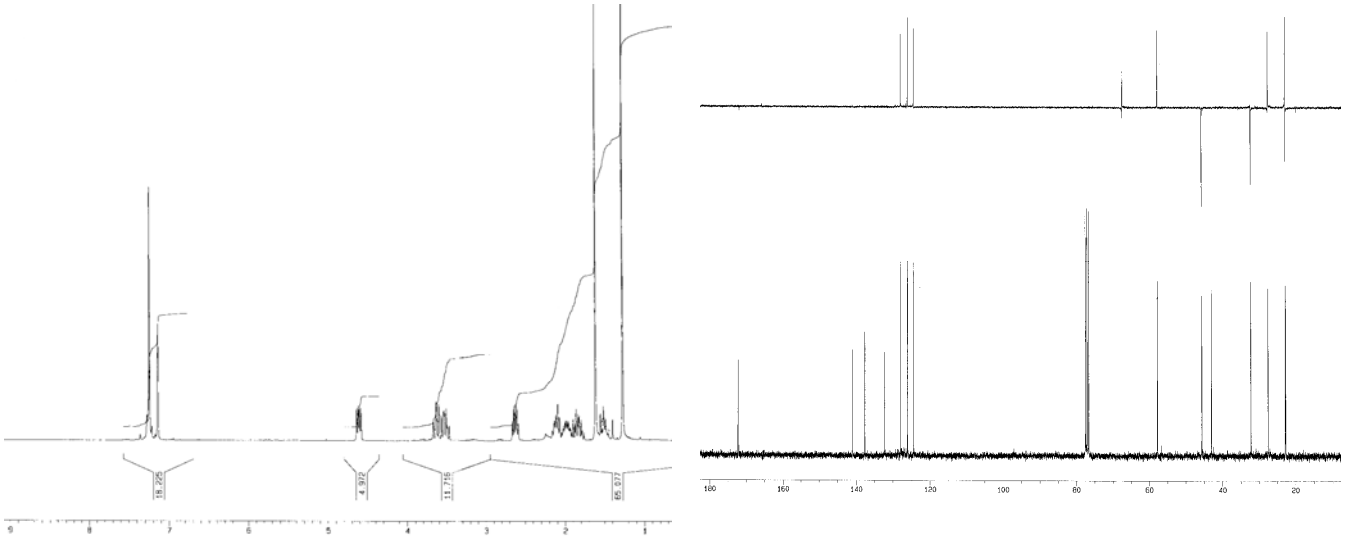
(6S, 10bR, 6R, 10bS)-6,9-Dimethyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7p')

Isolated as an oil (43% yield), HRMS Theory 215.1305, Found 215.1308. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.61 (d, *J* = 7.0 Hz, 3H), 1.94–2.05 (m, 2H), 2.06–2.13 (m, 1H), 2.36 (s, 3H), 2.58–2.64 (m, 1H), 3.34–3.42 (m, 2H), 3.69 (dd, *J* = 11.6, 7.6 Hz, 1H), 4.54 (t, *J* = 6 Hz, 1H), 7.01 (s, 1H), 7.12 (d, *J* = 7.8 Hz, 1H), 7.17 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 11.7 (CH_3), 21.1 (CH_3), 23.6 (CH_2), 31.1 (CH_2), 39.9 (CH), 45.1 (CH2), 58.5 (CH), 124.3 (CH), 128.3 (CH), 135.1 (C), 136.2 (C), 137.6 (C), 170.3 (C)

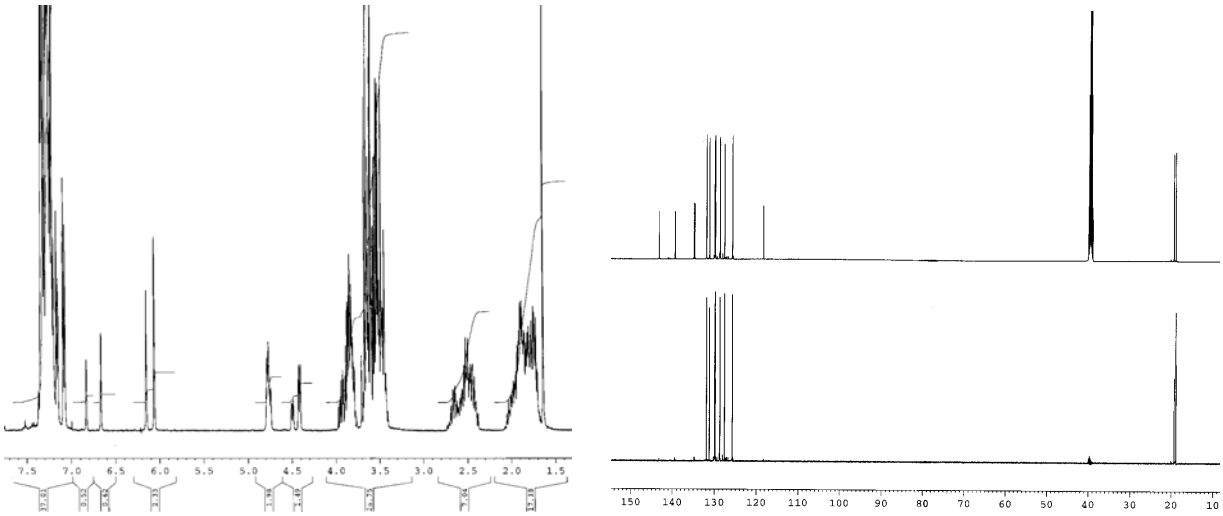


9-Chloro-6,6-dimethyl--2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquin-olin-5-one (7q)

Isolated as an oil (44% yield) initially of ~95% purity by HPLC, but deteriorated overnight at room temperature. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.05 (s, 3H), 1.61 (s, 3H), 1.74-2.18 (m, 3H), 2.62 (dt, *J* = 6, 11Hz, 1H), 3.46–3.68 (m, 2H), 4.61 (dd, *J* = 5.5, 10 Hz, 1H), 7.14 (s, 1H), 7.20–7.30 (m, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.7 (CH_2), 22.8 (CH_3), 27.6 (CH_3), 32.3 (CH_2), 43.0 (C), 45.7 (CH_2), 57.8 (CH), 124.5 (CH), 126.1 (CH), 128.0 (CH), 132.3 (C), 137.7 (C), 141.1 (C), 172.2 (C).

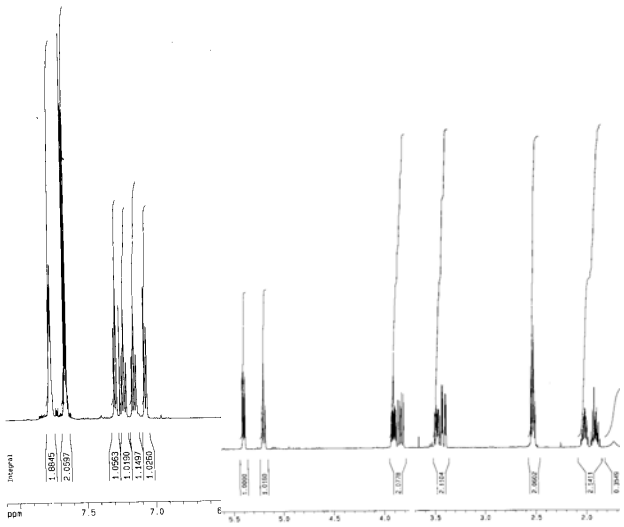


NMR of **2-Phenyl-1-(1'-phenylacetyl-2,3,4,5,4',5'-hexahydro-1'H-[2,3']bipyrrol-1-yl)ethanone (9):**

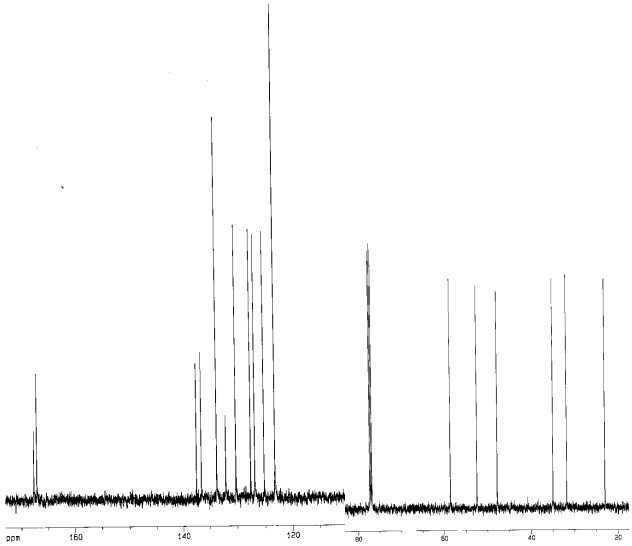


NMR of **2-((6S,11bS)-5-Oxo-2,3,5,6,7,11b-hexahydro-1H-benzo[c]pyrrolo[1,2-a]azepin-6-yl)-isoindole-1,3-dione (10)**

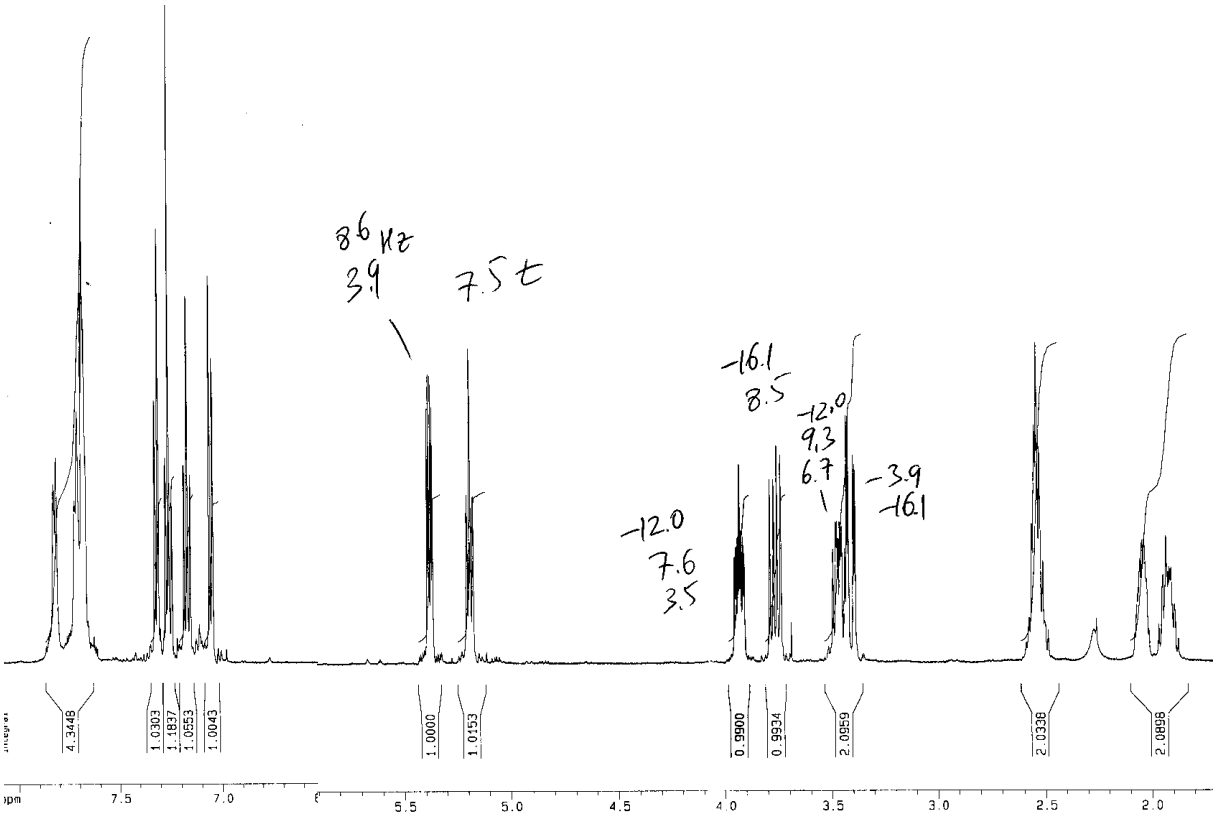
^{1}H NMR at 333K



13C NMR at 333K



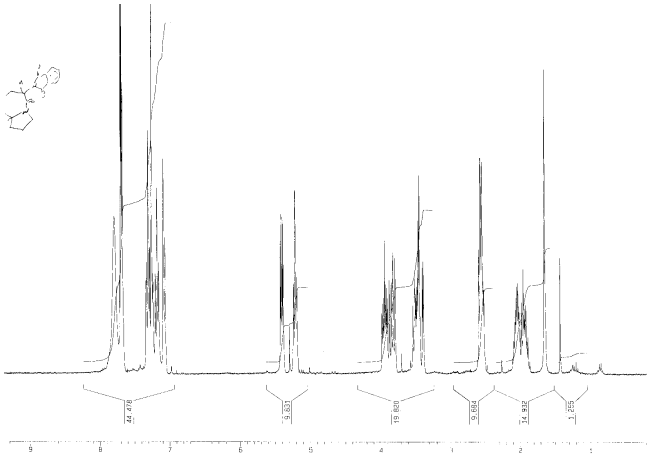
^{1}H NMR at 263K



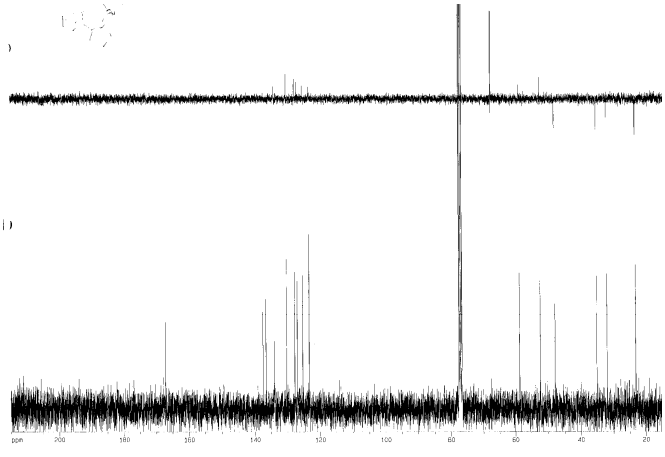
2-((6S,11bS),(6R,11bR)-5-Oxo-2,3,5,6,7,11b-hexahydro-1H-benzo[c]pyrrolo[1,2-a]azepin-6-yl)-isoindole-1,3-dione (10')

Isolated as a solid (Et_2O/petrol trituration, 80% yield) mpt. 91-4°C. ^{1}H NMR (300 MHz, $CDCl_3$): 1.80-2.15 (m, 2H), 2.48-2.62 (m, 2H), 3.36-3.56 (m, 2H), 3.75-3.98 (m, 2H), 5.21 (t, *J* = 7Hz, 1H), 5.40 (dd, *J* = 9, 4 Hz, 1H), 7.08 (d, *J* = 8 Hz, 1H), 7.18 (dt, *J* = 8, 1.5 Hz, 1H), 7.20 – 7.35 (m, 2H), 7.65-7.90 (m, 4H), ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 23.1, 31.9, 35.0, 47.9. 52.3, 58.6, 123.4, 125.3, 127.0, 127.7, 130.2, 133.9, 136.5, 137.4, 167.2 (phthalimide signals missing due to slow phthalimide rotation)

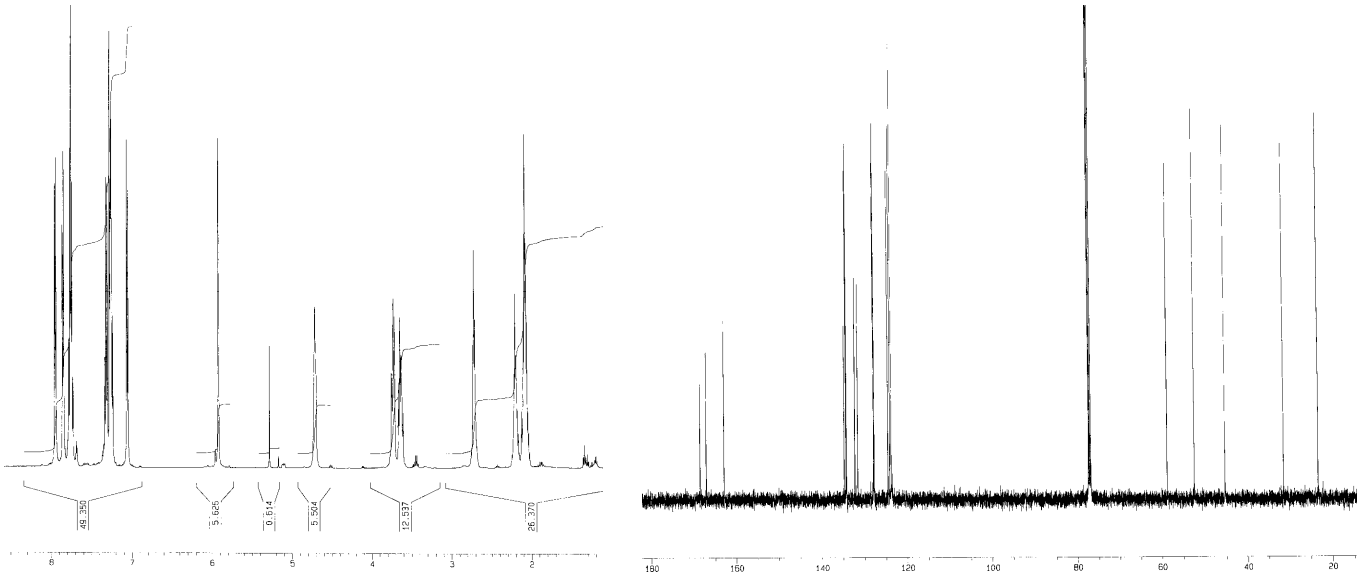
^{1}H NMR at 298K



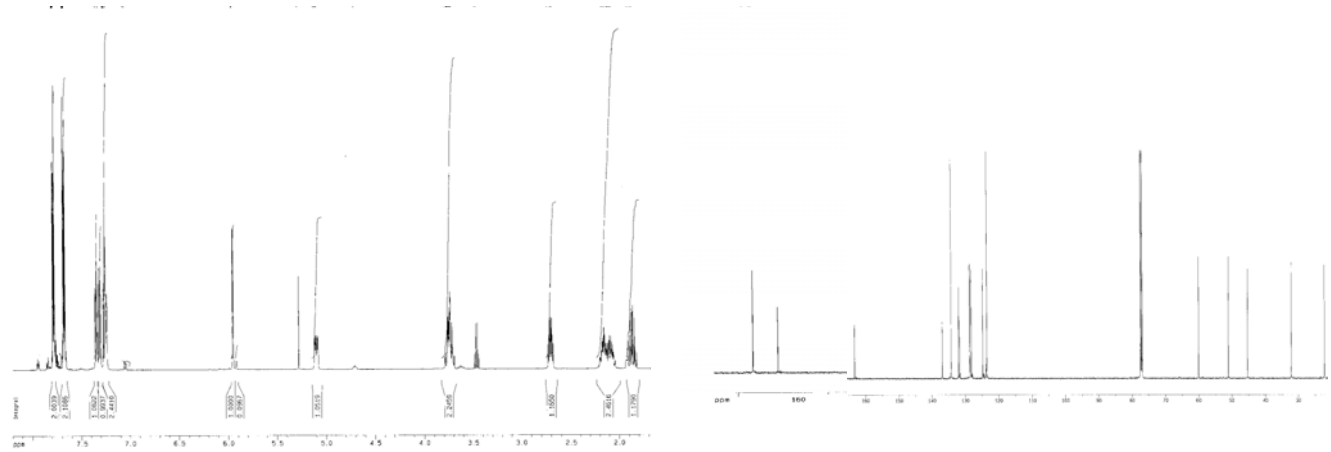
^{13}C NMR at 298K



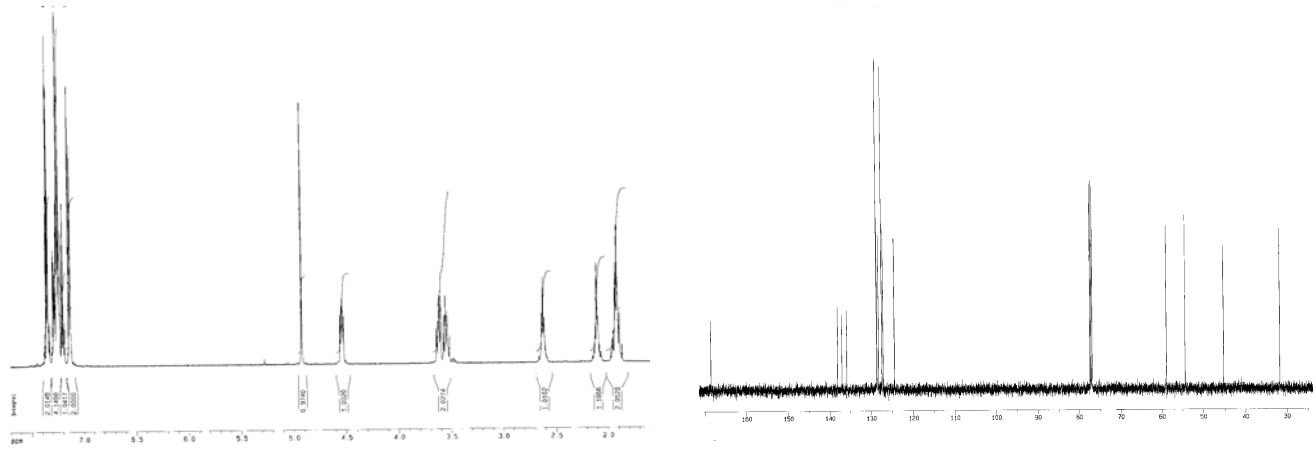
NMR of **2-((6s,10bR/6R10bS)-5-Oxo-1,2,3,6,10b-hexahydro-pyrrolo[2,1-a]isoquinolin-6-yl)isoindole-1,3-dione (11a)**



