Supporting information

Straightforward four-component access to spiroindolines

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General procedure for the synthesis of the tryptamine derived Ugi adducts.
Characterization data of compounds **1a**-**1l** and their corresponding ^{1}H and ^{13}C NMR spectra.

General Procedure for the spirocyclisation.
Characterization data of compounds **2c**-**2l** and their corresponding ^{1}H and ^{13}C NMR spectra.

General Procedure for the One Pot Reaction Ugi- spirocyclization.
Characterization data of compounds.

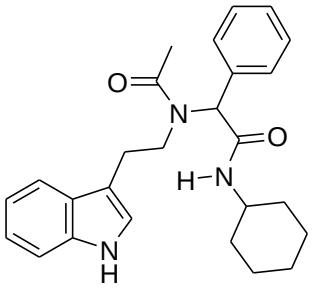
NMR spectra were recorded on a 400 MHz spectrometer, using deuterated solvent as reference and/or internal deuterium lock. Two-dimensional NMR spectroscopy [^{1}H -^{1}H COSY spectra, ^{1}H - ^{13}C COSY spectra (HSQC) and long-range ^{1}H -^{13}C COSY spectra (HMBC)], were carried out to determine the correlation between ^{1}H and ^{13}C. The chemical shifts for all NMR spectra are expressed in parts per million to high frequency of TMS reference. Coupling constants (*J*) are quoted in Hz and are recorded to the nearest 0.1 Hz.
The IR spectra were obtained using ATR accessories. High-resolution (HR) mass spectra were performed on a GC/MS system spectrometer. TLC was carried out using precoated plates of silica gel 60F254.

Electronic Supplementary Material (ESI) for Chemical Communications

General Procedure for the Ugi Multicomponent Reaction

To a solution of tryptamine (1 equiv) in anhydrous methanol (1 M) were added successively glacial acetic acid (1 equiv), isocyanide (1 equiv) and benzaldehyde (1 equiv). The reaction mixture was stirred at room temperature for 6-24 h under nitrogen atmosphere. The solvent was then removed under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel using a gradient of EtOAc in petroleum ether as eluant.

2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-cyclohexyl-2-phenylacetamide



$C_{26}H_{31}N_3O_2$
MW = 417.54 g/mol

1a

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), cyclohexyl isocyanide (250 µL, 2.0 mmol) and benzaldehyde (200 µL, 2.0 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 70:30) as eluant gave the desired product (89%) as a white solid.

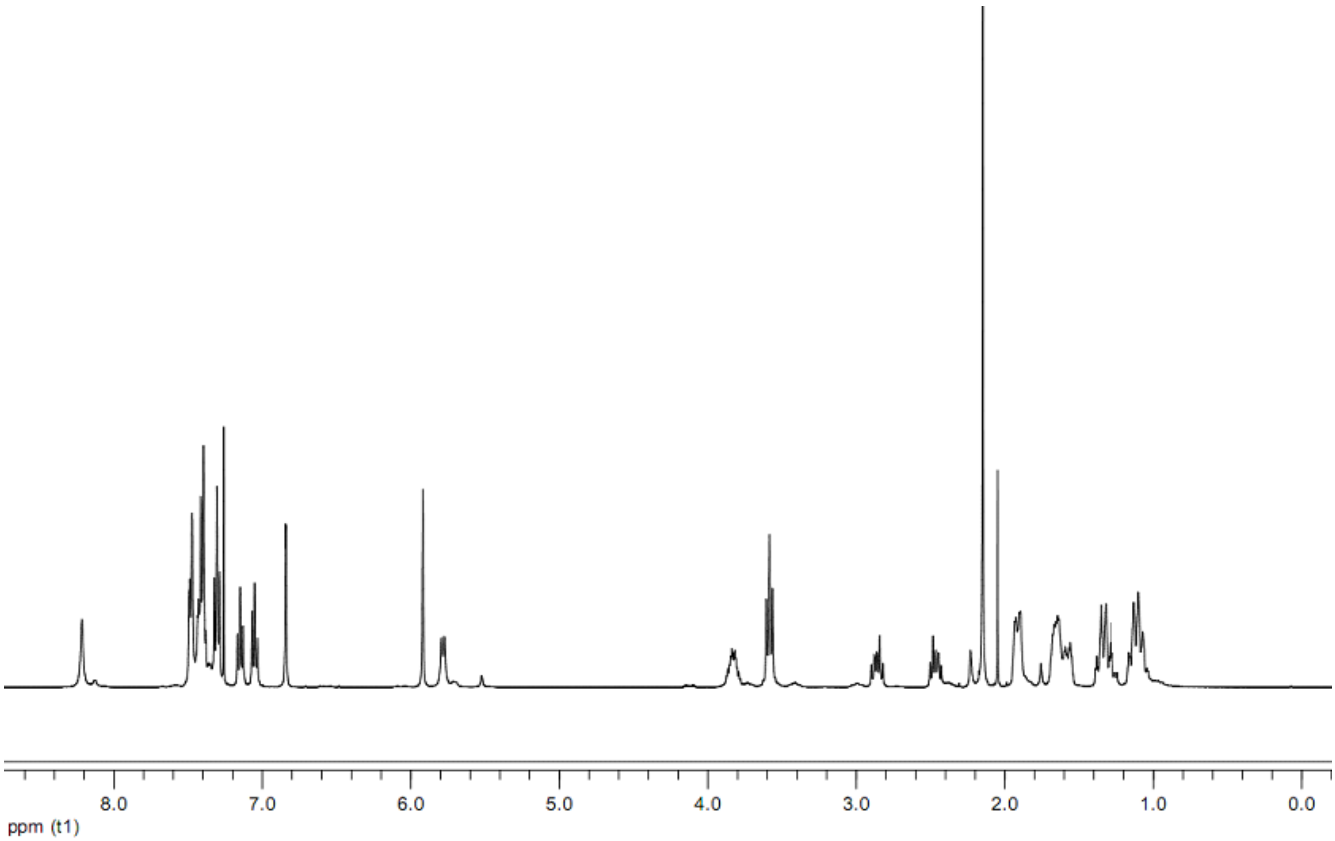
m.p. 203 °C.