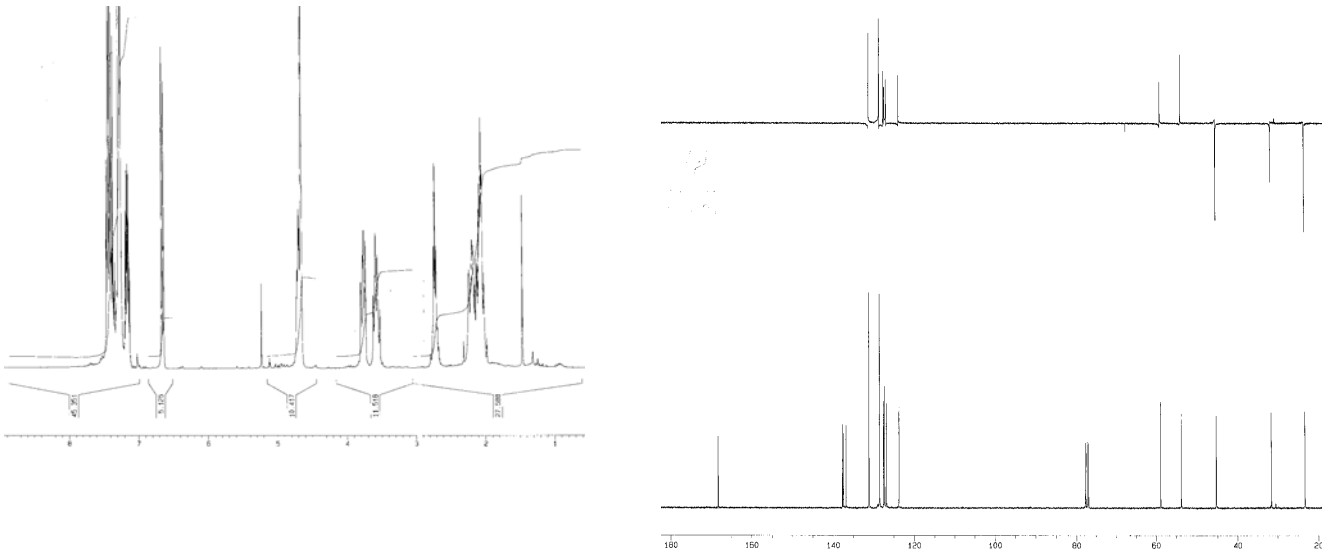
NMR of **2-((6R,10bR/6S10bS)-5-Oxo-1,2,3,6,10b-hexahydro-pyrrolo[2,1-a]isoquinolin-6-yl)isoindole-1,3-dione (11b)**



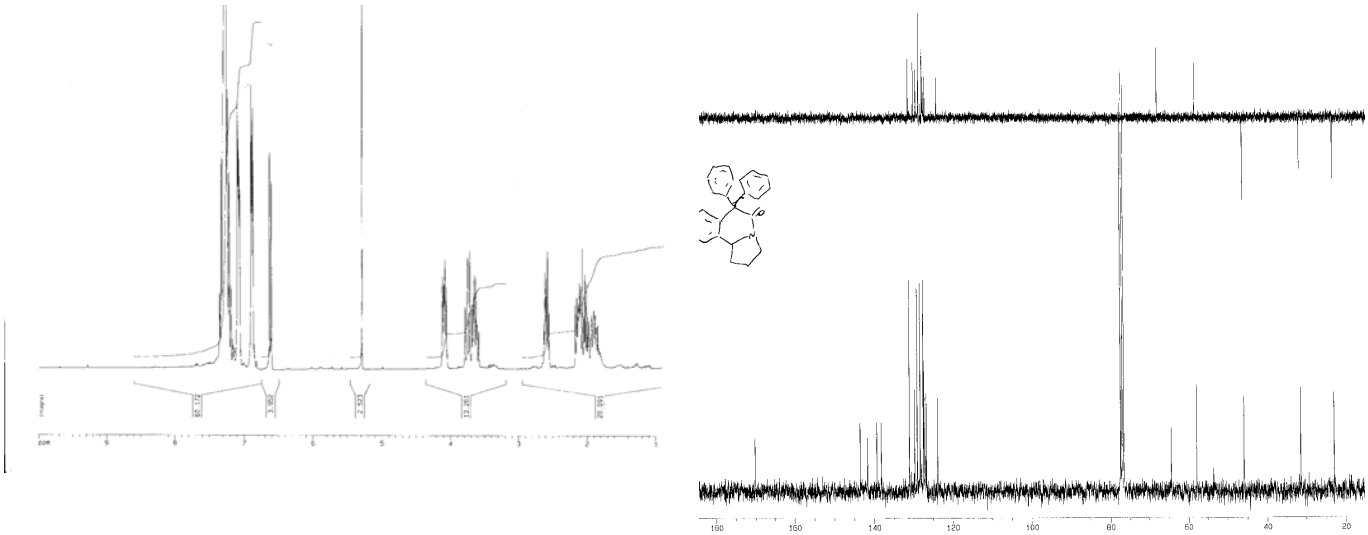
NMR of (6S, 10bS)/(6R, 10bR)-6-Phenyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquinolin-5-one (14a):



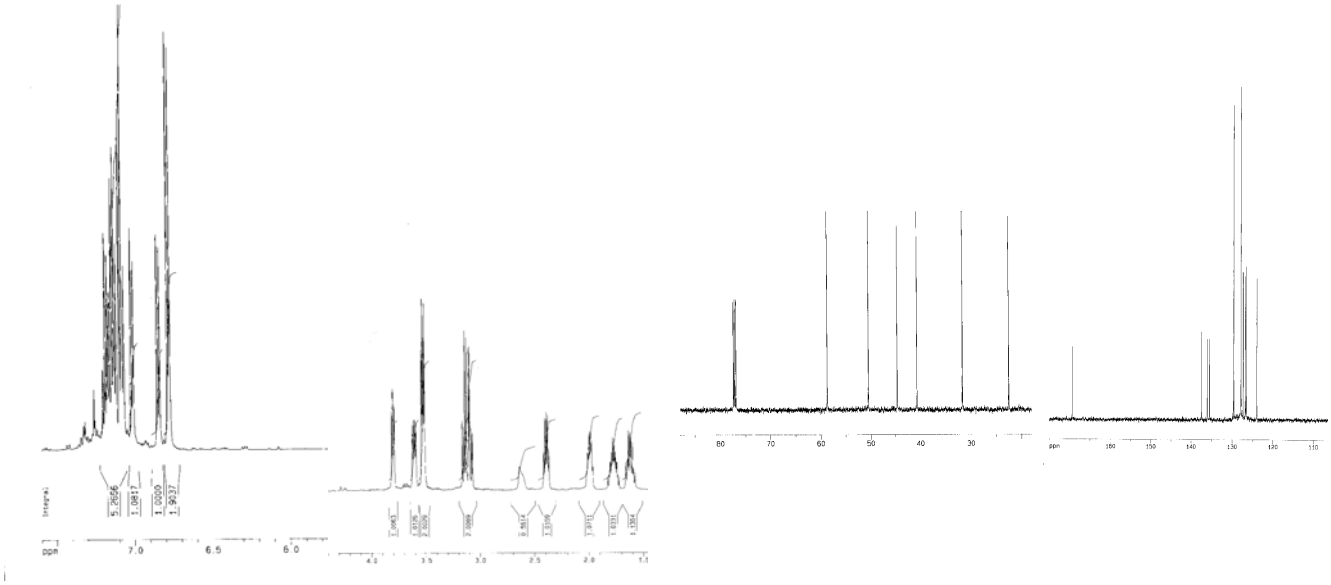
NMR of **(6S, 10bR)/(6R, 10bS)-6-Phenyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquinolin-5-one (14b)**



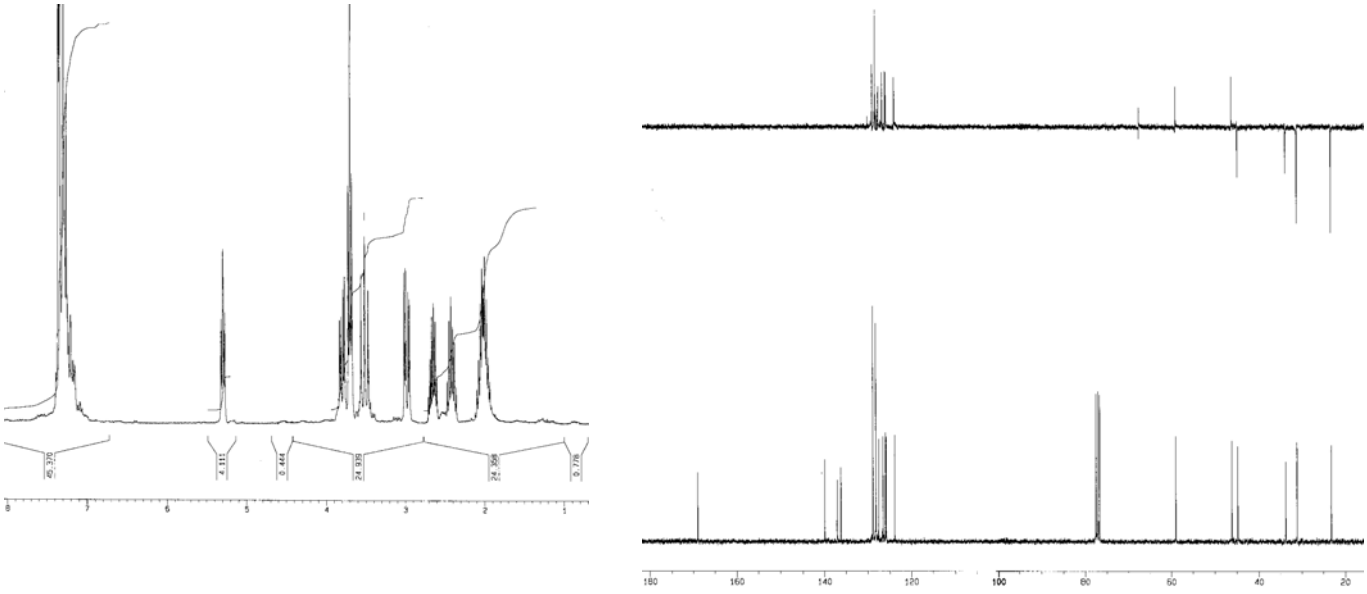
NMR of **6,6-Diphenyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]iso-quinolin-5-one (15)**



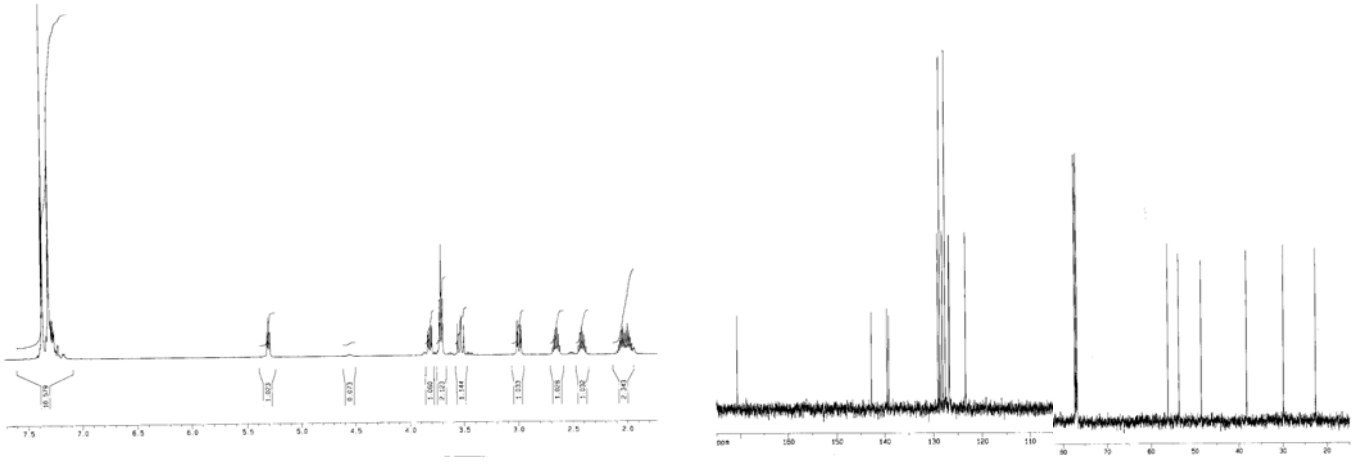
NMR of **(6R, 10bR)/(6S, 10bS)-6-Benzyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquinolin-5-one (16a)**



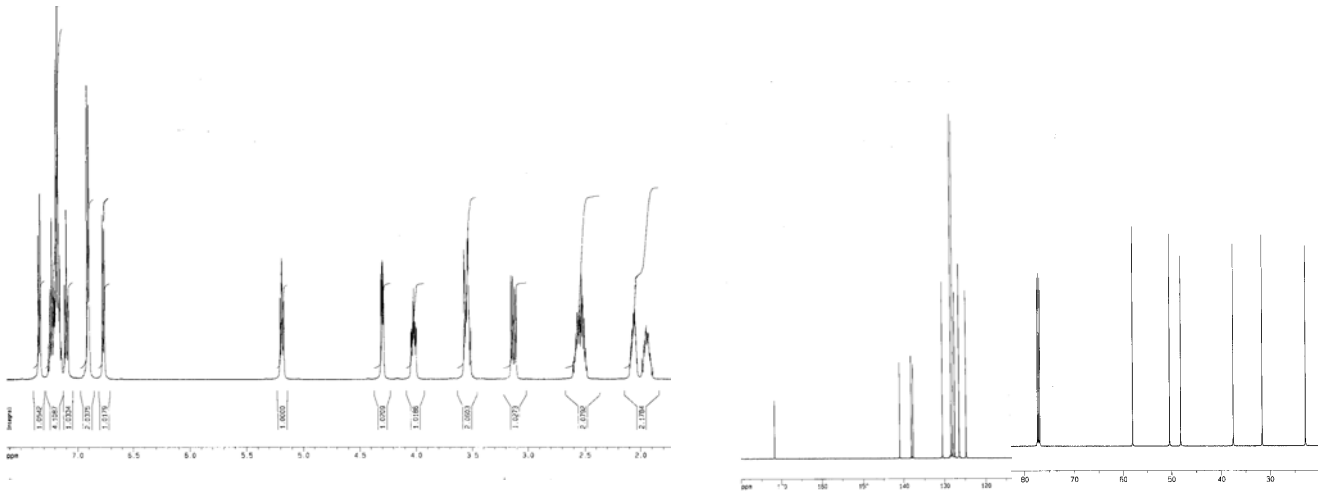
NMR of **(6S, 10bR)/(6R, 10bS)-6-Benzyl-2,3,6,10b-tetrahydro-1H-pyrrolo[2,1-a]isoquinolin-5-one (16b)**



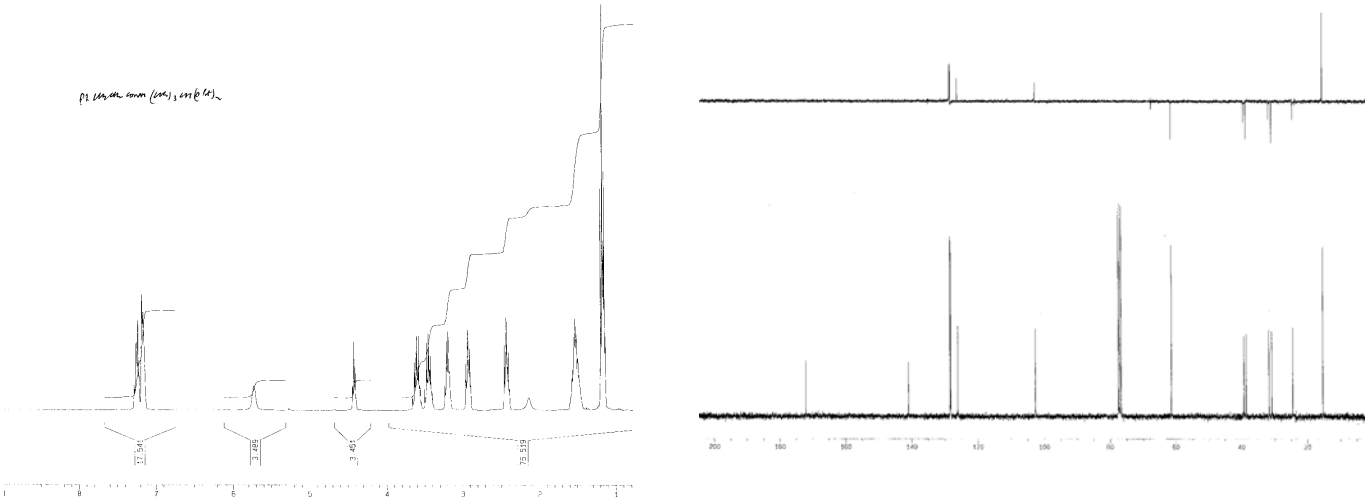
NMR of **(6S,11bR/6R,11bS)-6-Phenyl-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (17a)**



NMR of **(6S,11bS/6R,11bR)-6-Phenyl-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (17b)**

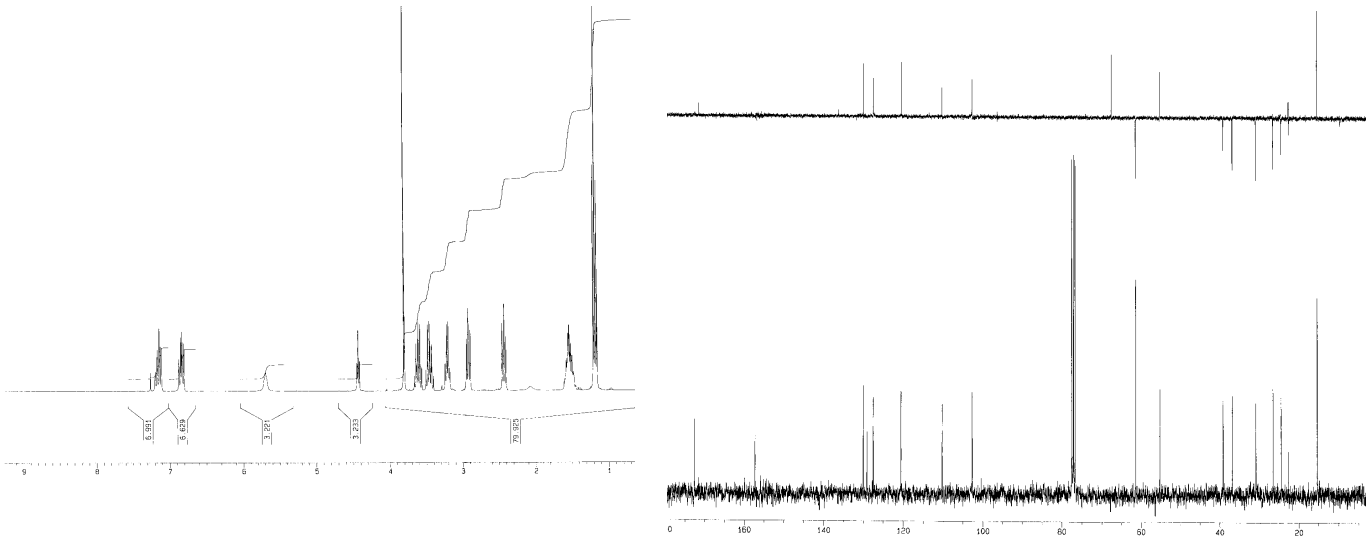


NMR of **N-(4,4-Diethoxybutyl)-3-phenylpropionamide (18a)**



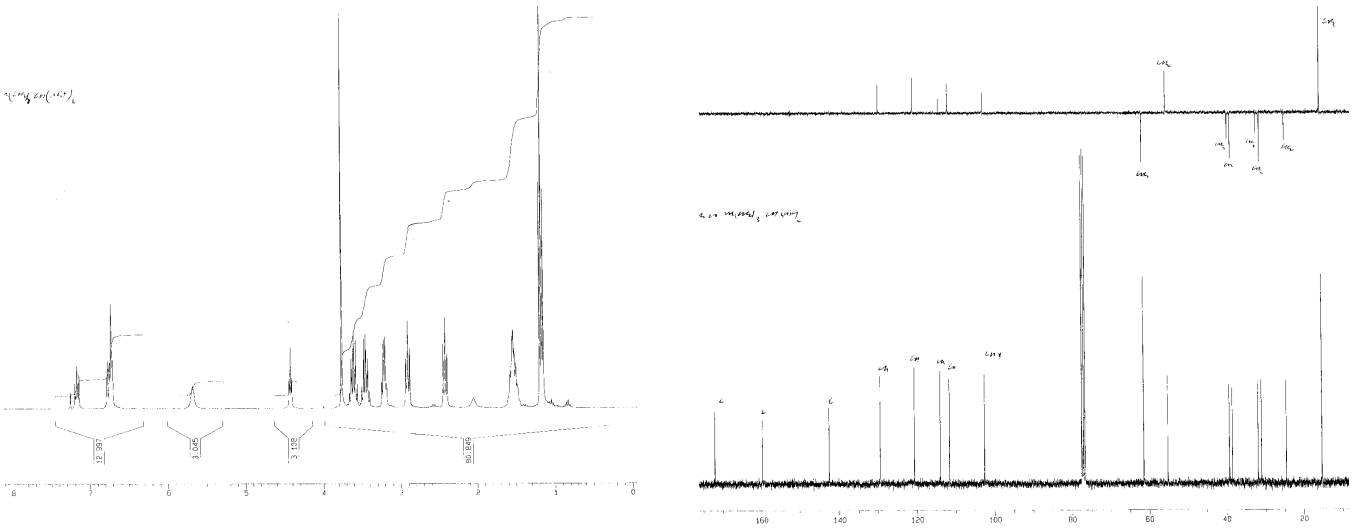
N-(4,4-Diethoxybutyl)-3-(2-methoxy-phenyl) propionamide (18b)

Isolated as an oil (100% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7.1 Hz, 6H), 1.44–1.62 (m, 4H), 2.43 (t, *J* = 7.5 Hz, 2H), 2.92 (t, *J* = 7.5 Hz, 2H), 3.20 (q, *J* = 6.3 Hz, 2H), 3.40–3.52 (m, 2H), 3.55–3.67 (m, 2H), 3.80 (s, 3H), 4.43 (t, *J* = 5 Hz, 1H). 5.69 (brs, 1H), 6.79–7.90 (m, 2H), 7.10–7.22 (m, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.6 (CH_2), 26.7 (CH_2), 31.0 (CH_2), 36.8 (CH_2), 39.2 (CH_2), 55.2 (CH_3), 61.4 (CH_2), 102.7 (CH), 110.2 (CH), 120.6 (CH), 127.5 (CH), 129.1 (C), 130.1 (CH), 157.3 (C), 172.6 (C).



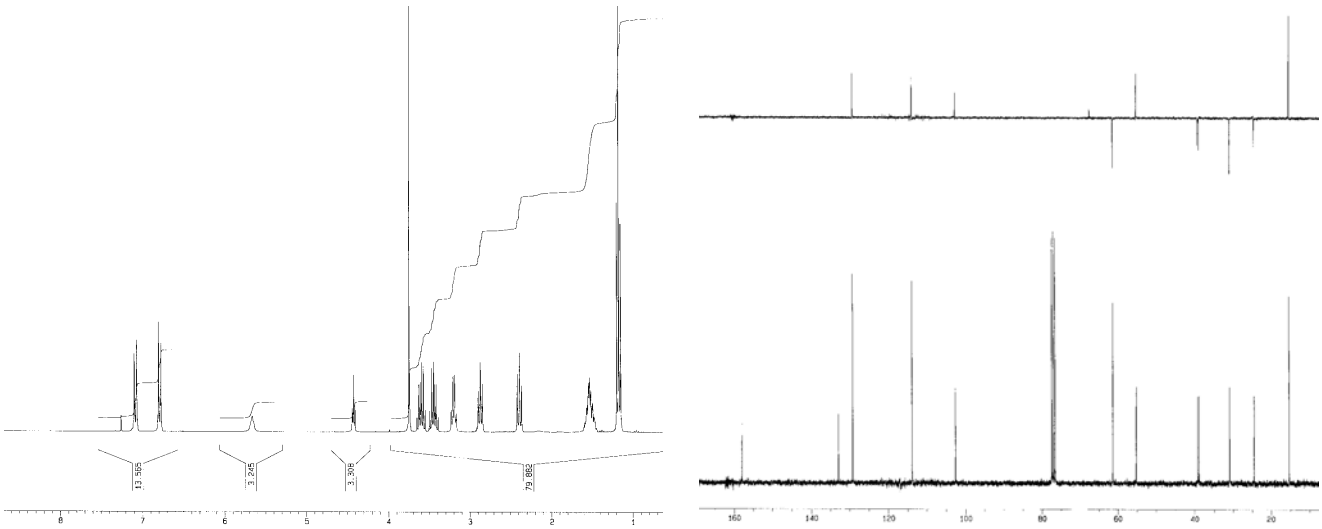
N-(4,4-Diethoxybutyl)-3-(3-methoxy-phenyl)propionamide (18c)

Isolated as an oil (100% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7 Hz, 6H), 1.42–1.60 (m, 4H), 2.42 (t, *J* = 8 Hz, 2H), 2.91 (t, *J* = 7 Hz, 2H), 3.21 (q, *J* = 7 Hz, 2H), 3.38–3.50 (m, 2H), 3.55–3.66 (m, 2H), 3.76 (s, 3H), 4.43 (t, *J* = 5 Hz, 1H), 5.68 (brs, 1H), 6.69–6.80 (m, 3H), 7.17 (t, *J* = 8 Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 31.0 (CH_2), 31.8 (CH_2), 38.5 (CH_2), 39.2 (CH_2), 55.1 (CH_3), 61.4 (CH_2), 102.6 (CH), 111.6 (CH), 114.0 (CH), 120.7 (CH), 129.5 (CH), 142.6 (C), 159.7 (C), 172.0 (C).



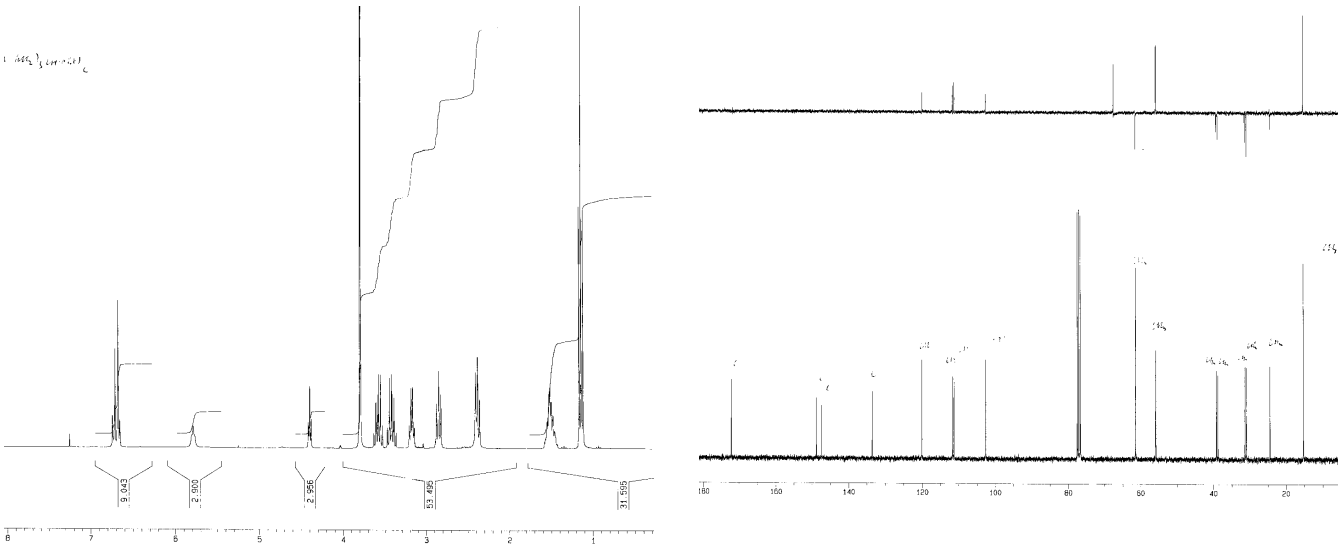
N-(4,4-Diethoxybutyl)-3-(4-methoxy-phenyl)propionamide (18d)

Isolated as an oil (98%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7 Hz, 6H), 1.42–1.60 (m, 4H), 2.39 (t, *J* = 8 Hz, 2H), 2.87 (t, *J* = 7 Hz, 2H), 3.20 (q, *J* = 7 Hz, 2H), 3.38–3.50 (m, 2H), 3.55–3.66 (m, 2H), 3.75 (s, 3H), 4.42 (t, *J* = 5 Hz, 1H), 5.67 (brs, 1H), 6.79 (d, *J* = 9 Hz, 2H), 7.08 (d, *J* = 9 Hz, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.6 (CH_2), 30.9 (CH_2), 31.0 (CH_2), 38.85 (CH_2), 39.2 (CH_2), 55.2 (CH_3), 61.4 (CH_2), 102.7 (CH), 113.9 (CH), 129.3 (CH), 133.0 (C), 158.0 (C), 172.1 (C); ν_{max}/cm^{-1} 3295, 2972, 1739, 1640, 1557, 1512, 1246, 1134, 1102, 1074, 1033, 1007, 823, 806.



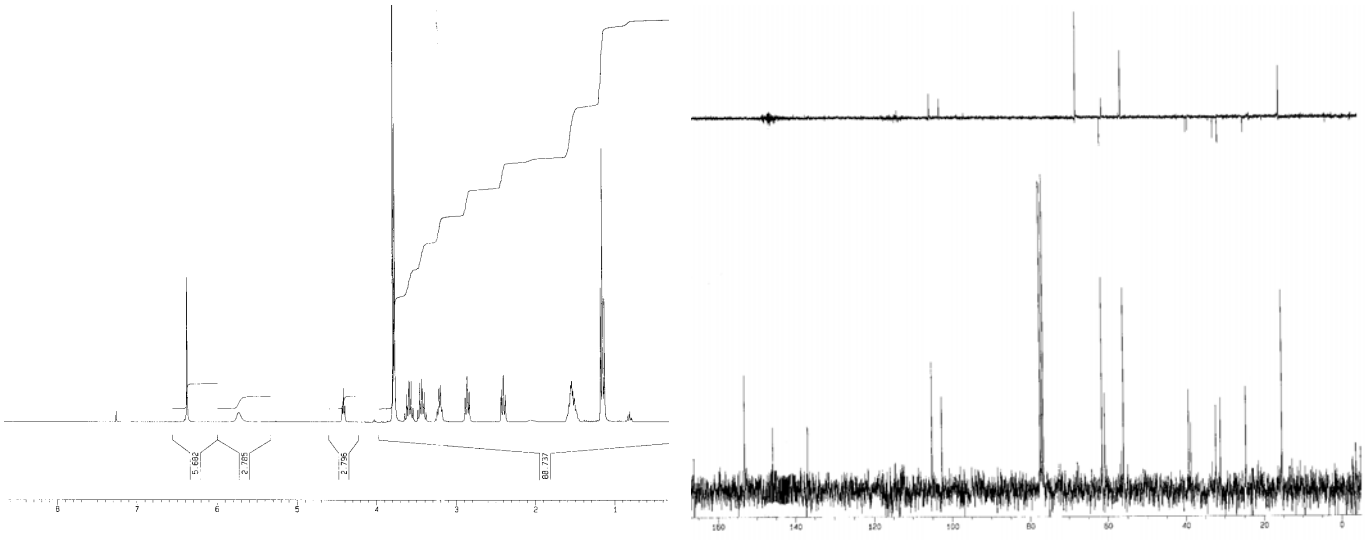
N-(4,4-Diethoxybutyl)-3-(3,4-di-methoxy-phenyl)propionamide (18e)

Isolated as an oil (100% yield); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7 Hz, 6H), 1.42–1.60 (m, 4H), 2.38 (t, *J* = 8 Hz, 2H), 2.85 (t, *J* = 7 Hz, 2H), 3.20 (q, *J* = 7 Hz, 2H), 3.36–3.48 (m, 2H), 3.52–3.64 (m, 2H), 3.79 (s, 3H), 3.80 (s, 3H), 4.40 (t, *J* = 5 Hz, 1H), 5.79 (brs, 1H), 6.65-6.77 (m, 3H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.9 (CH_2), 31.00 (CH_2), 31.4 (CH_2), 38.8 (CH_2), 39.2 (CH_2), 55.8 (CH_3), 55.9 (CH_3), 61.4 (CH_2), 102.6 (CH), 111.3 (CH), 111.7 (CH),120.1 (CH), 133.6 (C), 147.4 (C), 148.9 (C), 172.2 (C).



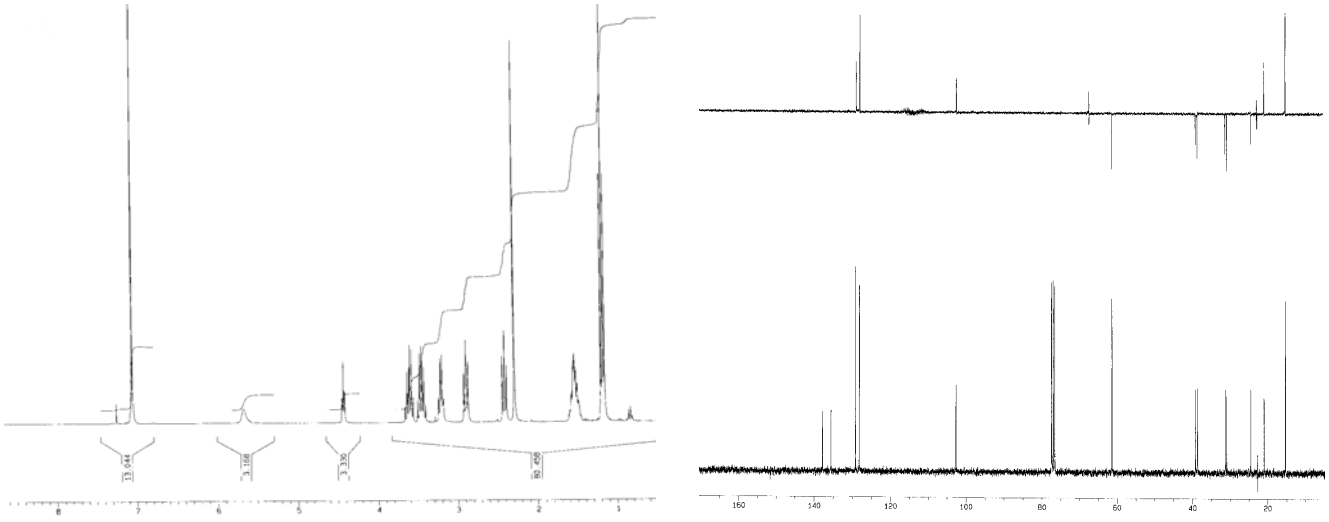
N-(4,4-Diethoxybutyl)-3-(3,4,5-tri-methoxy-phenyl)propionamide (18f).

Isolated as an oil (95% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16 (t, *J* = 7 Hz, 6H), 1.45–1.61 (m, 4H), 2.40 (t, *J* = 8.1 Hz, 2H), 2.86 (t, *J* = 8 Hz, 2H), 3.20 (q, *J* = 6 Hz, 2H), 3.39–3.52 (m, 2H), 3.55–3.68 (m, 2H), 3.77 (s, 3H), 3.79 (s, 6H), 4.42 (t, *J* = 5 Hz, 1H), 5.72 (brs, 1H), 6.41 (s, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 31.0 (CH_2), 32.2 (CH_2), 38.7 (CH_2), 39.3 (CH_2), 56.0 (CH_3), 60.8 (CH_3), 61.5 (CH_2), 102.7 (CH), 105.2 (CH), 136.8 (C), 145.8 (C), 153.2 (C), 171.9 (C); ν_{max}/cm^{-1} 2973, 2933, 1644, 1589, 1546, 1508, 1456, 1421, 1237, 1123, 1058, 1006, 824, 776.



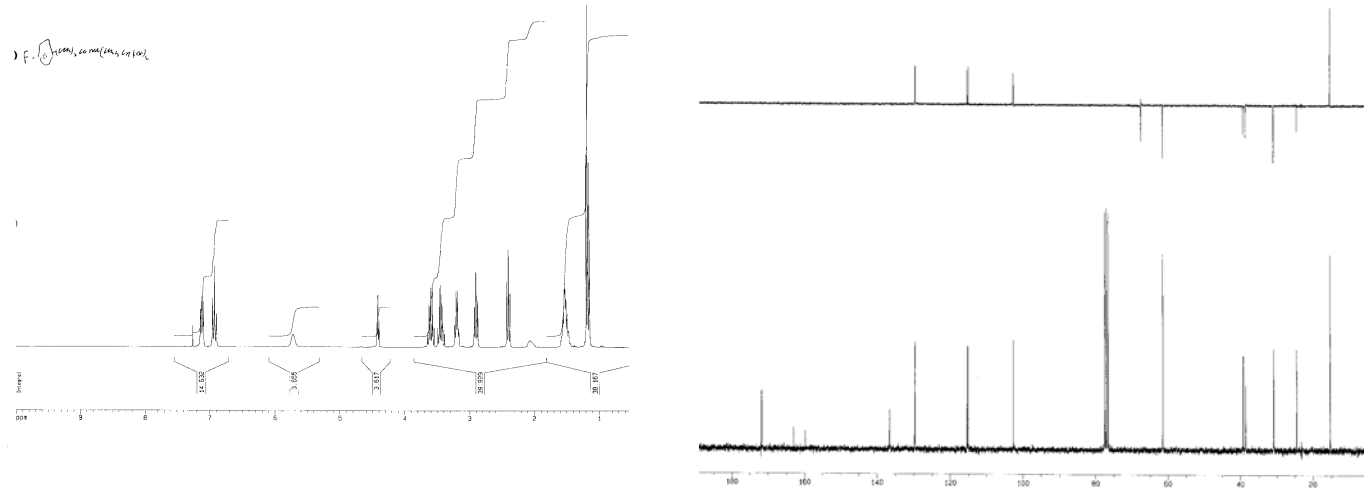
N-(4,4-Diethoxybutyl)-3-(4-methyl-phenyl)propionamide (18g)

Isolated as an oil (98% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7 Hz, 6H), 1.45–1.61 (m, 4H), 2.29 (s, 3H), 2.41 (t, *J* = 8 Hz, 2H), 2.90 (t, *J* = 8 Hz, 2H), 3.21 (q, *J* = 6 Hz, 2H), 3.39–3.52 (m, 2H), 3.55–3.68 (m, 2H), 4.33 (t, *J* = 5 Hz, 1H), 5.69 (brs, 1H), 7.07 (s, 4H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 21.0 (CH_3), 24.6 (CH_2), 31.0 (CH_2), 31.4 (CH_2), 38.7 (CH_2), 39.2 (CH_2), 61.4 (CH_2), 102.7 (CH), 128.2 (CH), 129.2 (CH), 135.6 (C), 137.9 (C), 172.1 (C).



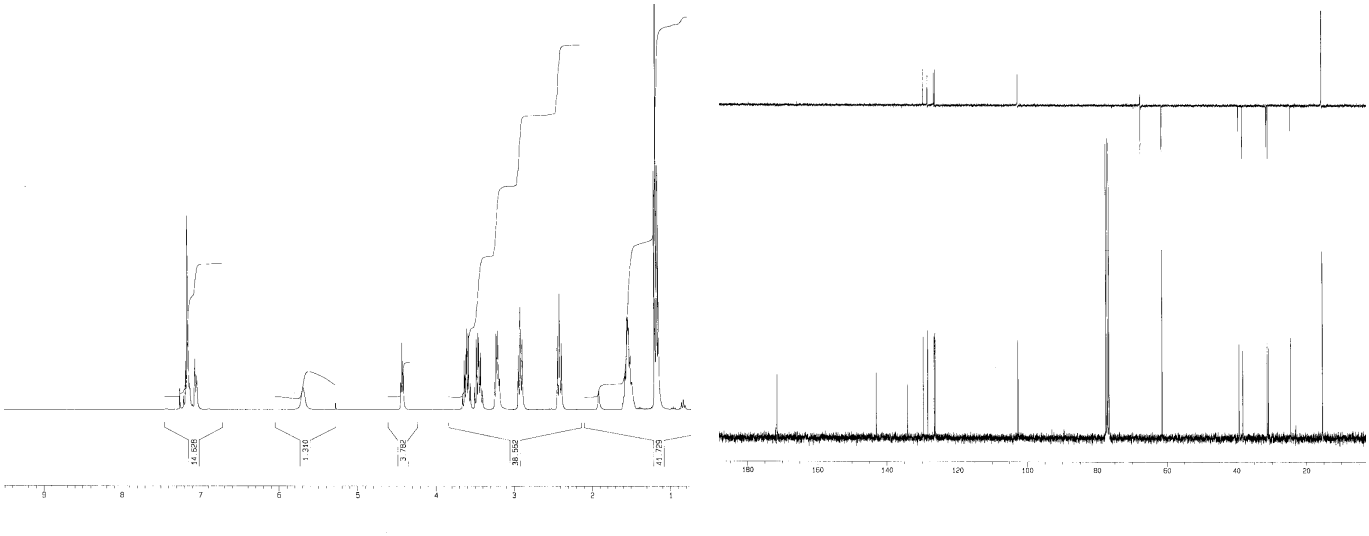
N-(4,4-Diethoxybutyl)-3-(4-fluoro-phenyl)propionamide (18h).