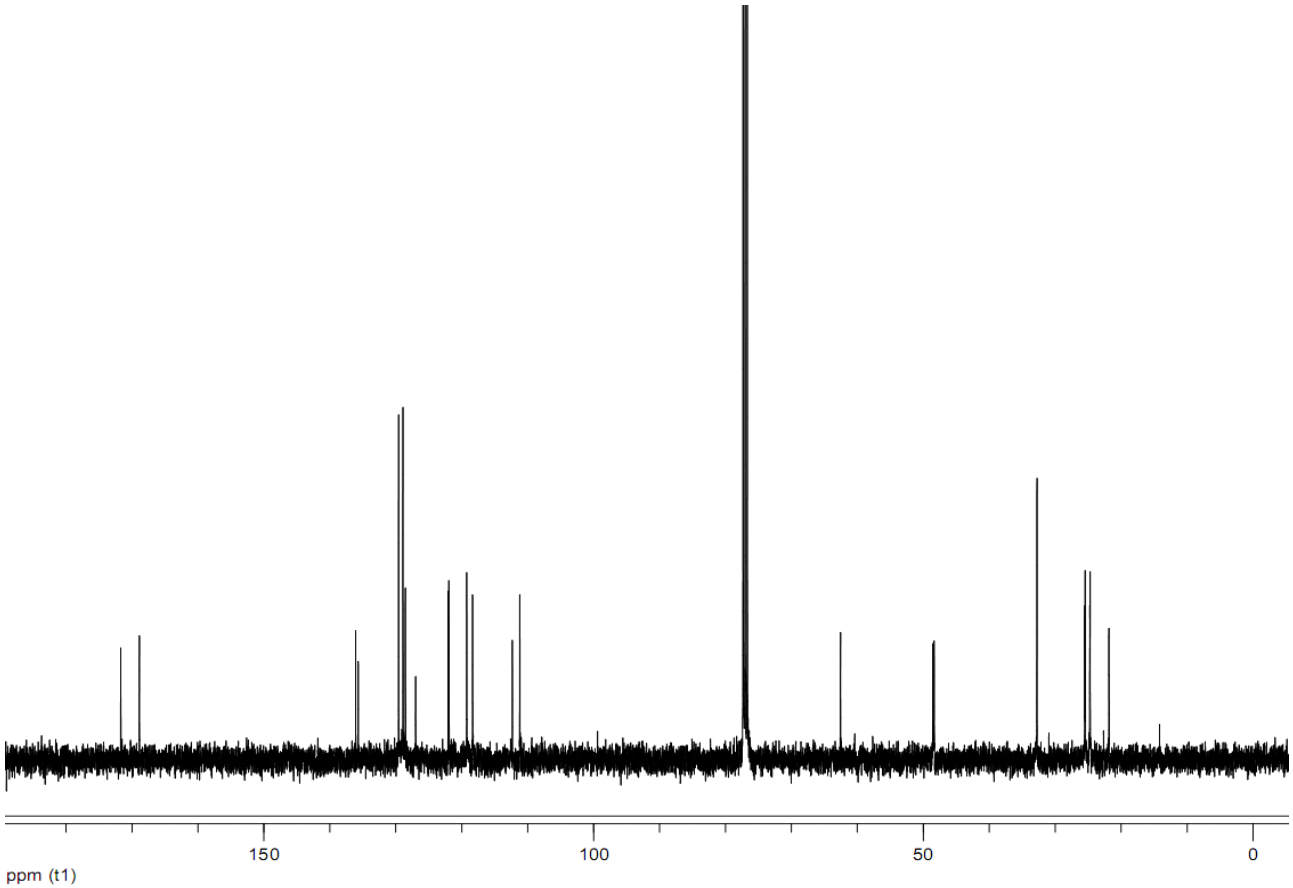
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (br s, 1H), 7.49-7.47 (m, 2H), 7.44-7.38 (m, 3H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 7.5 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 2.0 Hz, 1H), 5.92 (s, 1H), 5.78 (d, *J* = 7.8 Hz, 1H), 3.87-3.78 (m, 1H), 3.59 (t, *J* = 8.1 Hz, 2H), 2.90-2.82 (m, 1H), 2.50-2.43 (m, 1H), 2.15 (s, 3H), 1.93-1.90 (m, 2H), 1.68-1.64 (m, 2H), 1.60-1.56 (m, 1H), 1.39-1.28 (m, 2H), 1.16-1.04 (m, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.7, 168.9, 136.1, 135.7, 129.6 (2), 128.9 (2), 128.5, 127.0, 122.1, 122.0, 119.2, 118.3, 112.3, 111.2, 62.6, 48.3 (2), 32.8 (2), 25.5, 25.4, 24.7 (2), 21.8.

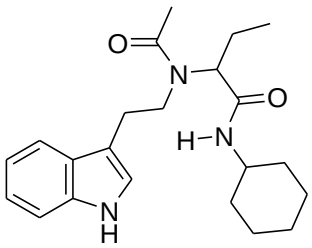
IR (thin film) 3285, 3059, 2930, 2854, 1656, 1625, 1546, 1452, 1352, 1230 cm^{-1}.

HRMS Calculated for $C_{26}H_{31}N_3O_2$ 417.2416, found 417.2417.





2-{Acetyl-[2-(1*H*-indol-3-yl)-ethyl]-amino}-N-cyclohexyl-butyramide



$C_{22}H_{31}N_3O_2$
MW = 369.50 g/mol

1b

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), cyclohexyl isocyanide (250 µL, 2.0 mmol) and propionaldehyde (150 µL, 2.0 mmol). Reaction time: 7h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (60:40 - 80:20) as eluant gave the desired product (89%) as a white solid.