Isolated as an oil (95% yield) ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7Hz, 6H), 1.45–1.62 (m, 4H), 2.39 (t, *J* = 8Hz, 2H), 2.90 (t, *J* = 8Hz, 2H), 3.21 (q, *J* = 6Hz, 2H), 3.38–3.51 (m, 2H), 3.54–3.67 (m, 2H), 4.42 (t, *J* = 5Hz, 1H), 5.72 (brs, 1H), 6.92 (t, *J* = 8.5 Hz, 2H), 7.12 (dd, *J* = 8.5, 5.5 Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.9 (CH_2), 31.0 (CH_2), 38.6 (CH_2), 39.2 (CH_2), 61.5 (CH_2), 102.6 (CH), 115.2 (CH, J_{C-F} = 20 Hz), 129.7 (CH, J_{C-F} = 8 Hz), 136.6 (C, J_{C-F} = 3 Hz), 161.4 (C, J_{C-F} = 240 Hz), 171.8 (C)



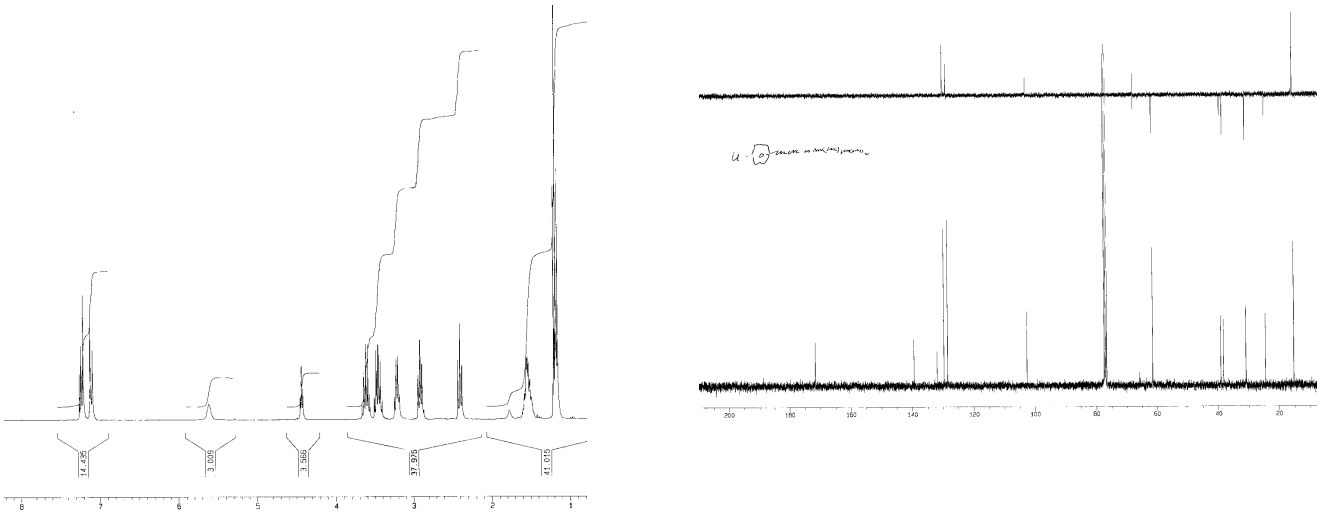
N-(4,4-Diethoxybutyl)-3-(3-chloro-phenyl)propionamide (18k)

Isolated as an oil (100% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7Hz, 6H), 1.45–1.62 (m, 4H), 2.41 (t, *J* = 8Hz, 2H), 2.92 (t, *J* = 8Hz, 2H), 3.21 (q, *J* = 6Hz, 2H), 3.38–3.51 (m, 2H), 3.54–3.67 (m, 2H), 4.43 (t, *J* = 5Hz, 1H), 5.69 (brs, 1H), 7.06 (d, *J* = 6.7 Hz, 1H), 7.10–7.22 (m, 3H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 30.9 (CH_2), 31.2 (CH_2), 38.1 (CH_2), 39.3 (CH_2), 61.5 (CH_2), 102.7 (CH), 126.4 (CH), 126.6 (CH), 128.5 (CH), 129.7 (CH), 134.2 (C), 143.0 (C), 171.5 (C); ν_{max}/cm^{-1} 2974, 2930, 2876, 1642, 1550, 1125, 1058, 997, 781, 680.



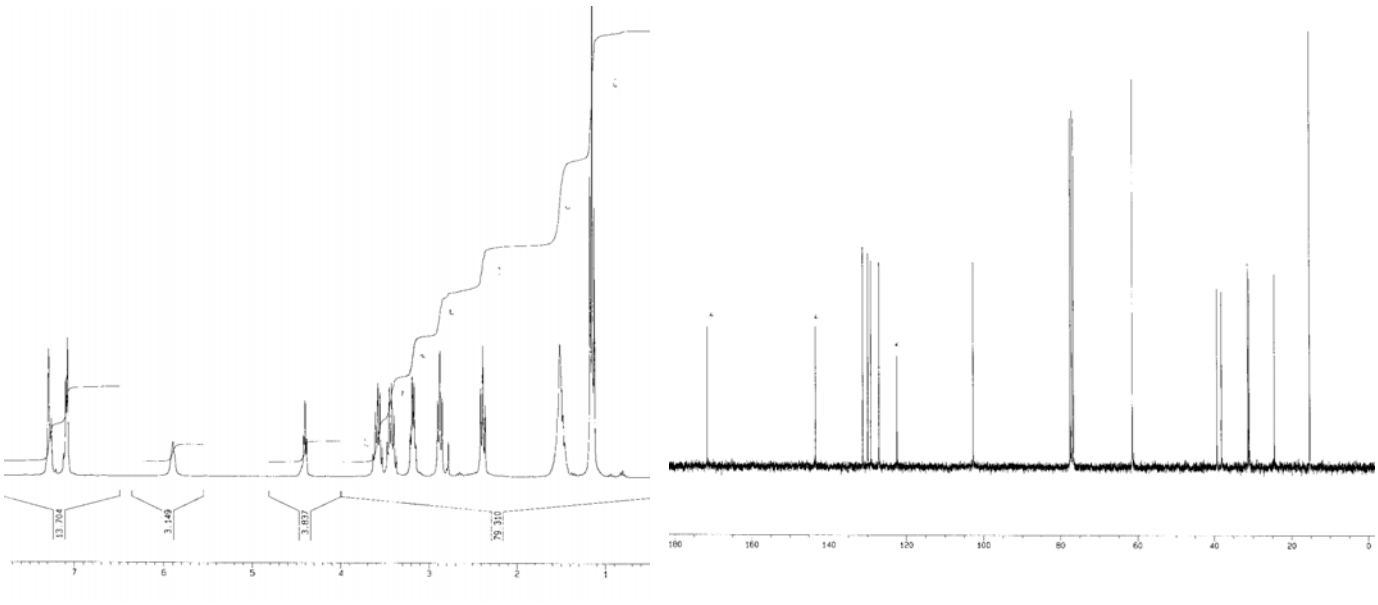
N-(4,4-Diethoxybutyl)-3-(4-chloro-phenyl)propionamide (18l)

Isolated as an oil (86%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.19 (t, *J* = 7Hz, 6H), 1.45-1.63 (m, 3H), 2.40 (t, *J* = 7.5 Hz, 2H), 2.91 (t, *J* = 7.5 Hz, 2H), 3.21 (q, *J* = 6 Hz, 2H), 3.40 – 3.52 (m, 2H), 3.56 – 3.70 (m, 2H), 4.43 (t, *J* = 5 Hz, 1H), 5.62 (brs, 1H), 7.14 (d, *J* = 7 Hz, 2H), 7.23 (d, *J* = 7Hz, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 31.0 (CH_2), 31.0 (CH_2), 38.3 (CH_2), 39.2 (CH_2), 61.5 (CH_2), 102.6 (CH), 128.6 (CH), 129.7 (CH), 132.0 (C), 139.4 (C), 171.6 (C).



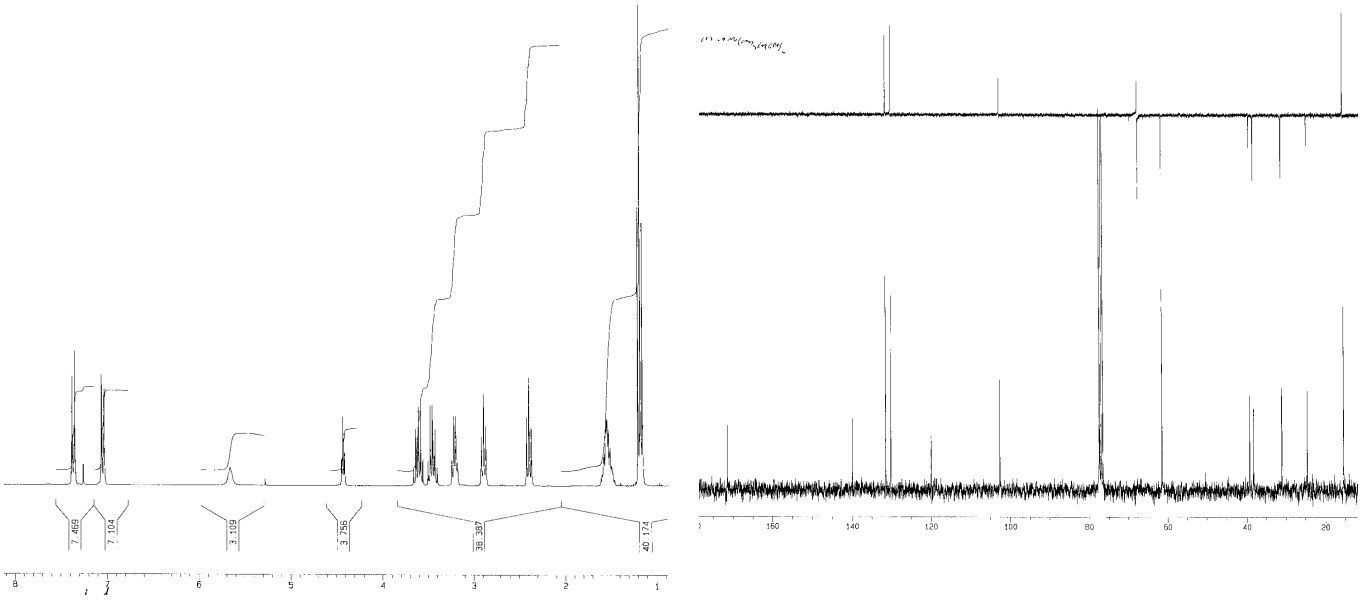
N-(4,4-Diethoxybutyl)-3-(3-bromo-phenyl)propionamide (18i)

Isolated as an oil (98% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7 Hz, 6H), 1.42–1.63 (m, 4H), 2.38 (t, *J* = 8Hz, 2H), 2.88 (t, *J* = 7.5Hz, 2H), 3.18 (q, *J* = 6 Hz, 2H), 3.36-3.50 (m 2H), 3.53-3.66 (m, 2H), 4.40 (t, *J* = 5 Hz, 1H), 5.90 (brs, 1H), 7.03-7.12 (m, 2H), 7.20–7.32 (m, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.5 (CH_2), 31.0 (CH_2), 31.3 (CH_2), 38.1 (CH_2), 39.2 (CH_2), 61.4 (CH_2), 102.6 (CH), 122.4 (C), 127.1 (CH), 129.3 (CH), 130.1 (CH), 131.4 (CH), 143.4 (C), 171.6 (C).

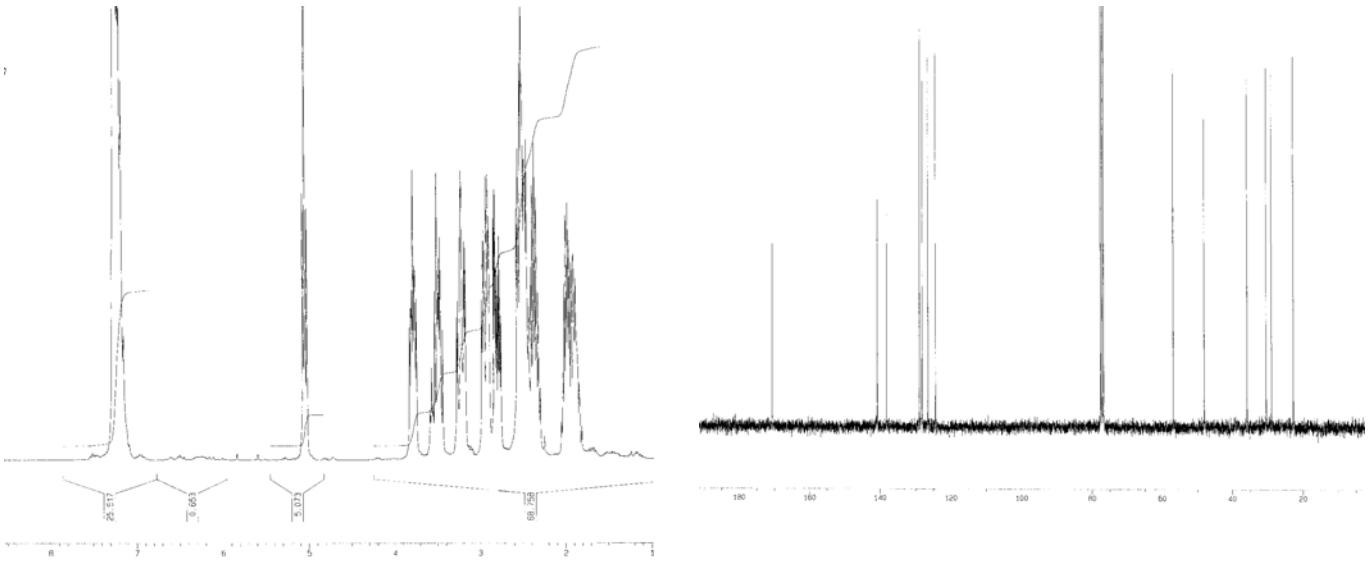


N-(4,4-Diethoxybutyl)-3-(4-bromo-phenyl)propionamide (18j)

Isolated as an oil which solidified on standing (87% yield), mpt 55-8°C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17 (t, *J* = 7Hz, 6H), 1.45–1.62 (m, 4H), 2.39 (t, *J* = 8Hz, 2H), 2.88 (t, *J* = 8Hz, 2H), 3.20 (q, *J* = 6Hz, 2H), 3.38–3.51 (m, 2H), 3.54–3.67 (m, 2H), 4.43 (t, *J* = 5Hz, 1H), 5.67 (brs, 1H), 7.08 (d, *J* = 7 Hz, 2H), 7.36 (d, *J* = 7 Hz, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 15.4 (CH_3), 24.5 (CH_2), 31.0 (CH_2), 31.1 (CH_2), 38.2 (CH_2), 39.2 (CH_2), 61.5 (CH_2), 102.6 (CH), 120.0 (C), 130.2 (CH), 131.5 (CH), 140.0 (C), 171.6 (C); ν_{max}/cm^{-1} 3306, 2971, 1638, 1543, 1489, 1147, 1132, 1080, 1070, 1038, 1012, 995, 811, 716, 691.

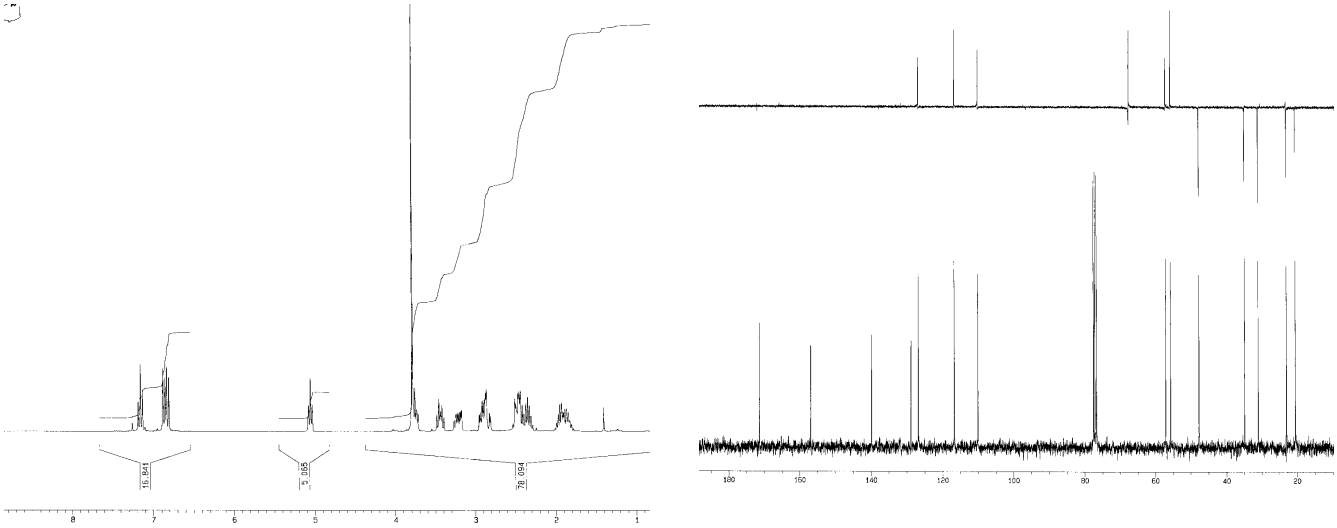


NMR of **1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19a)**



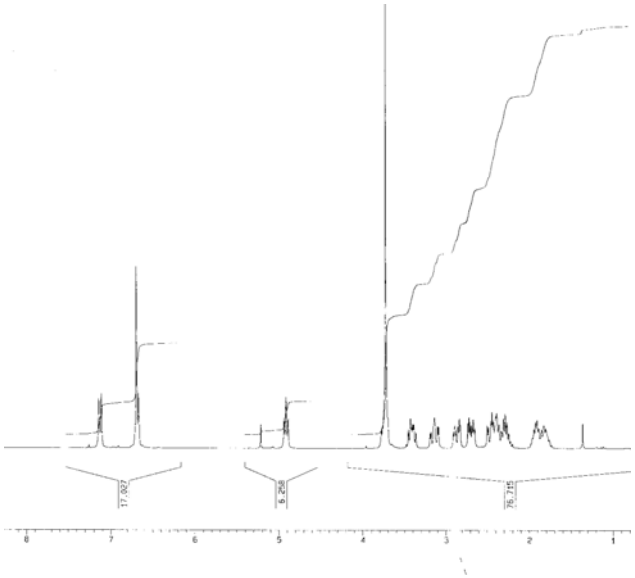
8-Methoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19b)

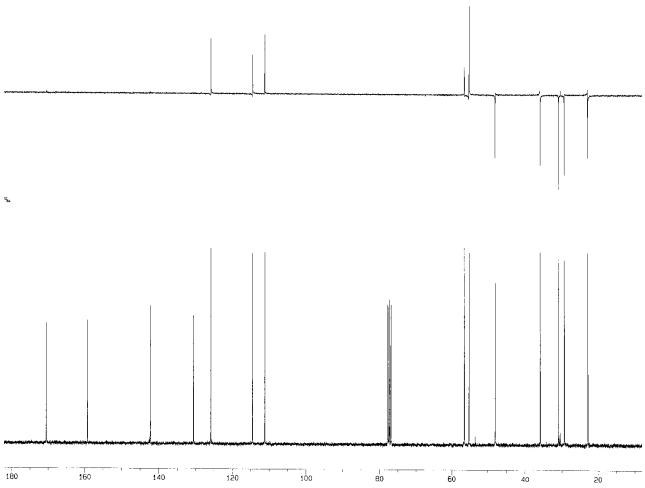
Isolated as an oil (13% yield), HRMS Theory 231.1254, Found 231.1254. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.76–2.02 (m, 2H), 2.22–2.55 (m, 3H), 2.78–2.95 (m, 2H), 3.15–3.29 (m, 1H), 3.36–3.50 (m, 1H), 3.70–3.81 (m, 4H including 3.79, s, 3H), 5.06 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 8Hz, 1H), 6.87 (d, *J* = 8 Hz, 1H), 7.16 (t, *J* = 8 Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 20.5 (CH_2), 23.0 (CH_2), 31.1 (CH_2), 34.9 (CH_2), 47.7 (CH_2), 55.7 (CH_3), 57.1 (CH), 110.0 (CH), 116.7 (CH), 126.8 (CH), 128.8 (C), 139.8 (C), 156.8 (C), 171.3 (C); ν_{max}/cm^{-1} 2972, 1611, 1585, 1474, 1400, 1267, 1089, 769, 742, 704.



9-Methoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19c)

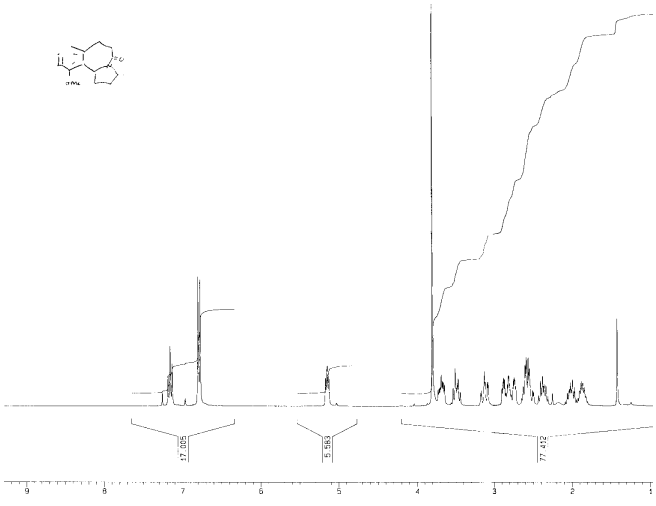
Isolated as the more polar isomer from the SiO2 column, eluting with Et_2O – 4% MeOH, as a white solid (68% yield), m. pt. 116-8°C (Et_2O/petrol). HRMS Theory 231.1254, Found 231.1257. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.73-2.01 (m, 2H), 2.18–2.51 (m, 3H), 2.62–2.75 (m, 1H), 2.80-2.91 (m, 1H), 3.05–3.20 (m, 1H), 3.32-3.45 (m, 1H), 3.72 (s, 3H), 4.91 (t, *J* = 7 Hz, 1H), 7.64-7.72 (m, 2H including 6.69, s), 7.13 (d, *J* = 9Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.8 (CH_2), 29.3 (CH), 30.9 (CH_2), 35.9 (CH_2), 48.00 (CH_2), 55.2 (CH_3), 56.6 (CH), 111.2 (CH), 114.5 (CH), 125.8 (CH), 130.5 (C), 142.3 (C), 159.2 (C), 170.5 (C).

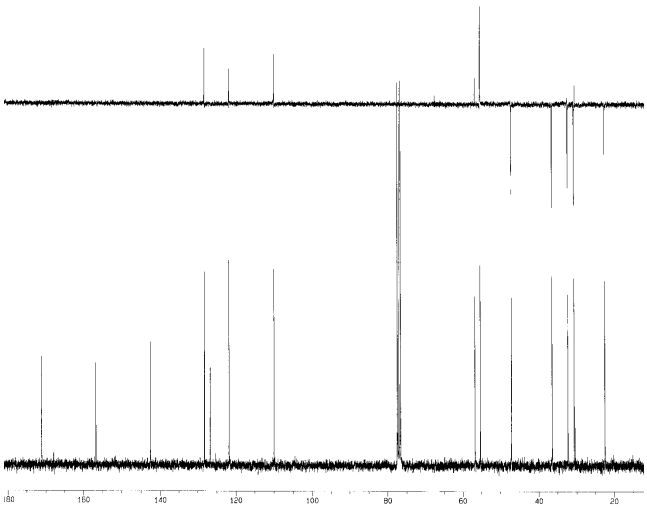




11-Methoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19c')

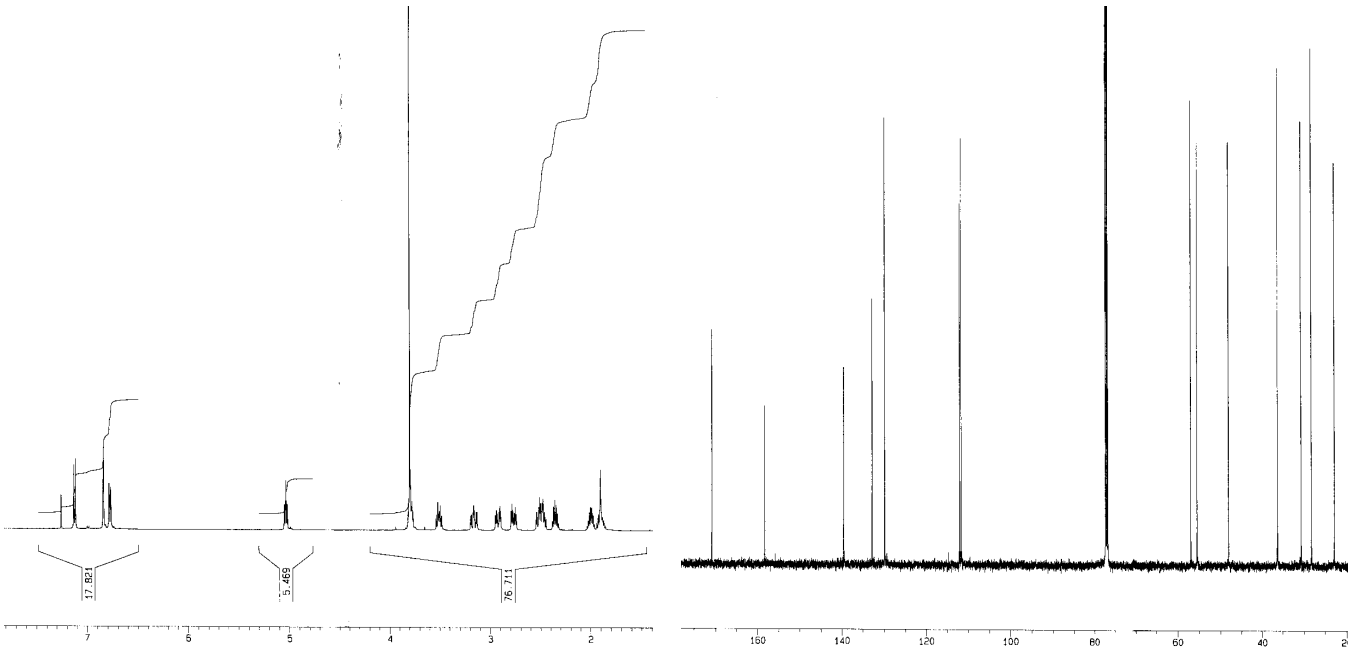
Isolated as the less polar isomer from the SiO_2 column, eluting with Et_2O – 2% MeOH, as an oil (68% yield), HRMS Theory 231.1254, Found 231.1259. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.78-2.11 (m, 2H), 2.30–2.45 (m, 1H), 2.49–2.66 (m, 2H), 2.70–2.91 (m, 2H), 3.04-3.19 (m, 1H), 3.41-3.55 (m, 1H), 3.61–3.74 (m, 1H), 3.80 (s, 3H), 5.15 (t, *J* = 7 Hz, 1H), 6.79 (d, *J* = 8 Hz, 2H), 7.16 (t, *J* = 8 Hz, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.4 (CH_2), 30.6 (CH_2), 32.3 (CH_2), 36.5 (CH_2), 47.1 (CH_2), 55.3 (CH_3), 56.7 (CH), 110.0 (CH), 121.8 (CH), 126.8 (C), 128.3 (CH), 142.6 (C), 156.7 (C), 171.1 (C); ν_{max}/cm^{-1} 1617, 1609, 1580, 1477, 1433, 1399, 1265, 1072, 827, 790, 774, 734.





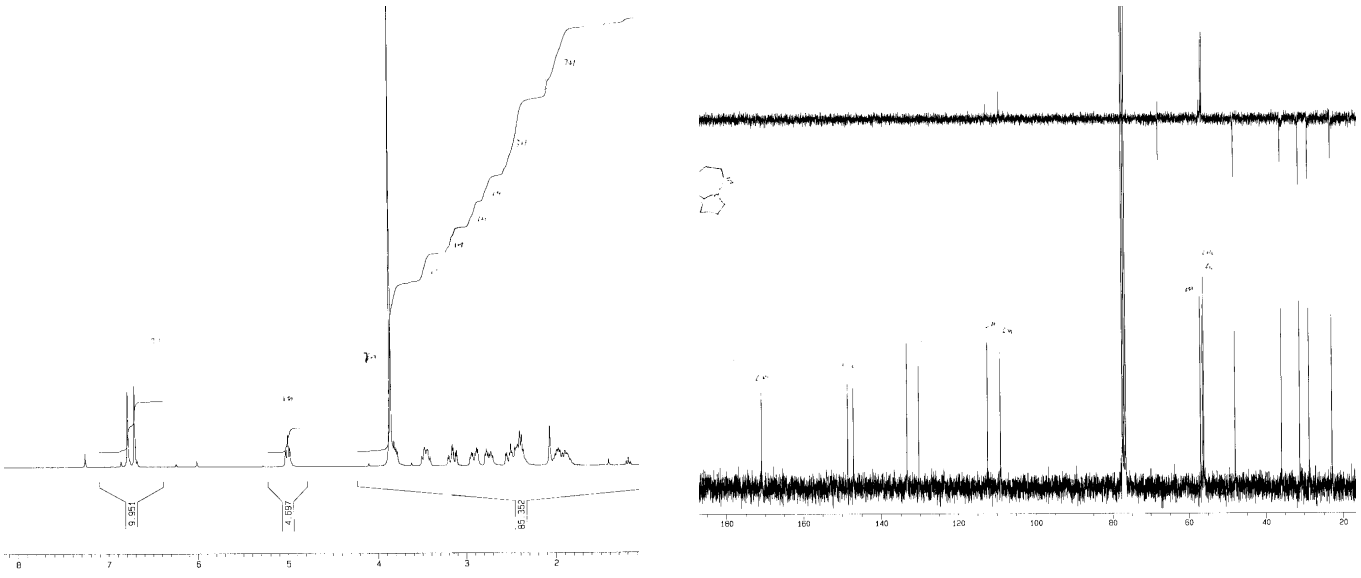
10-Methoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19d)

Isolated as a white solid, mp. 78-80°C; HRMS Theory 231.1254, Found 231.1257. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.85-1.98 (m, 1H), 2.00 – 2.05 (m, 1H)), 2.35 (tt, *J* = 6.1, 12.3 Hz, 1H), 2.44–2.55 (m, 2H), 2.77 (dt, *J* = 14.9, 4.7 Hz, 1H), 2.92 (ddd, *J* = 4.2, 5.5, 17.1 Hz, 1H), 3.16 (ddd, *J* = 3.9, 12.5, 15.7 Hz, 1H), 3.51 (dt, *J* = 7.5, 14.9 Hz, 1H), 3.78-3.84 (m, 4H including 3.80, s, 3H), 5.04 (t, *J* = 7.1 Hz, 1H), 6.78 (dd, *J* = 2.6, 8.2 Hz, 1H), 6.84 (d, *J* = 2.6 Hz, 1H), 7.12 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.9 (CH_2), 28.3 (CH_2), 30.7 (CH_2), 36.3 (CH_2), 48.1 (CH_2), 55.5 (CH_3), 56.9(CH), 111.7 (CH), 112.1 (CH), 129.9 (CH), 132.9 (C), 139.6 (C), 158.3 (C), 170.9 (C); ν_{max}/cm^{-1} 2971, 1610, 1450, 1438, 1404, 1351, 1305, 1237, 1184, 1027, 835, 814, 748, 693.



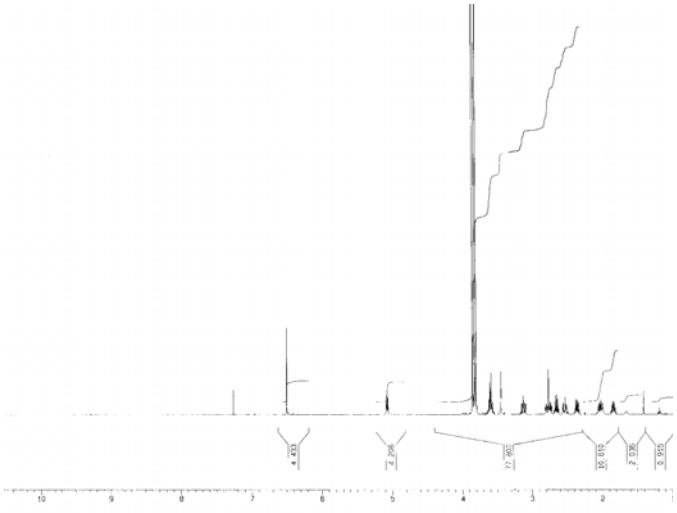
9,10-Dimethoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo-[1,2-a]azepin-5-one (19e)

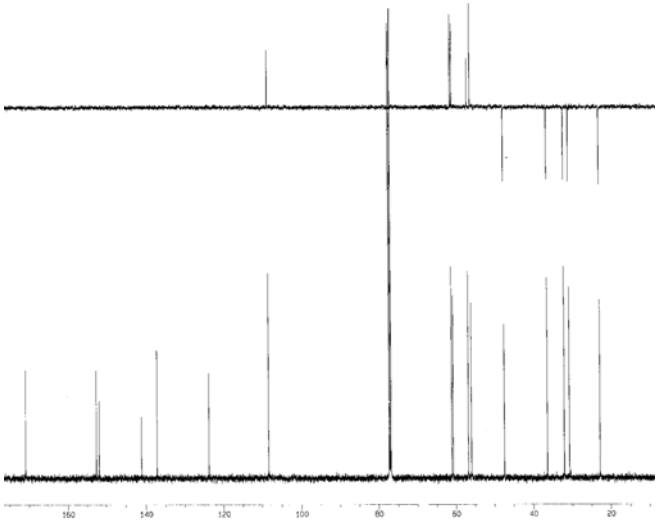
Isolated as a white solid (86% yield), m. pt. 98-102°C (Et_2O/petrol) HRMS Theory 261.1359, Found 261.1357. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.78-2.05 (m, 2H), 2.26-2.56 (m, 3H), 2.66-2.79 (m, 1H), 2.84-2.97 (m, 1H), 3.07–3.23 (m, 1H), 3.39–3.52 (m 1H), 3.75 – 3.88 (m, 7H including 3.86, s, 6H), 5.00 (t, *J* = 7Hz, 1H), 6.71 (s, 1H), 6.79 (s, 1H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.9 (CH_2), 28.7 (CH_2), 31.1 (CH_2), 35.9 (CH_2), 47.9 (CH_2), 56.0 (CH_3), 56.3 (CH_3), 56.9 (CH), 109.1 (CH), 112.4 (CH), 130.3 (C), 133.3 (C), 147.1 (C), 148.6 (C), 171.0 (C); ν_{max}/cm^{-1} 2940, 1603, 1519, 1461, 1440, 1406, 1279, 1210, 1159, 1113, 1091, 1002, 861, 765.



9,10,11-Trimethoxy-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19f)

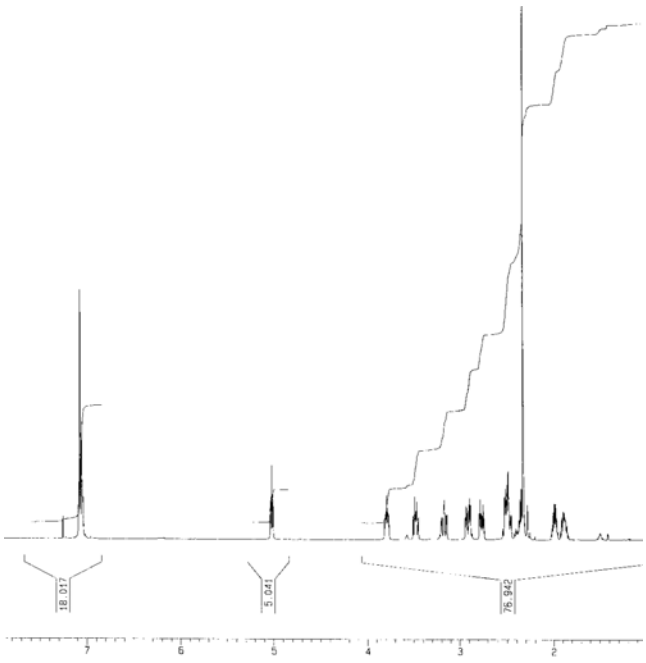
Isolated as a solid (90% yield), mpt 90-92°C (Et_2O/petrol), HRMS Theoretical Mass: 291.1465 Measured Mass: 291.1473. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.80–1.89 (m, 1H), 1.97–2.10 (m, 1H), 2.33–2.42 (m, 1H), 2.49–2.57 (m, 1H), 2.62–2.70 (m, 1H), 2.70-2.83 (m, 2H), 3.12 (m, 1H), 3.45 (brs, 1H), 3.55–3.64 (m, 2H), 3.81 (s, 3H), 3.83 (s, 3H), 3.86 (s, 3H), 5.07 (t, *J* = 6.7 Hz, 1H), 6.50 (s, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.8 (CH_2), 30.7 (CH_2), 32.0 (CH_2), 36.4 (CH_2), 47.4 (CH_2), 56.0 (CH_3), 56.8 (CH), 60.7 (CH_3), 61.1 (CH_3), 108.3 (CH), 123.8 (C), 137.1 (C), 141.1 (C), 151.9 (C), 152.6 (C), 170.9 (C); ν_{max}/cm^{-1} 2972, 2946, 2869, 1612, 1598, 1433, 1400, 1351, 1313, 1302, 1241, 1120, 1080, 1025, 996, 917, 853, 824, 778, 713.

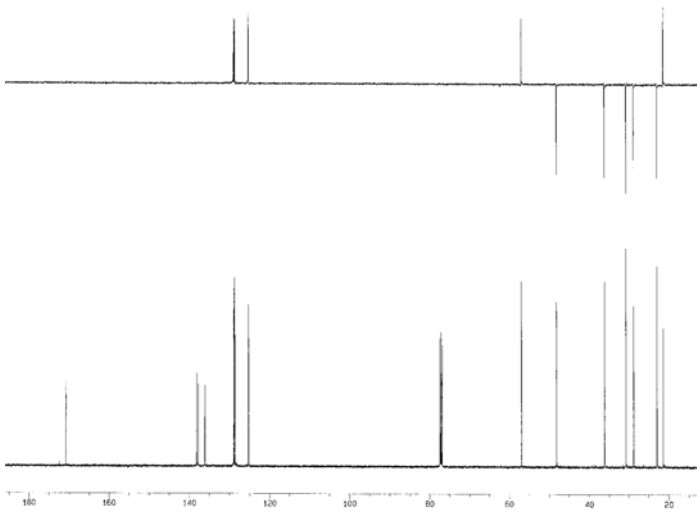




10-Methyl-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19g)

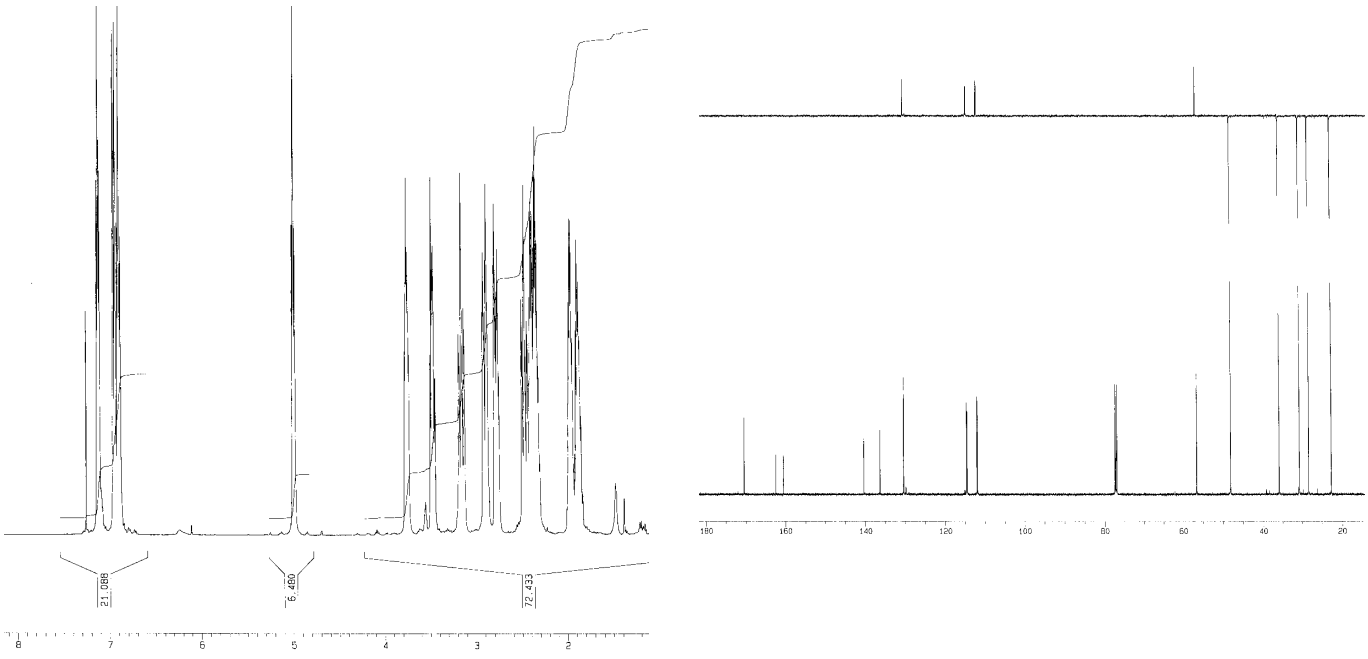
Isolated as an oil which solidifed on standing (75% yield), m. pt. 55-58°C. HRMS Theory 215.1305, Found 215.1306. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.83–1.93 (m, 1H), 1.95–2.03 (m, 1H), 2.22–2.39 (m, 4H including 2.33, s, 3H), 2.45–2.55 (m, 2H), 2.76 (dt, *J* = 15, 5.0Hz, 1H), 2.91 (dt, *J* = 17.1, 5.0 Hz, 1H), 3.17 (ddd, *J* = 4.0, 8.1, 11.8 Hz, 1H), 3.48 (dt, *J* = 11.8, 7.6 Hz, 1H), 3.79 (ddd, *J* = 4.5, 7.4, 11.9 Hz, 1H), 5.02 (t, *J* = 7.2 Hz, 1H), 7.03–7.10 (m, 3H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 21.4 (CH_3), 22.9 (CH_2), 28.7 (CH_2), 30.7 (CH_2), 36.2 (CH_2), 48.1 (CH_2), 57.0 (CH), 125.3 (CH), 128.7 (CH), 128.9 (CH), 136.1 (C), 137.8 (C), 138.1 (C), 170.8 (C); ν_{max}/cm^{-1} 1572, 1454, 1400, 831, 816, 751.