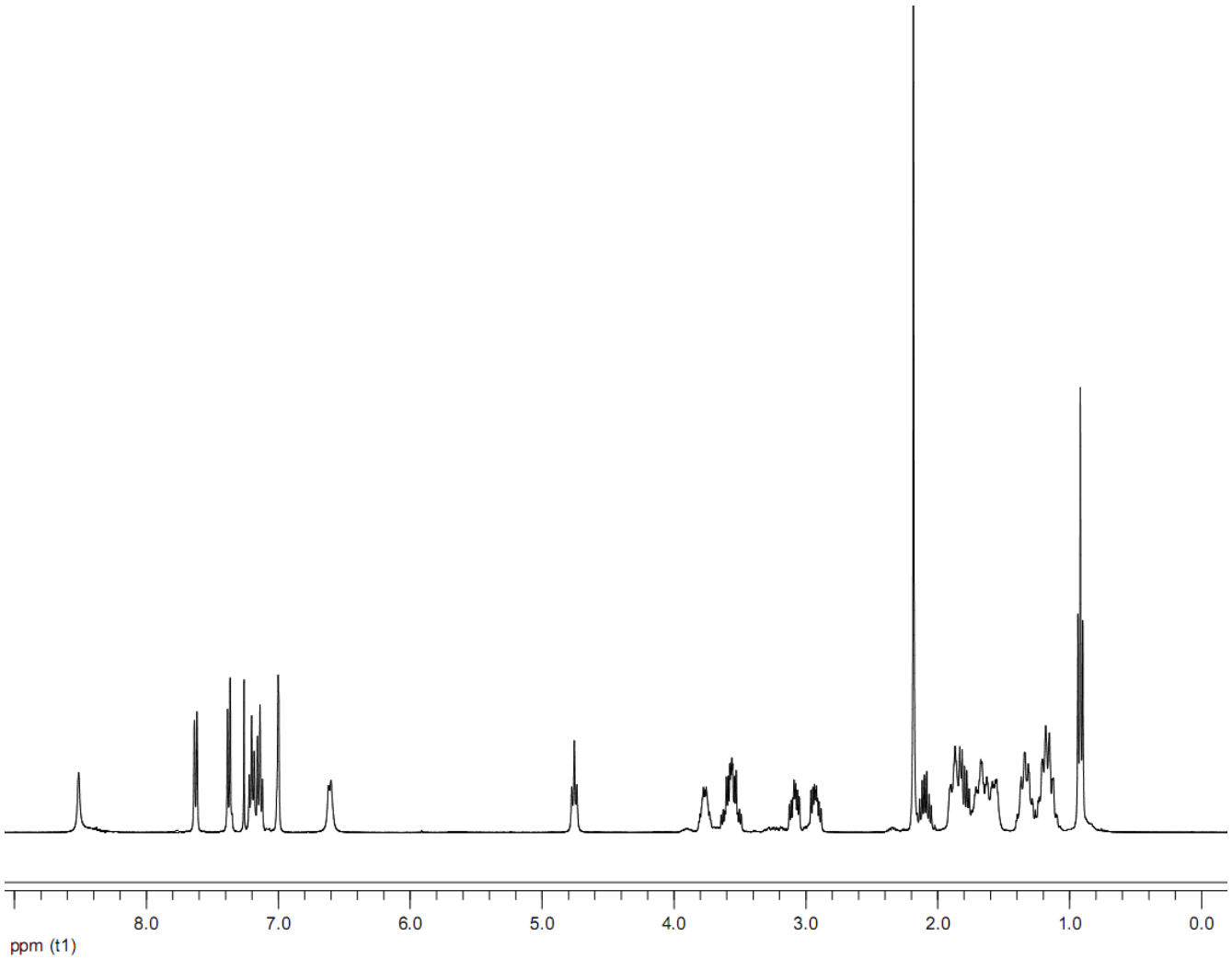
m.p. 140-142 °C.

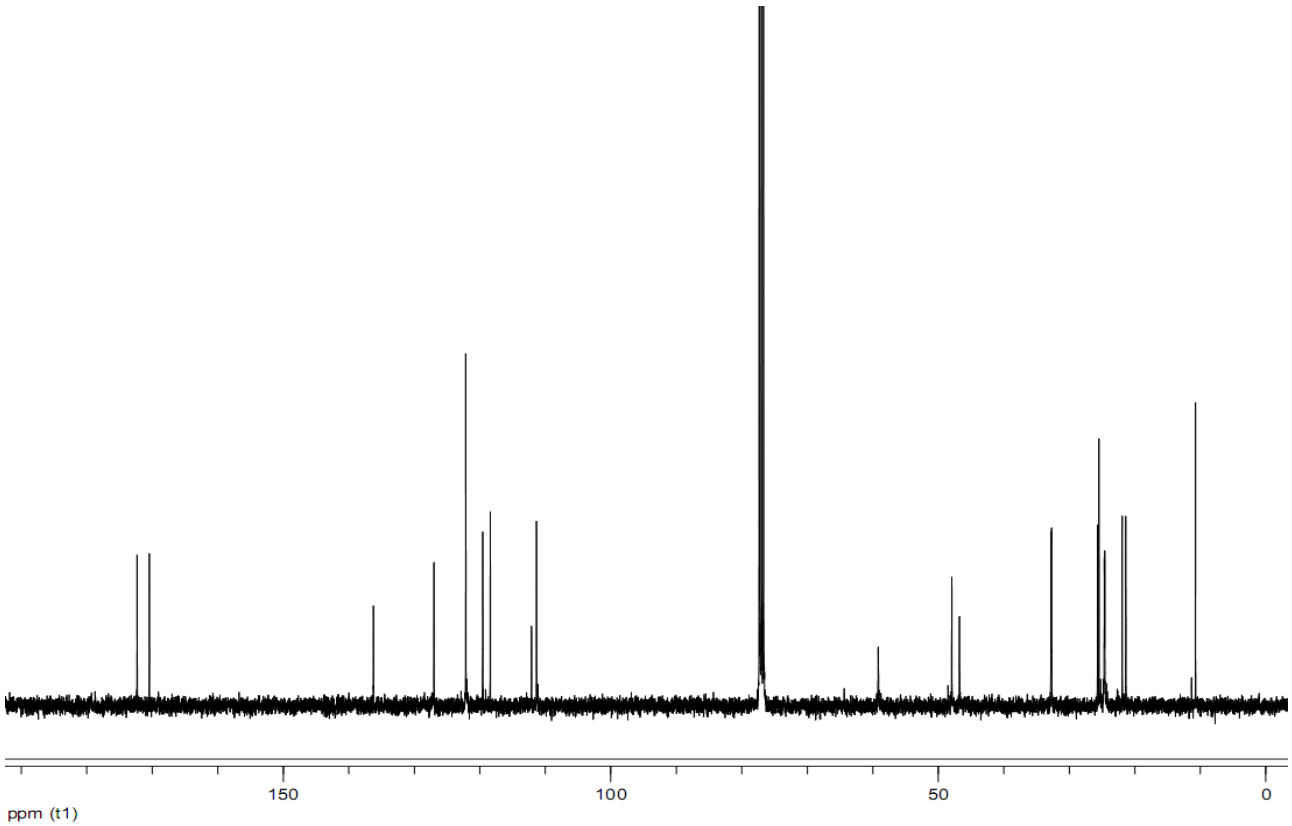
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (br s, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.00 (br s, 1H), 6.61 (br d, *J* = 7.7 Hz, 1H), 4.76 (t, *J* = 7.7 Hz, 1H), 3.81-3.72 (m, 1H), 3.64-3.49 (m, 2H), 3.13-3.05 (m, 1H), 2.96-2.88 (m, 1H), 2.19 (s, 3H), 2.16-2.50 (m, 1H), 1.90-1.76 (m, 3H), 1.71-1.55 (m, 3H), 1.40-1.26 (m, 2H), 1.24-1.09 (m, 3H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.3, 170.4, 136.2, 127.0, 122.1 (2), 119.5, 118.4, 112.1, 111.3, 59.2, 47.9, 46.7, 32.8, 32.7, 25.7, 25.5, 24.6 (2), 21.9, 21.4, 10.7.