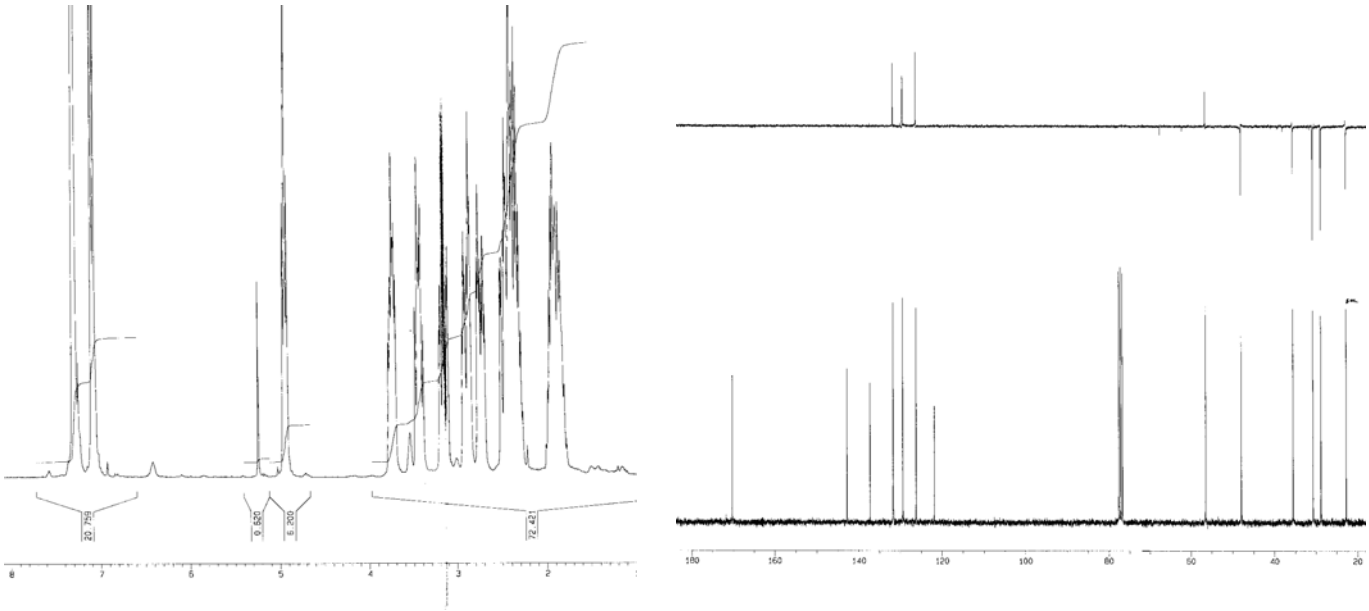
10-Fluoro-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19h)

Isolated as an oil (77% yield), HRMS Theory 219.1054, Found 219.1057. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.81–2.02 (m, 2H), 2.25–2.50 (m, 3H), 2.70–2.95 (m, 2H), 3.15 (ddd *J* = 4.1, 8.0, 12.0Hz, 1H), 3.40–3.51 (m, 1H), 3.70–3.80 (m, 1H), 4.99 (t, *J* = 7.1 Hz, 1H), 6.90 (dd, *J* = 8.3, 2.7 Hz, 1H), 6.95 (dd, *J* = 10.1, 2.1 Hz, 1H), 7.12 (dd, *J* = 8.3, 6.0 Hz, 1H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.8 (CH_2), 28.4 (CH_2), 30.8 (CH_2), 35.9 (CH_2), 48.1 (CH_2), 56.6 (CH), 112.0 (d, CH, J_{C-F} = 22.8Hz), 114.6 (d, CH, J_{C-F} = 20.8Hz), 130.4 (d, CH, J_{C-F} = 8.1 Hz), 136.4 (d, C, J = 3.1 Hz), 140.4 (d, C, J_{C-F} = 6.5 Hz), 161.5 (d, C, J_{C-F} = 245 Hz), 170.49 (C)



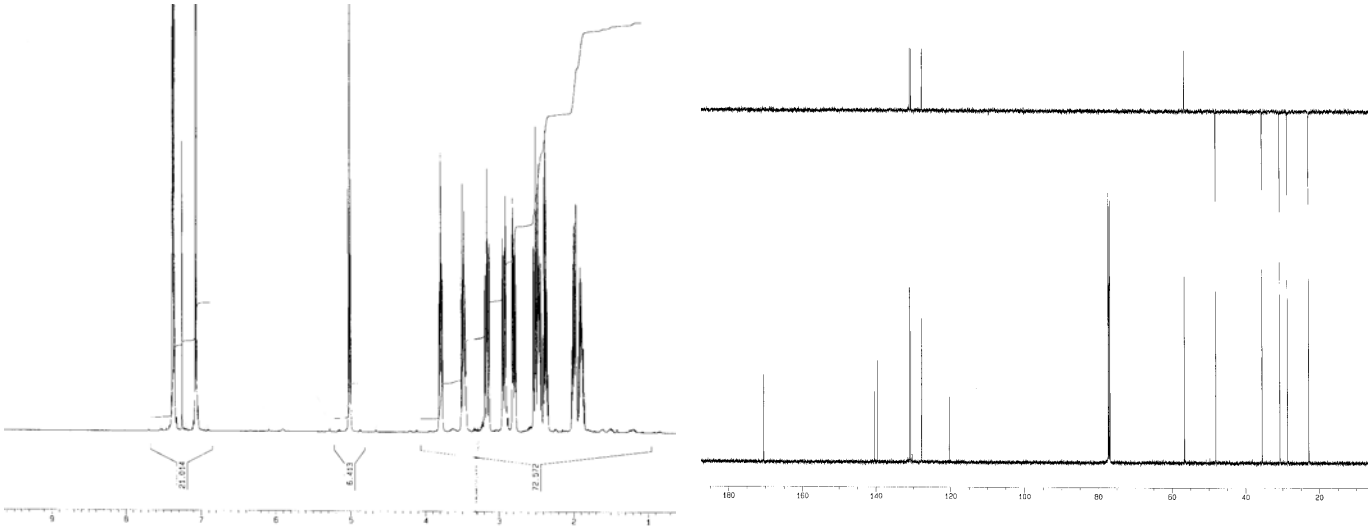
9-Bromo-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19i)

Isolated as an oil (75% yield) which solidified on standing, mpt 107-10°C. HRMS Theory: 279.0253, Found 279.0254. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.68-2.03 (m, 2H), 2.23-2.56 (m, 4H), 2.65-2.79 (m, 1H), 2.80-2.96 (m, 1H), 3.06-3.22 (m, 1H), 3.36-3.49 (m, 1H), 3.66-3.79 (m, 1H), 4.94 (t, *J* = 7Hz, 1H), 7.09 (d, *J* = 9 Hz, 1H), 7.25-7.35 (m, 2H); ^{13}C NMR and DEPT (75 MHz, $CDCl_3$) δ = 22.7 (CH_2), 28.8 (CH_2), 29.8 (CH_2), 30.7 (CH_2), 35.6 (CH_2), 48.0 (CH_2), 56.5 (CH), 121.9 (C), 126.3 (CH), 129.5 (CH), 131.8 (CH), 137.3 (C), 170.3 (C); ν_{max}/cm^{-1} 2947, 2871, 1611, 1601, 1437, 1400, 1310, 1139, 852, 828, 811, 761, 702, 655.



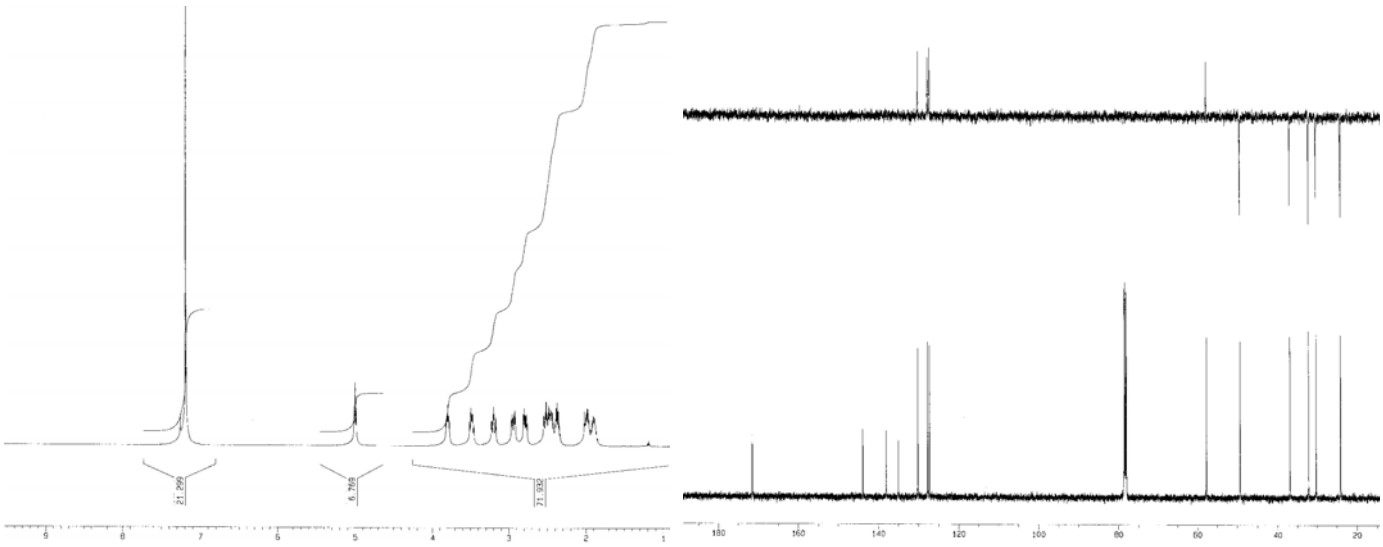
10-Bromo-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19j)

Isolated as a white crystalline solid (78% yield), m. pt 86-9°C (Et_2O/petrol), HRMS Theory: 279.0253, Found 279.0259. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.88–2.08 (m, 2H), 2.35–2.60 (m, 3H), 2.78–2.87 (m, 1H), 2.90–3.00 (m, 1H), 3.15 (dt, *J* = 12.3, 4.0 Hz, 1H), 3.46–3.55 (m, 1H), 3.76–3.85 (m, 1H), 5.08 (t, *J* = 7.1 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 7.32–7.41 (m, 2H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.8 (CH_2), 28.7 (CH_2), 30.8 (CH_2), 35.7 (CH_2), 48.1 (CH_2), 56.6 (CH), 120.3 (C), 127.9 (CH), 130.8 (CH), 131.1 (CH), 139.7 (C), 140.5 (C), 170.4 (C); ν_{max}/cm^{-1} 2936, 2862, 1604, 1448, 1437, 1403, 831, 814, 738.



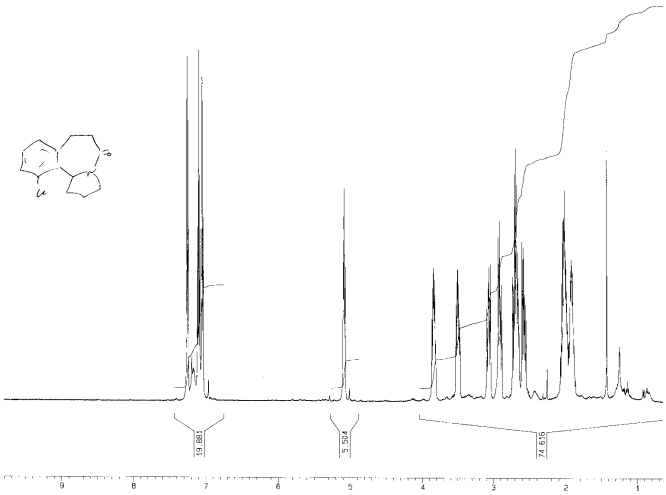
9-Chloro-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19k)

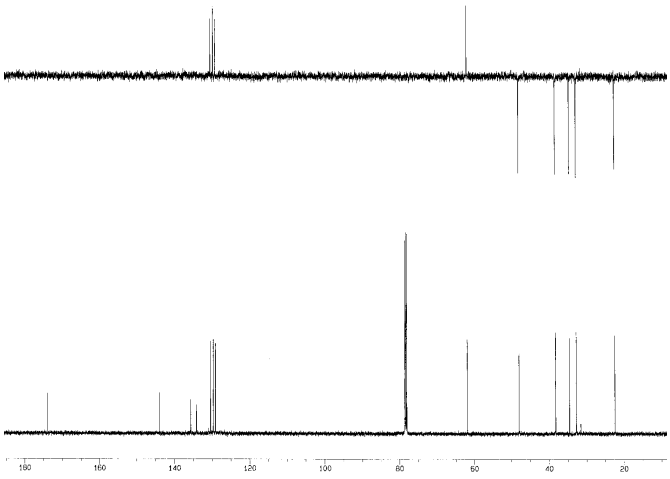
19k, the more polar isomer from the column (SiO_2, Et_2O + 3% MeOH), isolated as a solid (78% yield), mpt 83-6°C (Et_2O/petrol) HRMS Theory: 235.0758, Found 235.0765. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.81–2.02 (m, 2H), 2.30–2.55 (m, 4H), 2.77 (dt, *J* = 15, 5.0Hz, 1H), 2.94 (dt, *J* = 17.1, 5.0 Hz, 1H), 3.19 (m, 1H), 3.40–3.51 (m, 1H), 3.76–3.82 (m, 1H), 5.00 (t, *J* = 7.1 Hz, 1H), 7.19 (s, 3H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 23.0 (CH_2), 29.1 (CH_2), 31.0 (CH_2), 35.7 (CH_2), 48.2 (CH_2), 56.6 (CH), 126.2 (CH), 126.7 (CH), 129.1 (CH), 133.9 (C), 136.9 (C), 142.7 (C), 170.5 (C); ν_{max}/cm^{-1} 2949, 2874, 1598, 1571, 1438, 1398, 1303, 860, 833, 664.



11-Chloro-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19k')

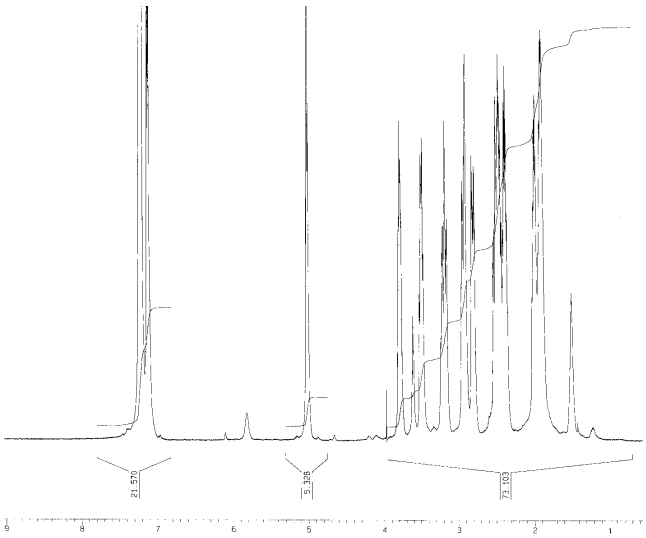
19k', the less polar siomer from the column (SiO_2, Et_2O + 2% MeOH), isolated as an oil (9% yield), HRMS Theory: 235.0758, Found 235.0757. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.85–2.07 (m, 3H), 2.50–2.75 (m, 3H), 2.86–2.98 (m, 1H,), 3.04–3.12 (m, 1H), 3.45–3.55 (m, 1H), 3.78–3.90 (m, 1H), 5.08 (t, *J* = 7.1 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 21.3 (CH_2), 31.7 (CH_2), 35.5 (CH_2), 37.2 (CH_2), 46.9 (CH_2), 60.8 (CH), 128.1 (CH), 128.7 (CH), 129.4 (CH), 133.1 (C), 134.7 (C), 142.8 (C), 172.7 (C); ν_{max}/cm^{-1} 2954, 2879, 1619, 1447, 1427, 1401, 785, 730..

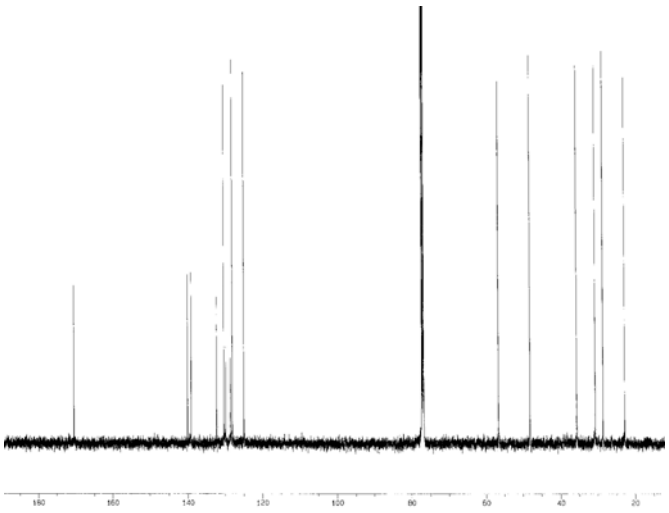




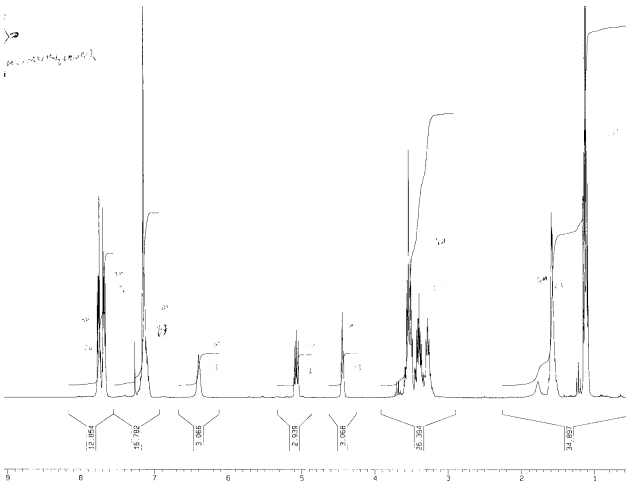
10-Chloro-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (19l)

Isolated as a white solid (90% yield) mpt 53-6°C. HRMS Theory: 235.0758, Found 235.0758. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.80–2.07 (m, 2H), 2.28–2.60 (m, 3H), 2.72–2.98 (m, 2H,), 3.10–3.25 (m, 1H), 3.40–3.55 (m, 1H), 3.72–3.86 (m, 1H), 5.02 (t, *J* = 6.9 Hz, 1H), 7.13 (d, *J* = 7.9 Hz, 1H), 7.15–7.32 (m, 2H), ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 22.8 (CH_2), 38.6 (CH_2), 30.7 (CH_2), 35.7 (CH_2), 48.1 (CH_2), 56.7 (CH), 125.0 (CH), 128.1 (CH), 130.4 (CH), 132.3 (C), 139.1 (C), 140.1 (C), 170.5 (C); ν_{max}/cm^{-1} 3304, 2945, 1641, 1585, 1541, 1490, 1449, 1405, 839.



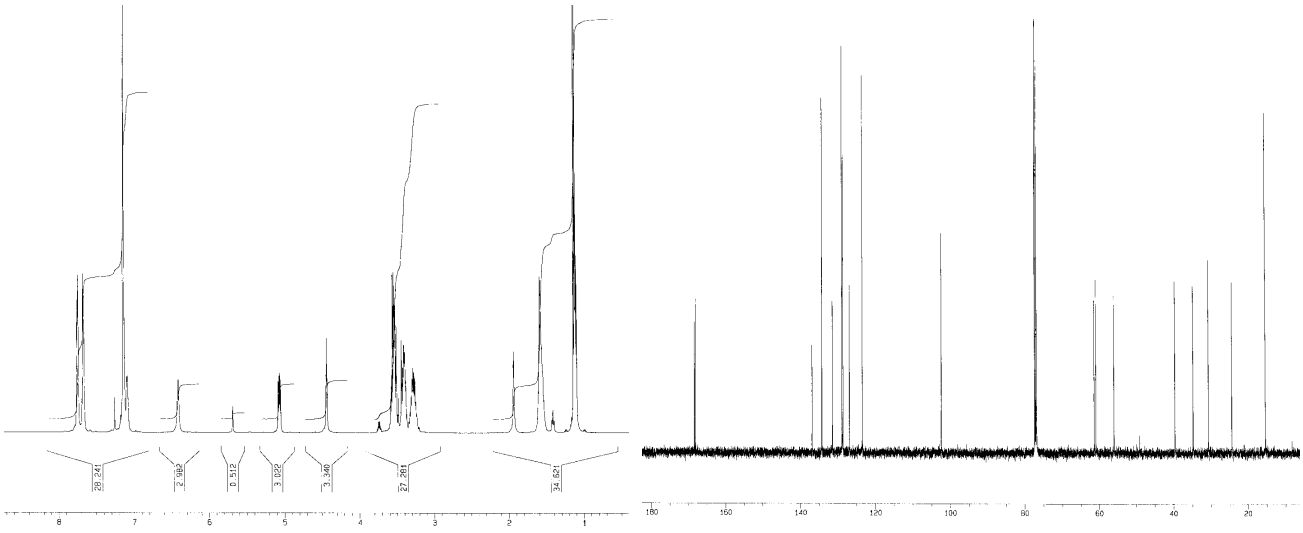


NMR of **(S)-N-(4,4-Diethoxybutyl)-2-[4-(1,3-dioxo-1,3-dihydroisoindol-2-yl)-3-phenylpropionamide (20)**