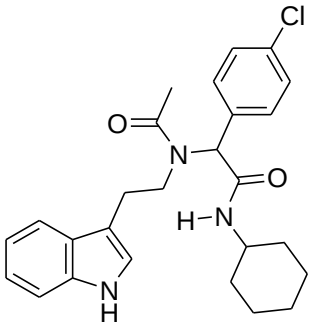
IR (thin film) 3291, 3055, 2930, 1655, 1624, 1534, 1450, 1353, 1294 cm^{-1}.

HRMS Calculated for $C_{22}H_{31}N_3O_2$ 369.2416, found 369.2398.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-2-(4-chlorophenyl)-N-cyclohexylacetamide



$C_{26}H_{30}ClN_3O_2$
MW = 451.99 g/mol

1c

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), cyclohexyl isocyanide (250 µL, 2.0 mmol) and *p*-chlorobenzaldehyde (281 mg, 2.0 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 70:30) as eluant gave the desired product (89%) as a white solid.

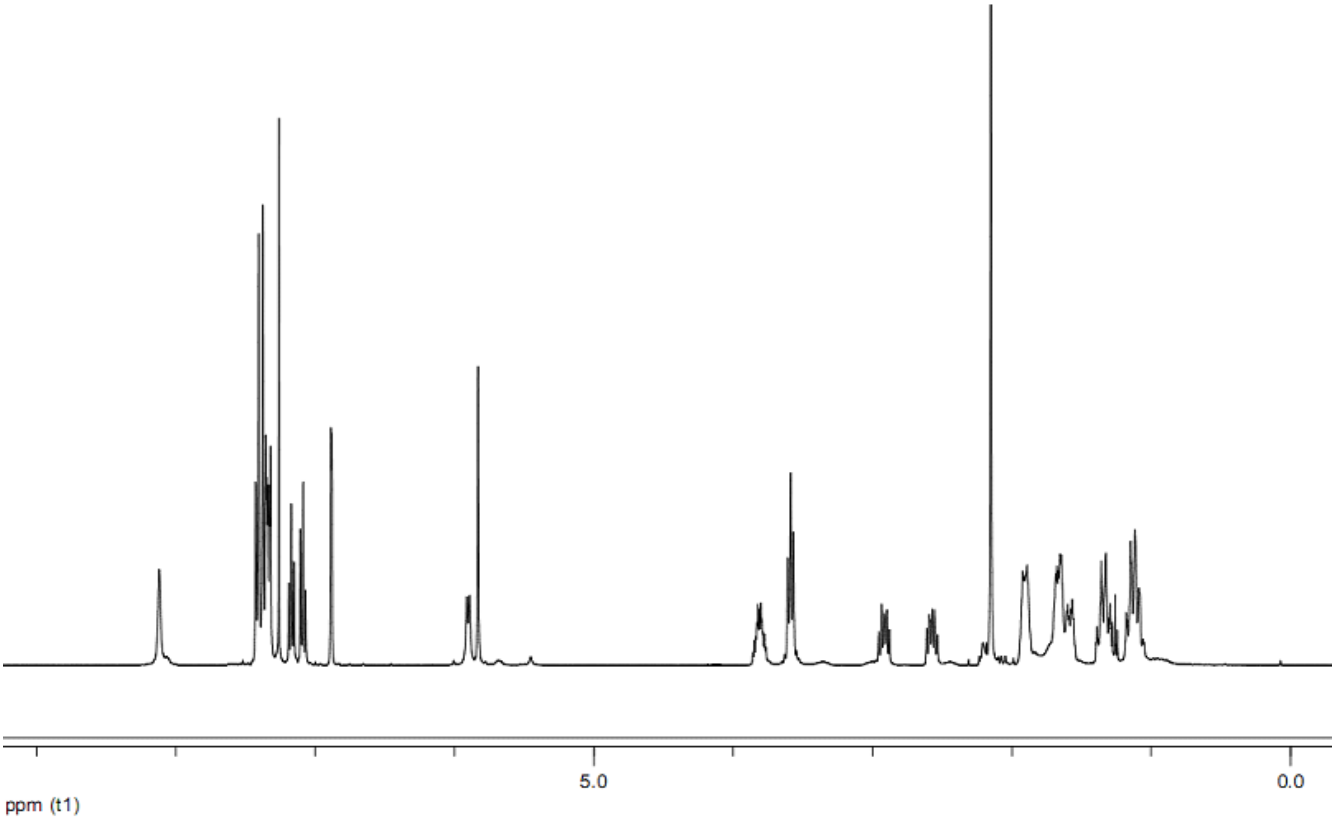
m.p. 179-180 °C.

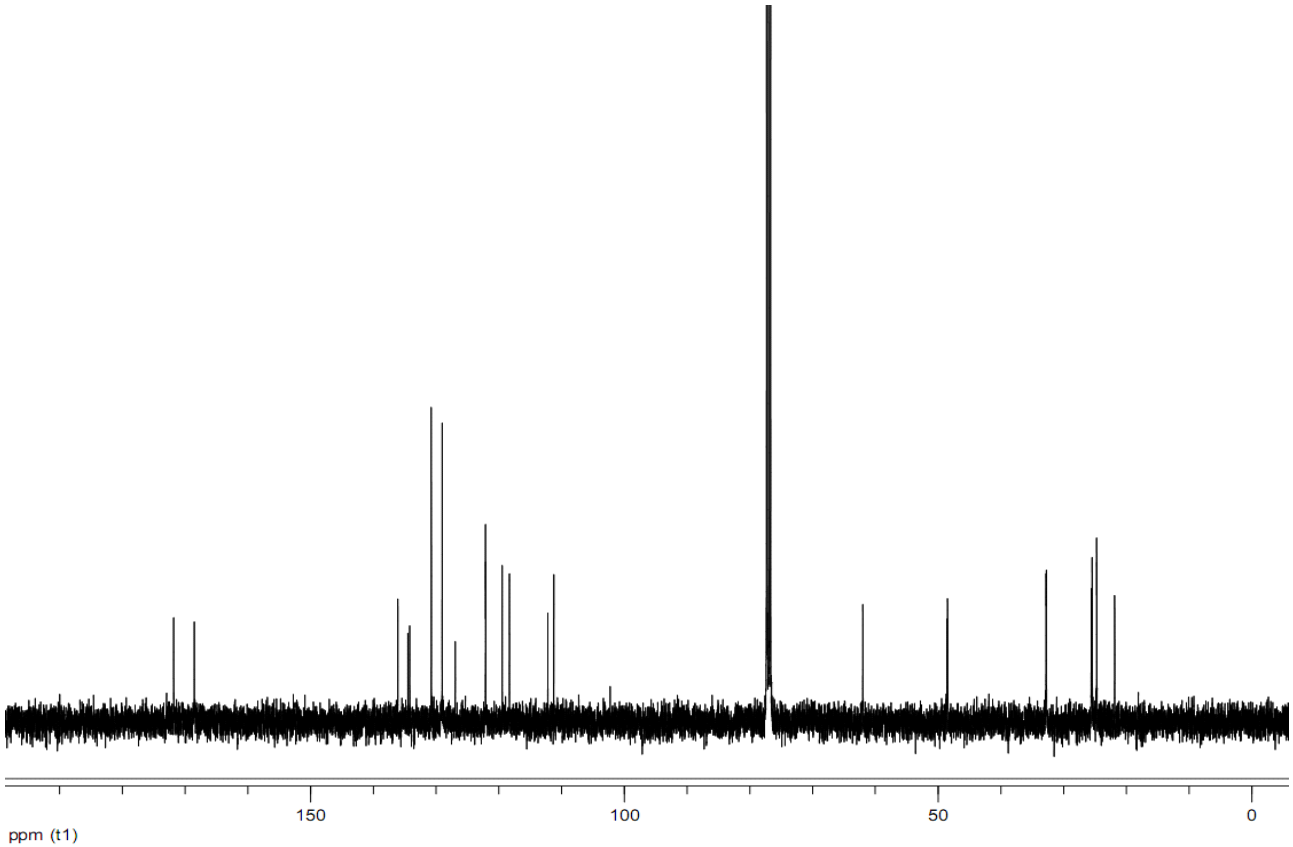
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 1H),), 7.33 (d, *J* = 7.8 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.8 Hz, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 5.90 (br d, *J* = 7.8 Hz, 1H), 5.83 (s, 1H), 3.86-3.77 (m, 1H), 3.59 (t, *J* = 8.1 Hz, 2H), 2.92 (dt, *J* = 15.4, 7.7 Hz, 1H), 2.61-2.54 (m, 1H), 2.15 (s, 3H), 1.92-1.89 (m, 2H), 1.68-1.65 (m, 2H), 1.61-1.56 (m, 1H), 1.40-1.29 (m, 2H), 1.18-1.05 (m, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.8, 168.5, 136.1, 134.5, 134.2, 130.8 (2), 129.0 (2), 126.9, 122.2, 122.1, 119.5, 118.3, 112.2, 111.2, 62.0, 48.6, 48.5, 32.8, 32.7, 25.6, 25.4, 24.7 (2), 21.9.

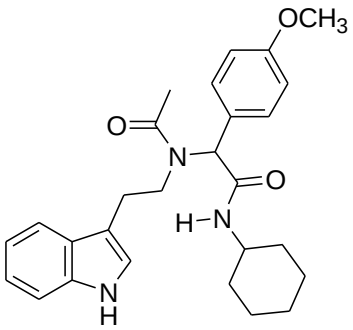
IR (thin film) 3292, 3056, 2932, 2854, 1660, 1621, 1541, 1450, 1294, 1250, 1421 cm^{-1}.

HRMS Calculated for $C_{26}H_{30}ClN_3O_2$ 451.2027, found 451.2027.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-cyclohexyl-2-(4-methoxyphenyl)acetamide



$C_{27}H_{33}N_3O_3$
MW = 447.57 g/mol

1d

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), cyclohexyl isocyanide (250 µL, 2.0 mmol) and *p*-metoxybenzaldehyde (240 µL, 2.0 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 60:40) as eluant gave the desired product (83%) as a white solid.