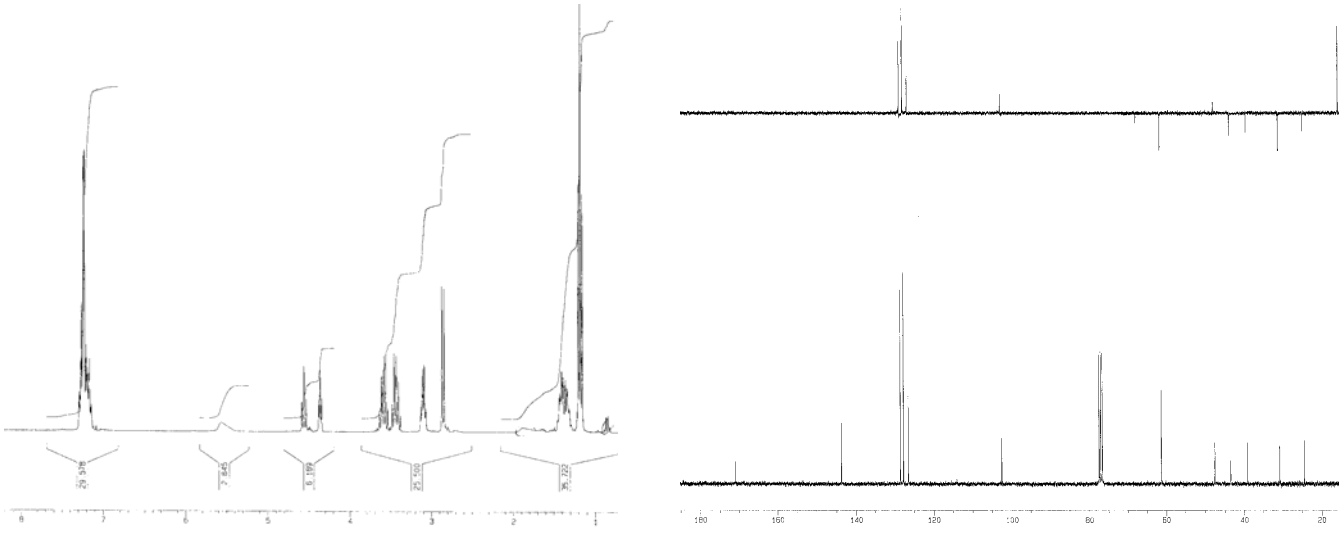


(R,S)-N-(4,4-Diethoxybutyl)-2-[4-(1,3-dioxo-1,3-dihydroisoindol-2-yl)-3-phenylpropionamide (20')

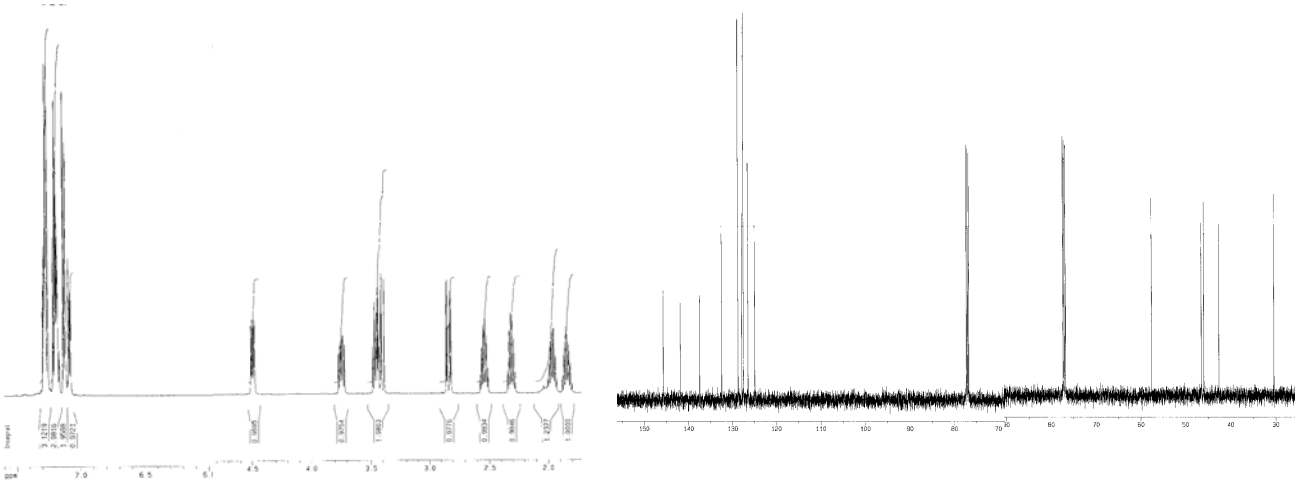
Isolated as a solid (Et_2O trituration, 100% yield) m.pt. 110-2°C. ^{1}H NMR (500 MHz, $CDCl_3$): δ= 1.11 (t, *J* = 7 Hz, 3H), 1.13 (t, *J* = 7Hz, 3H), 1.51-1.67 (m, 4H), 3.18-3.62 (m, 8H), 4.44 (t, *J* = 5 Hz, 1H), 5.07 (dd, *J* = 6, 10Hz, 1H), 6.40 (brs, 1H), 7.06-7.20 (m, 5H), 7.63-7.71 (m, 2H), 7.71-7.79 (m, 2H); ^{13}C NMR and DEPT (125.8 MHz, $CDCl_3$) δ = 15.3 (CH_3), 24.4 (CH_2), 30.8 (CH_2), 34.8 (CH_2), 39.6 (CH_2), 56.0 (CH), 60.9 (CH_2), 61.4 (CH_2), 102.4 (CH), 123.5 (CH), 127.0 (CH), 128.7 (CH), 128.9 (CH), 131.5 (C), 134.3 (C), 136.9 (C), 168.0 (C), 168.4 (C). ν_{max}/cm^{-1} 3303, 2973, 2929, 1774, 1711, 1642, 1545, 1380, 1104, 1065, 873, 720, 706.



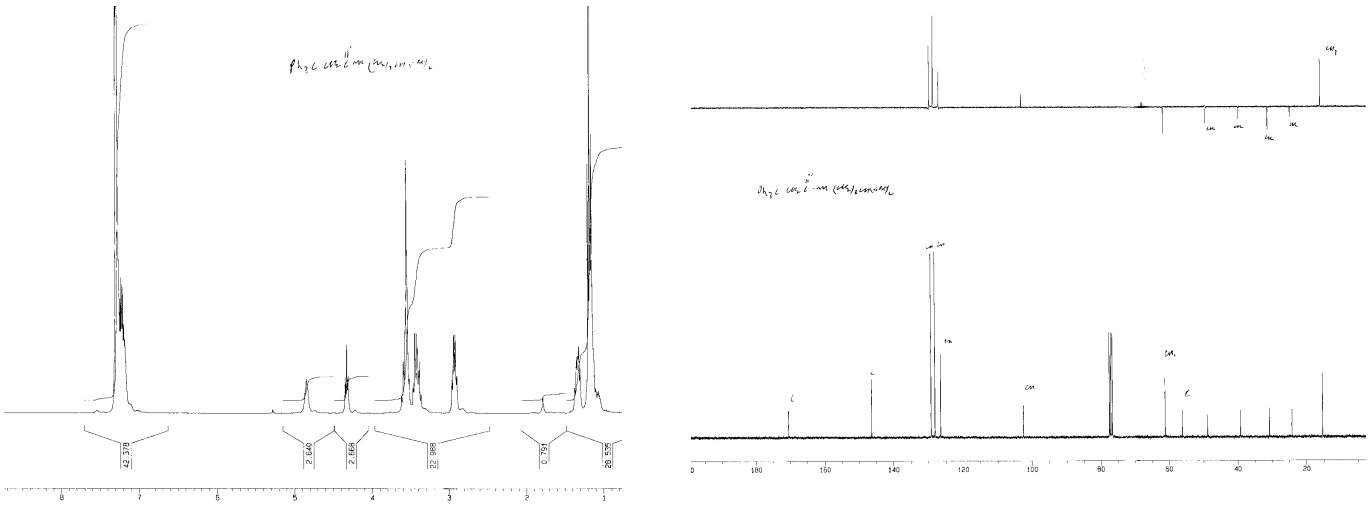
NMR of **N-(4,4-Diethoxybutyl)-3,3-diphenylpropionamide (21)**



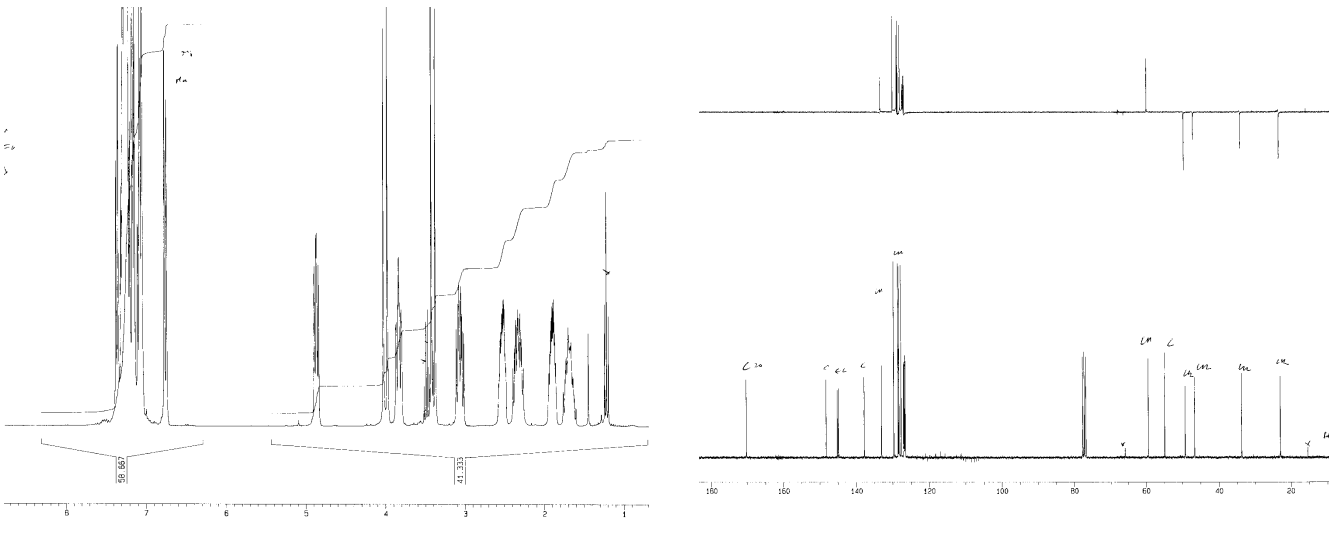
NMR of **7-Phenyl-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (22)**



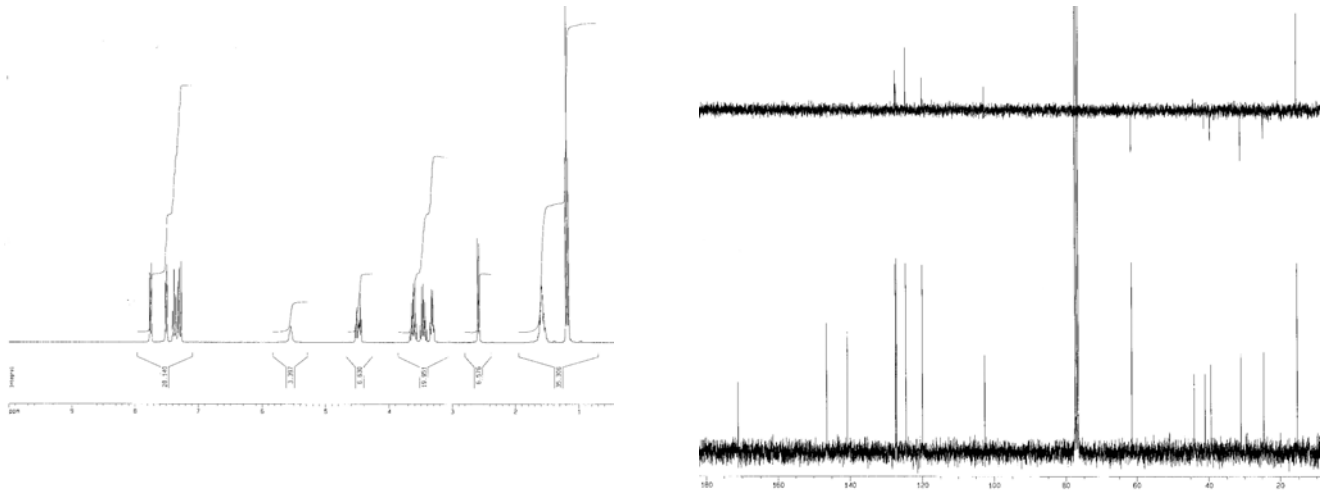
NMR of N-(4,4-Diethoxybutyl)-3,3,3-triphenylpropionamide (23)



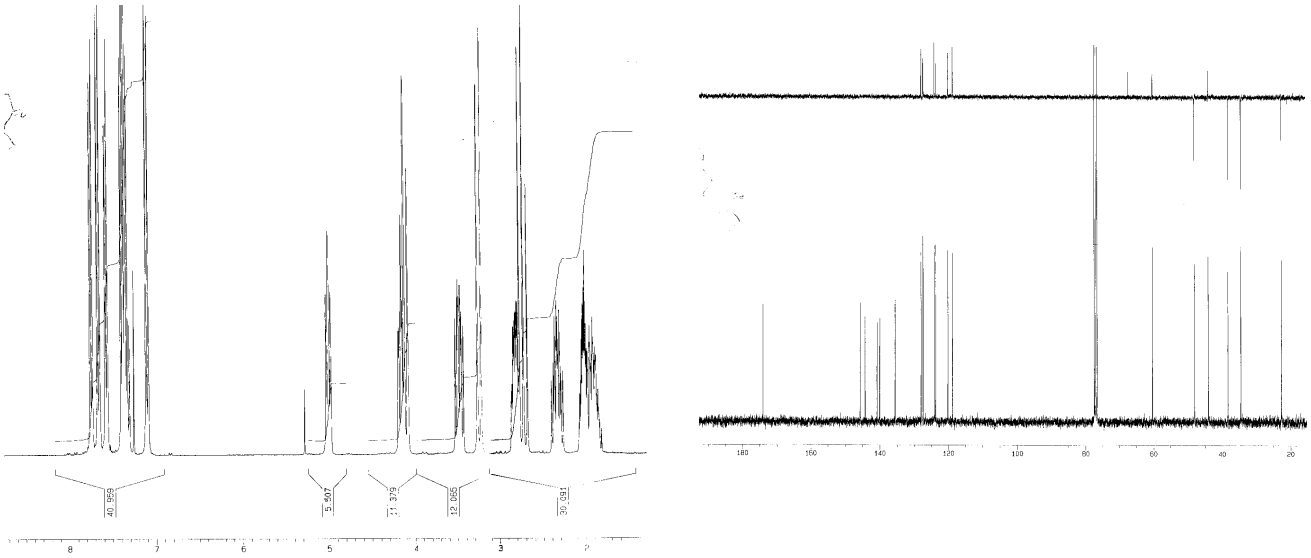
NMR of 7,7-Diphenyl-1,2,3,6,7,11b-hexahydro-benzo[c]pyrrolo[1,2-a]azepin-5-one (24)



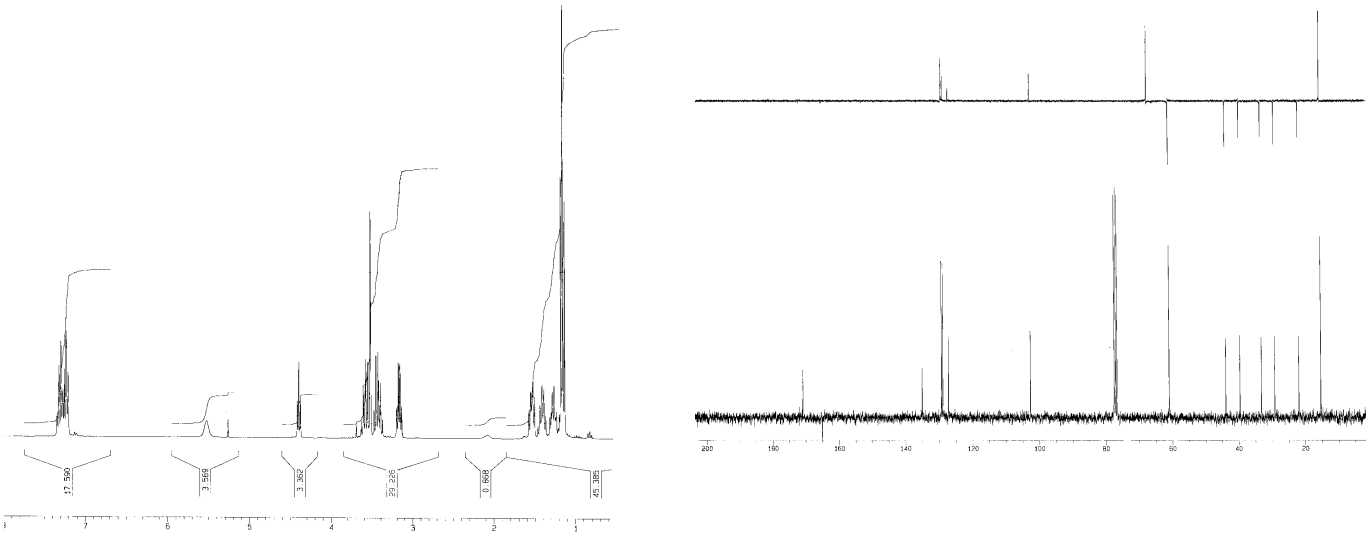
NMR of **N-(4,4-Diethoxybutyl)-2-(9H-fluoren-9-yl)-acetamide (25)**



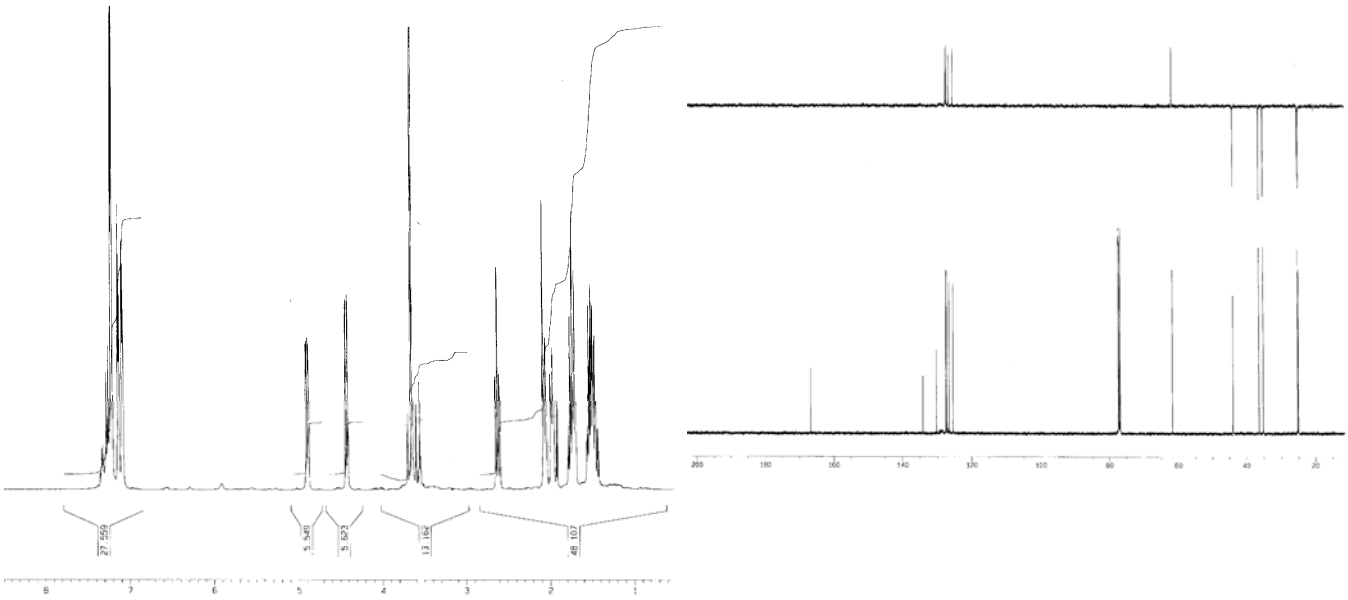
NMR of **1,3,4,5,6,14b-Hexahydro-fluoreno[1,9-cd]-pyrrolo[3,4-a]azepine-2-one (26).**



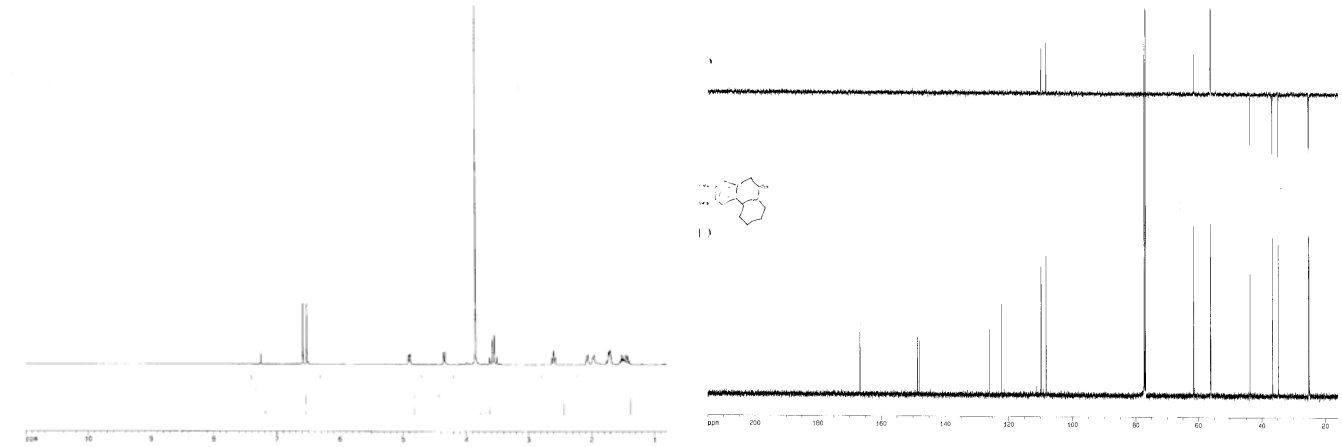
NMR of **N-(5,5-Diethoxypentyl)-2-phenylacetamide (29)**



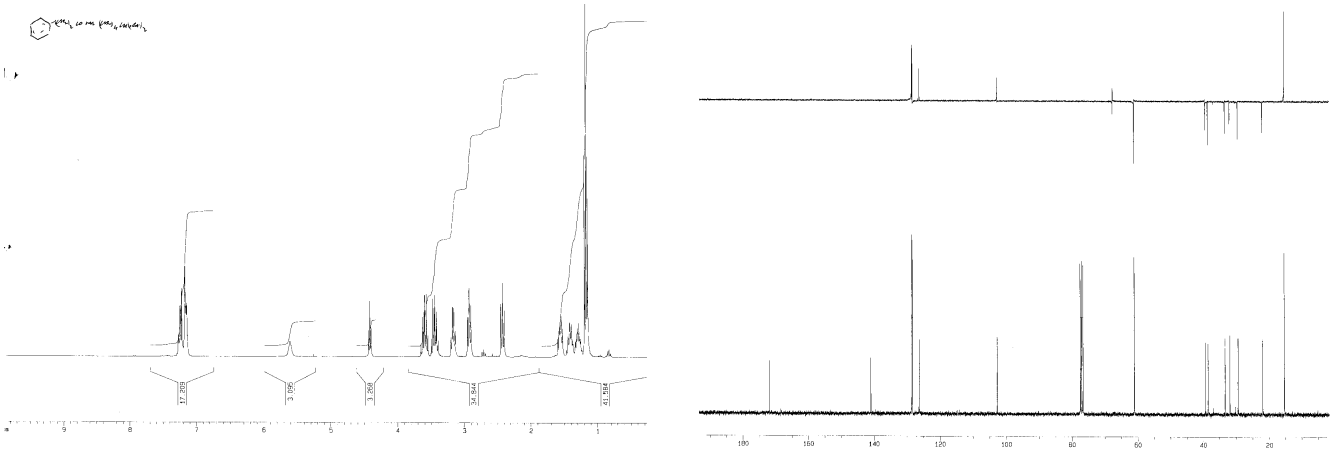
NMR of **1,2,3,4,7,11b-hexahydro-benzo[a]quinolizin-6-one (31)**



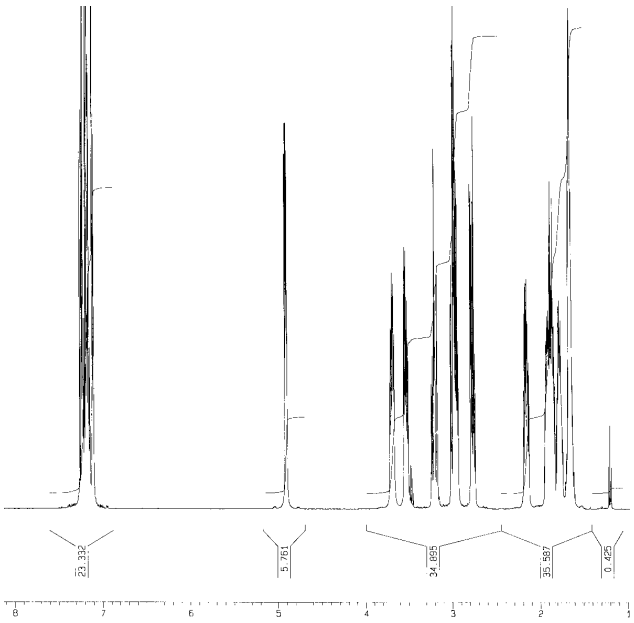
NMR of **9,10-Dimethoxy-1,2,3,4,7,11b-hexahydro-benzo[a]-quinolizin-6-one (32)**



NMR of **N-(5,5-diethoxypentyl)-3-phenylpropionamide (33)**



NMR of **1,3,4,7,8,12b-Hexahydro-2H-pyrido[2,1-a][2]benzazepin-6-one (34)**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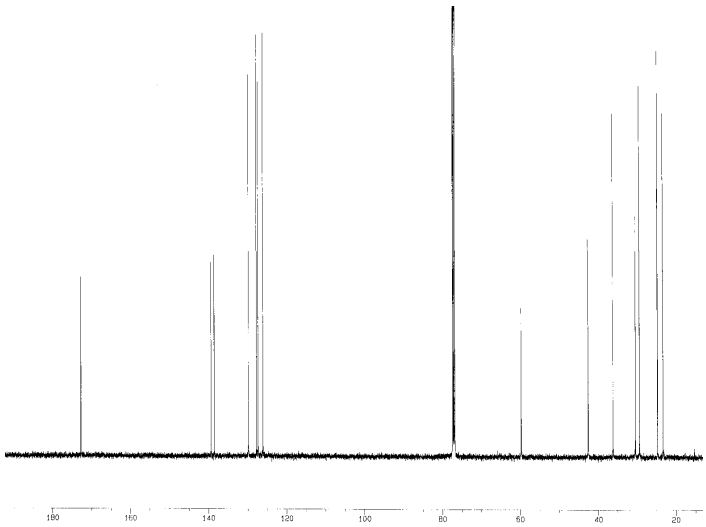
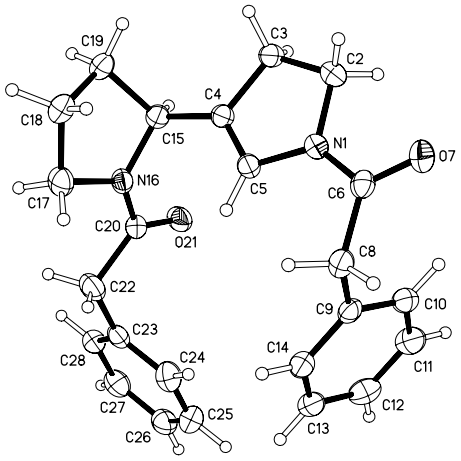
5.761
34.855
35.587
0.425
8
7
6
5
4
3
2
1


180
160
140
120
100
80
60
40
20

Supplementary Material for the Crystal Structure of [9]



A molecule from the crystal structure of **[9]** showing the numbering scheme employed. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 50% probability level. Hydrogen atoms are displayed with an arbitrarily small radius.

Table 1. Sample and crystal data for [9].

Crystallization solvents	Methanol and water	
Crystallization method	Slow evaporation	
Crystal habit	Colourless block	
Crystal size	0.24 x 0.12 x 0.06 mm	
Moiety formula	$C_{24}H_{26}N_2O_2$	
Empirical formula	$C_{24}H_{26}N_2O_2$	
Formula weight	374.47	
Temperature	150(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 5.12520(10) Å	α= 90°
	b = 19.6810(2) Å	β= 96.1731(6)°
	c = 9.80400(10) Å	γ = 90°
Volume	983.19(2) $Å^3$	
Z	2	
Density (calculated)	1.265 Mg/m^3	
Absorption coefficient	0.636 mm^{-1}	
F(000)	400	

Table 2. Data collection and structure refinement for [9].

Diffractometer	Bruker AXS SMART 6000
Radiation source	Normal focus sealed tube, CuK□
Data collection method	ω scans
Theta range for data collection	4.49 to 73.28°
Index ranges	$-5 \leq h \leq 5, -24 \leq k \leq 23, -11 \leq l \leq 12$
Reflections collected	9563
Independent reflections	3747 [R(int) = 0.0122]
Coverage of independent reflections	95.7 %
Absorption correction	Integration
Max. and min. transmission	0.9629 and 0.8922
Structure solution technique	Direct methods
Structure solution program	SHELXTL V6.10 UNIX (Bruker, 2001)
Refinement technique	Full-matrix least-squares on F^2
Refinement program	SHELXTL V6.10 UNIX (Bruker, 2001)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$

Data / restraints / parameters	3747 / 1 / 254
Goodness-of-fit on F^2	1.060
Δ/σ_{max}	0.000
Final R indices	
3692 data; I>2□(I)	R1 = 0.0304, wR2 = 0.0813
all data	R1 = 0.0309, wR2 = 0.0818
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.1087P]$ where $P = [MAX(F_o^2, 0) + 2F_c^2]/3$
Absolute structure parameter	-0.05(17)
Largest diff. peak and hole	0.172 and -0.161 eÅ^{-3}

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for [9].

U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
N1	0.4835(2)	0.42895(6)	0.37514(10)	0.0245(2)
C2	0.3230(3)	0.48278(7)	0.30337(13)	0.0292(3)
C3	0.1580(3)	0.51117(7)	0.41237(13)	0.0253(3)
C4	0.2588(2)	0.47314(6)	0.54131(13)	0.0222(2)
C5	0.4359(3)	0.42735(6)	0.51465(12)	0.0228(3)
C6	0.6541(3)	0.39167(7)	0.30965(13)	0.0245(3)
O7	0.6814(2)	0.40284(5)	0.18891(10)	0.0341(2)
C8	0.8031(3)	0.33563(7)	0.39100(13)	0.0256(3)
C9	0.6325(3)	0.27430(6)	0.41320(13)	0.0240(3)
C10	0.4393(3)	0.25253(7)	0.31179(14)	0.0283(3)
C11	0.2862(3)	0.19627(8)	0.33265(17)	0.0345(3)
C12	0.3256(3)	0.16017(7)	0.45500(17)	0.0349(3)
C13	0.5186(3)	0.18118(7)	0.55555(15)	0.0329(3)
C14	0.6706(3)	0.23815(7)	0.53549(15)	0.0295(3)
C15	0.1510(2)	0.48733(6)	0.67513(13)	0.0227(2)
N16	0.2484(2)	0.43972(6)	0.78384(10)	0.0236(2)
C17	0.4603(3)	0.46922(7)	0.87958(14)	0.0307(3)
C18	0.5033(3)	0.53929(7)	0.81886(14)	0.0281(3)
C19	0.2396(3)	0.55641(7)	0.73837(14)	0.0268(3)
C20	0.1184(3)	0.38185(7)	0.80454(13)	0.0250(3)
O21	-0.0754(2)	0.36409(5)	0.72847(10)	0.0327(2)
C22	0.2249(4)	0.34034(8)	0.92952(15)	0.0385(4)
C23	0.1019(3)	0.27109(7)	0.93135(13)	0.0294(3)
C24	0.1856(3)	0.21973(8)	0.84999(15)	0.0365(3)
C25	0.0737(4)	0.15538(8)	0.85046(16)	0.0403(4)
C26	-0.1222(3)	0.14207(8)	0.93313(16)	0.0382(3)
C27	-0.2079(3)	0.19327(9)	1.01397(15)	0.0371(3)
C28	-0.0962(3)	0.25734(8)	1.01348(14)	0.0322(3)

Table 4. Selected bond lengths (Å) for [9].

N1-C6	1.3549(17)	N1-C5	1.4153(16)
N1-C2	1.4728(17)	C2-C3	1.5376(19)
C3-C4	1.5115(17)	C4-C5	1.3248(18)
C4-C15	1.5035(17)	C6-O7	1.2266(17)
C6-C8	1.5180(19)	C8-C9	1.5198(19)
C9-C14	1.390(2)	C9-C10	1.3931(19)
C10-C11	1.385(2)	C11-C12	1.390(2)
C12-C13	1.383(2)	C13-C14	1.391(2)
C15-N16	1.4660(16)	C15-C19	1.5422(18)
N16-C20	1.3460(18)	N16-C17	1.4759(16)
C17-C18	1.527(2)	C18-C19	1.5278(19)
C20-O21	1.2268(16)	C20-C22	1.5242(18)
C22-C23	1.502(2)	C23-C24	1.384(2)
C23-C28	1.389(2)	C24-C25	1.390(2)
C25-C26	1.381(3)	C26-C27	1.383(2)
C27-C28	1.385(2)		

Table 5. Selected bond angles (°) for [9].

C6-N1-C5	129.28(11)	C6-N1-C2	121.24(11)
C5-N1-C2	109.41(11)	N1-C2-C3	104.48(10)
C4-C3-C2	103.72(11)	C5-C4-C15	128.49(11)

C5-C4-C3	110.59(11)	C15-C4-C3	120.87(11)
C4-C5-N1	111.62(11)	O7-C6-N1	120.45(13)
O7-C6-C8	122.28(12)	N1-C6-C8	117.27(11)
C6-C8-C9	112.81(11)	C14-C9-C10	118.77(13)
C14-C9-C8	120.04(12)	C10-C9-C8	121.18(12)
11-C10-C9	120.73(13)	C10-C11-C12	120.26(14)
C13-C12-C11	119.27(14)	C12-C13-C14	120.58(13)
C9-C14-C13	120.38(13)	N16-C15-C4	112.78(10)
N16-C15-C19	102.05(10)	C4-C15-C19	113.25(11)
C20-N16-C15	121.00(11)	C20-N16-C17	125.41(11)
C15-N16-C17	112.38(10)	N16-C17-C18	103.57(11)
C17-C18-C19	104.10(11)	C18-C19-C15	102.66(11)
O21-C20-N16	122.07(12)	O21-C20-C22	122.19(12)
N16-C20-C22	115.74(12)	C23-C22-C20	112.41(12)
C24-C23-C28	118.87(14)	C24-C23-C22	119.96(15)
C28-C23-C22	121.17(14)	C23-C24-C25	120.67(15)
C26-C25-C24	120.04(15)	C25-C26-C27	119.59(15)
C26-C27-C28	120.31(15)	C27-C28-C23	120.51(13)

Table 6. Selected torsion angles (°) for [9].

C6-N1-C2-C3	179.37(11)	C5-N1-C2-C3	-3.48(14)
N1-C2-C3-C4	4.19(13)	C2-C3-C4-C5	-3.75(14)
C2-C3-C4-C15	178.58(11)	C15-C4-C5-N1	179.15(12)
C3-C4-C5-N1	1.71(15)	C6-N1-C5-C4	178.08(12)
C2-N1-C5-C4	1.23(15)	C5-N1-C6-O7	-175.38(13)
C2-N1-C6-O7	1.15(19)	C5-N1-C6-C8	5.69(19)
C2-N1-C6-C8	-177.78(12)	O7-C6-C8-C9	-104.91(15)
N1-C6-C8-C9	73.99(14)	C6-C8-C9-C14	-144.20(12)
C6-C8-C9-C10	36.86(17)	C14-C9-C10-C11	0.6(2)
C8-C9-C10-C11	179.52(13)	C9-C10-C11-C12	-0.8(2)
C10-C11-C12-C13	0.2(2)	C11-C12-C13-C14	0.6(2)
C10-C9-C14-C13	0.20(19)	C8-C9-C14-C13	-178.77(12)
C12-C13-C14-C9	-0.8(2)	C5-C4-C15-N16	-4.49(19)
C3-C4-C15-N16	172.72(11)	C5-C4-C15-C19	110.77(15)
C3-C4-C15-C19	-72.02(14)	C4-C15-N16-C20	-90.68(13)
C19-C15-N16-C20	147.50(11)	C4-C15-N16-C17	101.25(12)
C19-C15-N16-C17	-20.57(13)	C20-N16-C17-C18	-170.50(12)
C15-N16-C17-C18	-3.06(14)	N16-C17-C18-C19	25.85(13)
C17-C18-C19-C15	-38.44(13)	N16-C15-C19-C18	35.63(12)
C4-C15-C19-C18	-85.87(12)	C15-N16-C20-O21	5.10(19)
C17-N16-C20-O21	171.53(13)	C15-N16-C20-C22	-174.10(12)
C17-N16-C20-C22	-7.66(19)	O21-C20-C22-C23	11.3(2)
N16-C20-C22-C23	-169.55(13)	C20-C22-C23-C24	78.78(19)
C20-C22-C23-C28	-101.09(16)	C28-C23-C24-C25	-0.1(2)
C22-C23-C24-C25	-179.97(14)	C23-C24-C25-C26	-0.3(2)
C24-C25-C26-C27	0.7(2)	C25-C26-C27-C28	-0.8(2)
C26-C27-C28-C23	0.4(2)	C24-C23-C28-C27	0.1(2)
C22-C23-C28-C27	179.93(13)		

Table 7. Anisotropic atomic displacement parameters ($Å^2$) for [9].

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2}U^{11} + ... + 2hka^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N1	0.0288(6)	0.0244(5)	0.0200(5)	0.0008(4)	0.0019(4)	0.0015(4)
C2	0.0362(7)	0.0279(7)	0.0232(6)	0.0041(5)	0.0019(5)	0.0041(5)
C3	0.0281(7)	0.0215(6)	0.0254(6)	0.0013(4)	-0.0005(5)	0.0005(5)
C4	0.0238(6)	0.0205(6)	0.0218(6)	0.0002(4)	-0.0009(4)	-0.0019(4)
C5	0.0261(7)	0.0226(6)	0.0194(5)	0.0005(5)	0.0009(4)	-0.0013(5)
C6	0.0256(7)	0.0243(6)	0.0233(6)	-0.0042(5)	0.0015(4)	-0.0043(5)
O7	0.0449(6)	0.0361(5)	0.0224(5)	-0.0024(4)	0.0088(4)	0.0018(4)
C8	0.0228(7)	0.0285(6)	0.0253(6)	-0.0049(5)	0.0016(4)	0.0014(5)
C9	0.0242(7)	0.0228(6)	0.0253(6)	-0.0051(5)	0.0035(5)	0.0050(5)
C10	0.0302(7)	0.0250(6)	0.0289(6)	-0.0016(5)	-0.0007(5)	0.0036(5)
C11	0.0299(7)	0.0301(7)	0.0414(8)	-0.0044(6)	-0.0058(6)	0.0001(5)
C12	0.0332(8)	0.0243(7)	0.0484(8)	-0.0011(6)	0.0092(6)	-0.0006(5)
C13	0.0446(9)	0.0252(7)	0.0299(6)	0.0010(5)	0.0084(6)	0.0069(6)
C14	0.0331(8)	0.0278(7)	0.0267(6)	-0.0048(5)	-0.0004(5)	0.0052(5)
C15	0.0211(6)	0.0234(6)	0.0235(6)	-0.0010(5)	0.0016(4)	0.0010(4)
N16	0.0244(6)	0.0261(6)	0.0196(5)	-0.0019(4)	-0.0003(4)	0.0001(4)
C17	0.0275(7)	0.0340(7)	0.0291(7)	-0.0020(6)	-0.0044(5)	-0.0041(5)
C18	0.0251(7)	0.0280(7)	0.0313(6)	-0.0067(5)	0.0034(5)	-0.0013(5)
C19	0.0271(7)	0.0244(6)	0.0291(6)	-0.0061(5)	0.0039(5)	0.0014(5)
C20	0.0292(7)	0.0244(6)	0.0208(6)	-0.0022(5)	-0.0003(5)	0.0014(5)
O21	0.0371(6)	0.0287(5)	0.0296(5)	0.0024(4)	-0.0096(4)	-0.0058(4)
C22	0.0496(9)	0.0321(7)	0.0300(7)	0.0058(6)	-0.0141(6)	-0.0088(6)
C23	0.0360(8)	0.0282(7)	0.0215(6)	0.0035(5)	-0.0076(5)	-0.0003(5)
C24	0.0380(9)	0.0434(9)	0.0284(7)	-0.0010(6)	0.0052(6)	-0.0006(6)
C25	0.0537(10)	0.0342(8)	0.0321(7)	-0.0070(6)	0.0013(7)	0.0059(7)
C26	0.0496(10)	0.0294(7)	0.0332(7)	0.0037(6)	-0.0065(6)	-0.0065(6)
C27	0.0398(9)	0.0418(8)	0.0298(7)	0.0073(6)	0.0042(6)	-0.0022(7)
C28	0.0392(8)	0.0315(7)	0.0252(6)	-0.0005(6)	-0.0001(5)	0.0072(6)

Table 8. Hydrogen atom coordinates and isotropic atomic displacement parameters ($Å^2$) for [9].

	x	y	z	U
H2A	0.2089	0.4639	0.2245	0.035
H2B	0.4356	0.5186	0.2698	0.035
H3A	0.1851	0.5607	0.4242	0.030
H3B	-0.0311	0.5022	0.3871	0.030
H5A	0.5204	0.3972	0.5810	0.027
H8A	0.8756	0.3539	0.4813	0.031
H8B	0.9523	0.3210	0.3418	0.031
H10A	0.4123	0.2765	0.2274	0.034
H11A	0.1537	0.1823	0.2631	0.041
H12A	0.2211	0.1215	0.4694	0.042
H13A	0.5477	0.1565	0.6391	0.040
H14A	0.8011	0.2524	0.6058	0.035
H15A	-0.0451	0.4855	0.6614	0.027
H17A	0.6216	0.4413	0.8832	0.037
H17B	0.4061	0.4731	0.9732	0.037
H18A	0.6447	0.5379	0.7576	0.034
H18B	0.5498	0.5731	0.8922	0.034
H19A	0.2602	0.5906	0.6663	0.032
H19B	0.1131	0.5736	0.7997	0.032
H22A	0.4172	0.3353	0.9299	0.046
H22B	0.1917	0.3652	1.0138	0.046
H24A	0.3207	0.2285	0.7932	0.044
H25A	0.1320	0.1205	0.7939	0.048
H26A	-0.1976	0.0980	0.9344	0.046
H27A	-0.3440	0.1845	1.0701	0.044
H28A	-0.1556	0.2921	1.0698	0.039

Table 9. Selected hydrogen bond information for [9] (Å and °).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C5-H5A...O21#1	0.95	2.48	3.3301(15)	148.6
C8-H8A...O21#1	0.99	2.42	3.3474(16)	156.2
C14-H14A...O21#1	0.95	2.55	3.2999(17)	135.7

#1 x+1,y,z