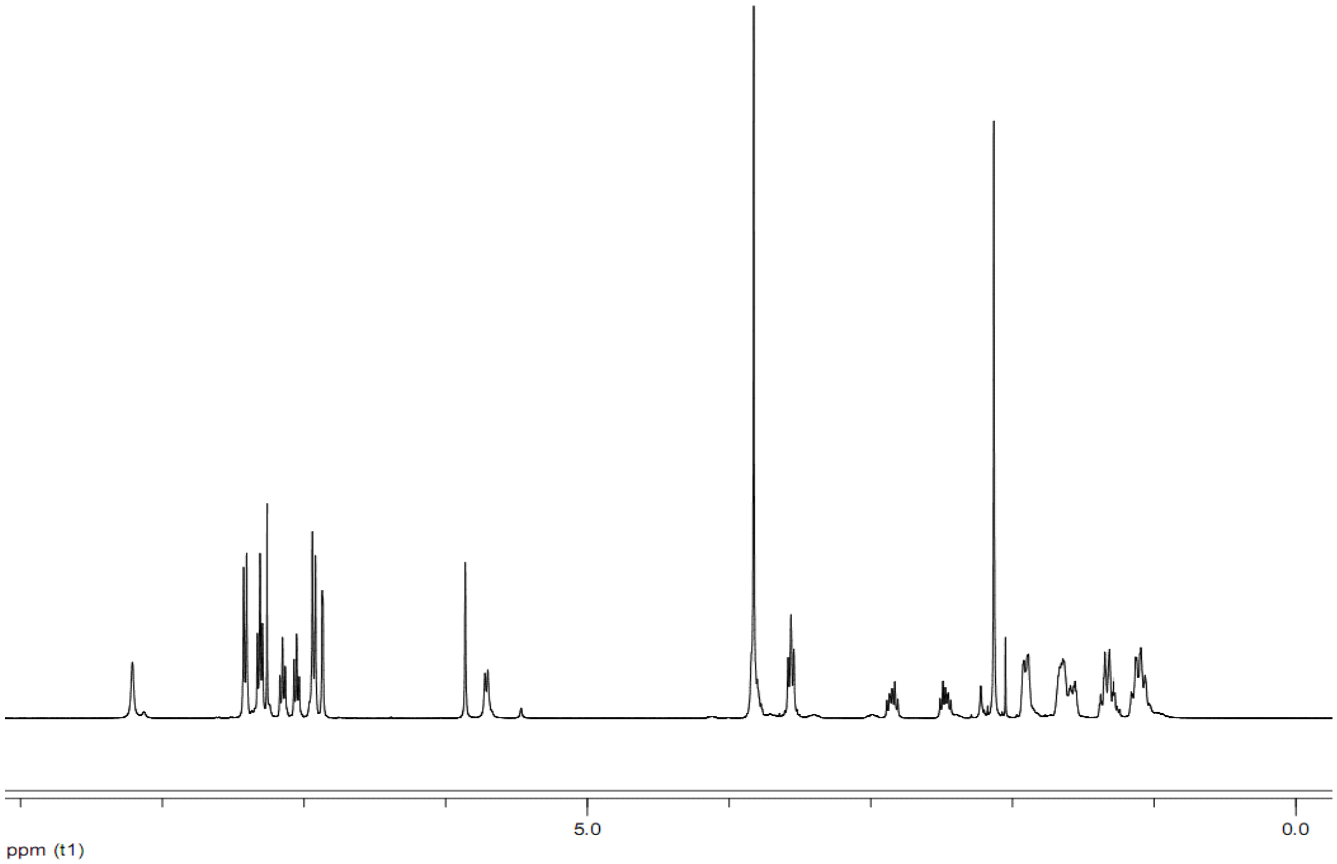
m.p. 198-199 °C.

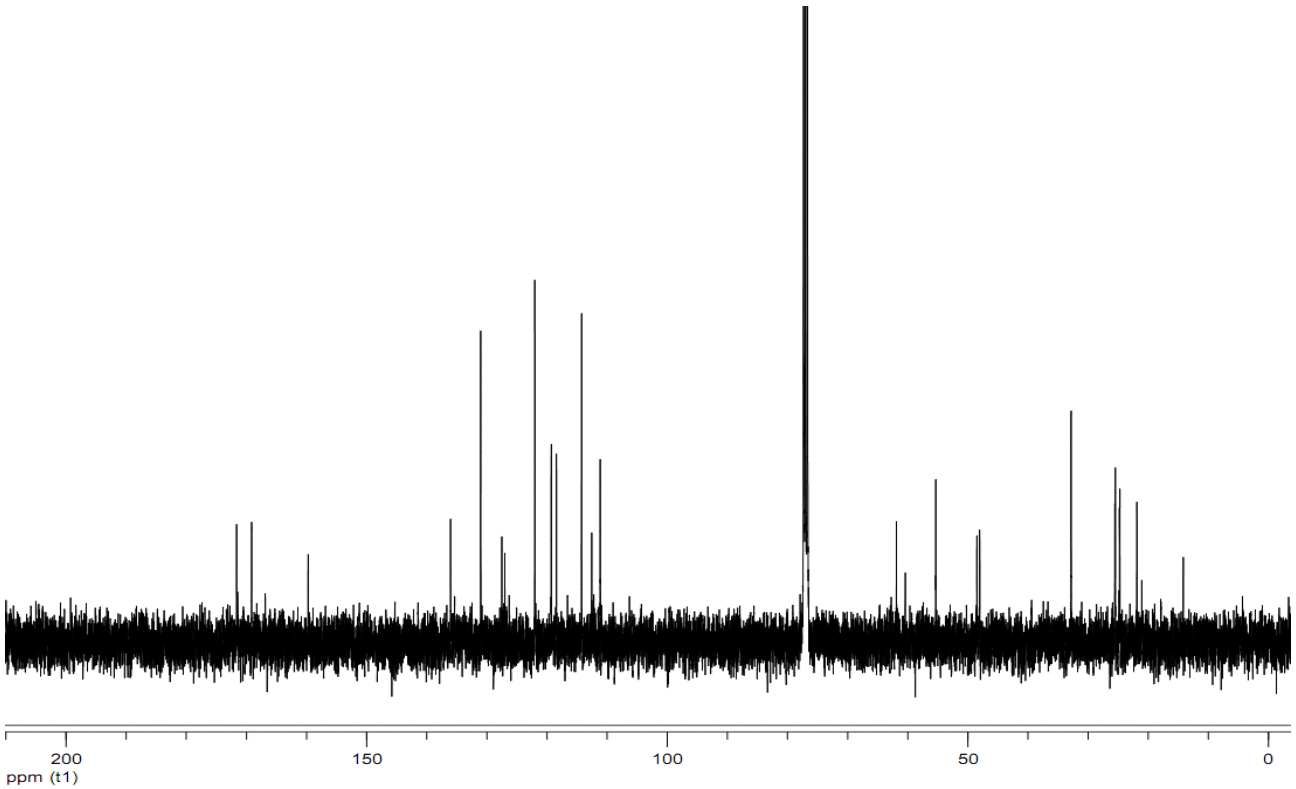
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.30 (d, *J* = 7.4 Hz, 1H), 7.15 (t, *J* = 7.4 Hz, 1H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 2.0 Hz, 1H), 5.86 (s, 1H), 5.71 (br d, *J* = 8.1 Hz, 1H), 3.85-3.77 (m, 1H), 3.83 (s, 3H), 3.56 (t, *J* = 8.1 Hz, 2H), 2.88-2.81 (m, 1H), 2.51-2.43 (m, 1H), 2.13 (s, 3H), 1.93-1.89 (m, 2H), 1.68-1.63 (m, 2H), 1.60-1.55 (m, 1H), 1.38-1.28 (m, 2H), 1.16-1.03 (m, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.6, 169.1, 159.7, 136.0, 131.0 (2), 127.5, 127.0, 122.0 (2), 119.3, 118.4, 114.2, 112.6, 111.1 (2), 61.9, 55.3, 48.5, 48.0, 32.8 (2), 25.5 (2), 24.8, 24.7, 21.9.

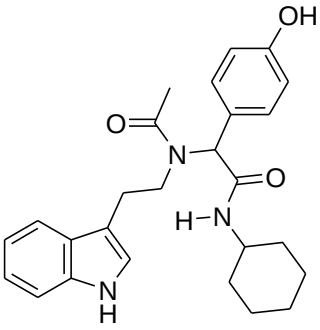
IR (thin film) 3292, 3056, 2932, 2850, 1656, 1625, 1577, 1464, 1253, 1034 cm^{-1}.

HRMS Calculated for $C_{27}H_{33}N_3O_3$ 447.2522, found 447.2520.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-cyclohexyl-2-(4-hydroxyphenyl)acetamide



$C_{26}H_{31}N_3O_3$
MW = 433.54 g/mol

1e

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 μL, 2.0 mmol), cyclohexyl isocyanide (250 μL, 2.0 mmol) and *p*-hydroxybenzaldehyde (302 mg, 2.0 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 – 70:30) as eluant gave the desired product (93%) as a white solid.

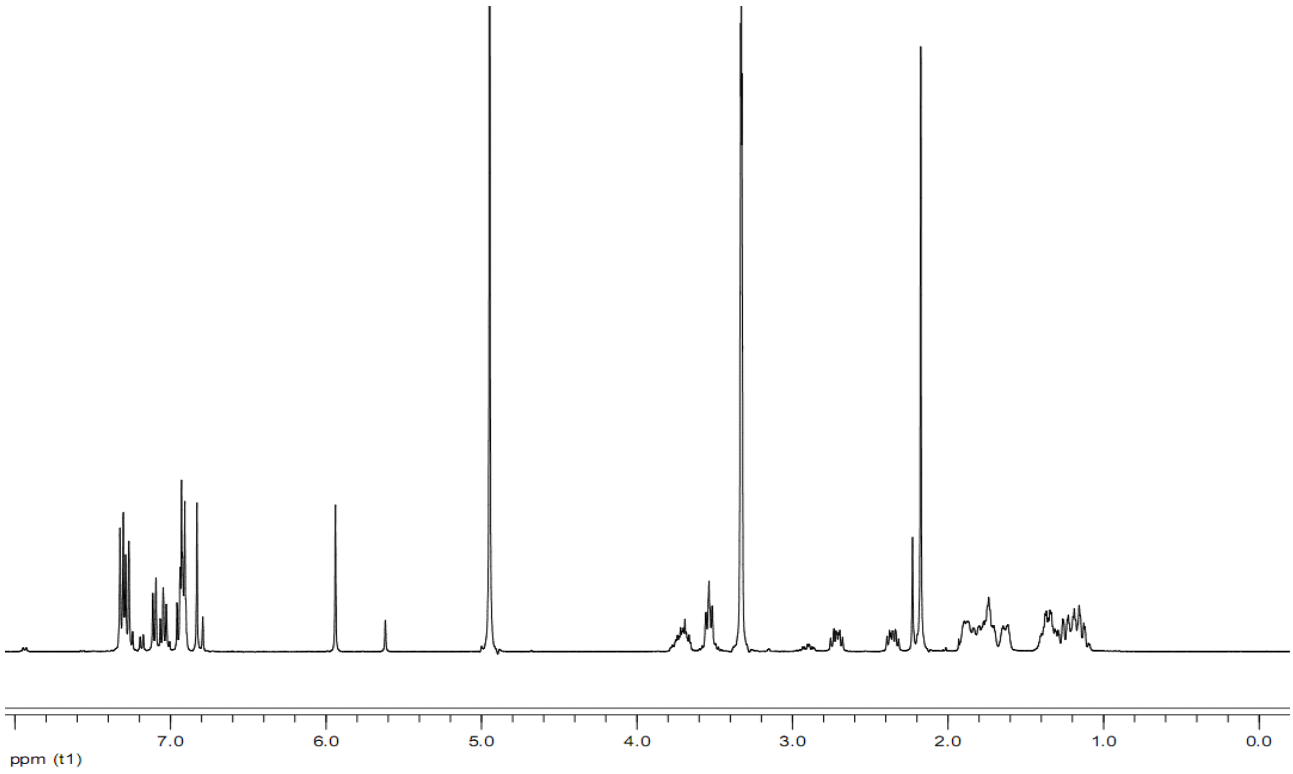
m.p. 167-169 °C.

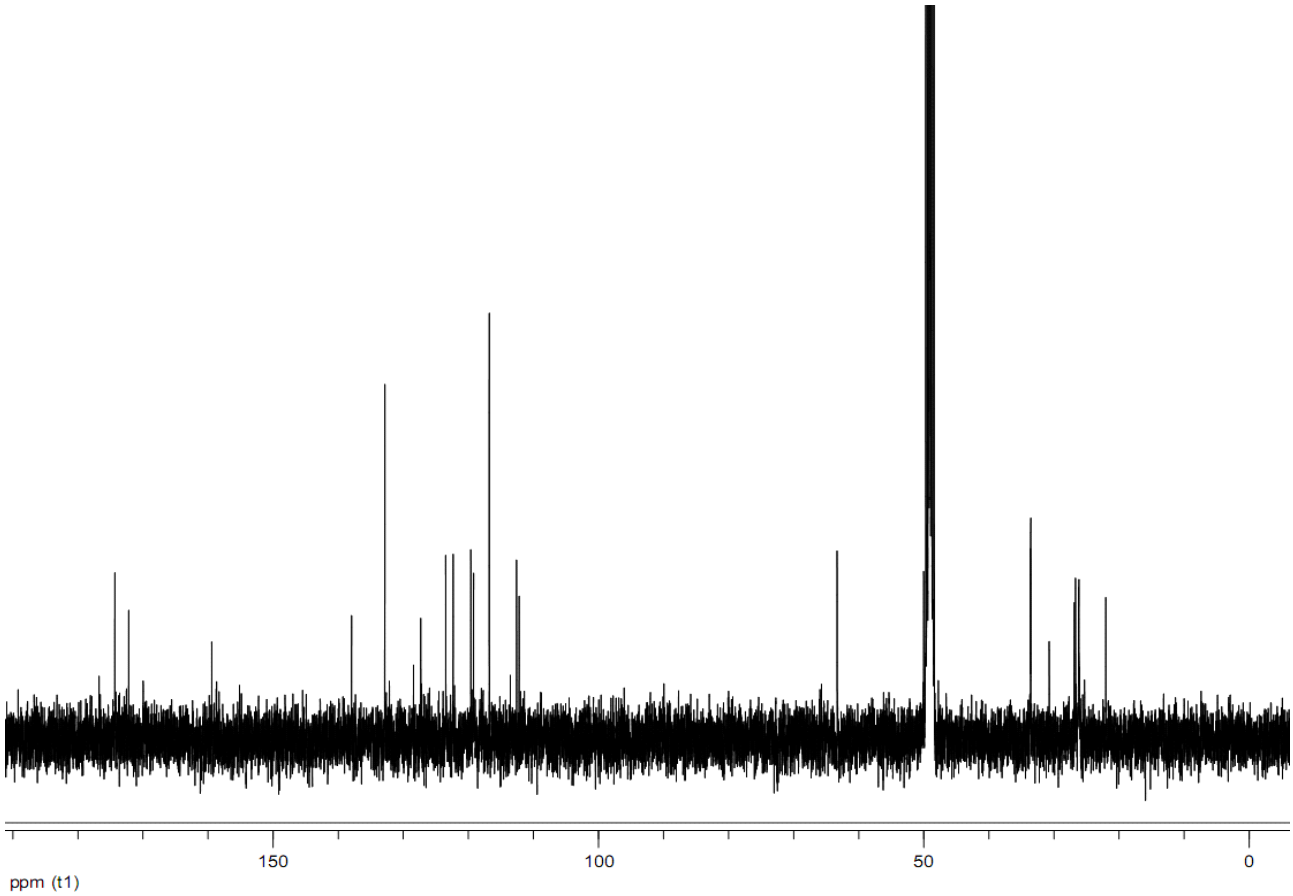
^{1}H NMR (400 MHz, CD_3OD) δ 7.30 (d, *J* = 8.6 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 6.94-6.88 (m, 3H) 6.81 (s, 1H), 5.91 (s, 1H), 3.73-3.64 (m, 1H), 3.52 (t, *J* = 8.5 Hz, 2H), 2.70 (td, *J* = 12.6, 4.8 Hz, 1H), 2.37-2.30 (m, 1H), 2.16 (s, 3H), 1.88-1.69 (m, 4H), 1.63-1.60 (m, 1H), 1.30-1.27 (m, 2H), 1.25-1.07 (m, 3H).

^{13}C NMR (100.6 MHz, CD_3OD) δ 174.3, 172.2, 159.4, 138.0, 132.8 (2), 127.3, 123.5, 122.3, 119.6, 119.2, 116.8 (2), 112.6, 112.2, 63.3, 50.0, 49.1, 33.6 (2), 30.7, 26.9, 26.7, 26.2 (2), 22.0.

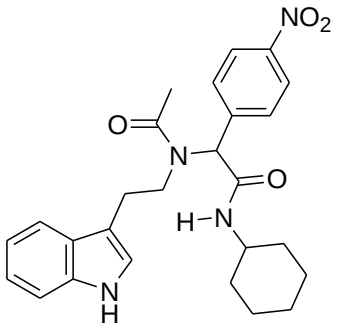
IR (thin film) 3285, 2928, 2853, 1656, 1616, 1456, 1364, 1230 cm^{-1}.

HRMS Calculated for $C_{26}H_{31}N_3O_3$ 433.2365, found 433.2375.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-cyclohexyl-2-(4-nitrophenyl)acetamide



$C_{26}H_{30}N_4O_4$
MW = 462.54 g/mol

1f

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), cyclohexyl isocyanide (250 µL, 2.0 mmol) and *p*-nitrobenzaldehyde (302 mg, 2.0 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 – 70:30) as eluant gave the desired product (90%) as a yellow solid.

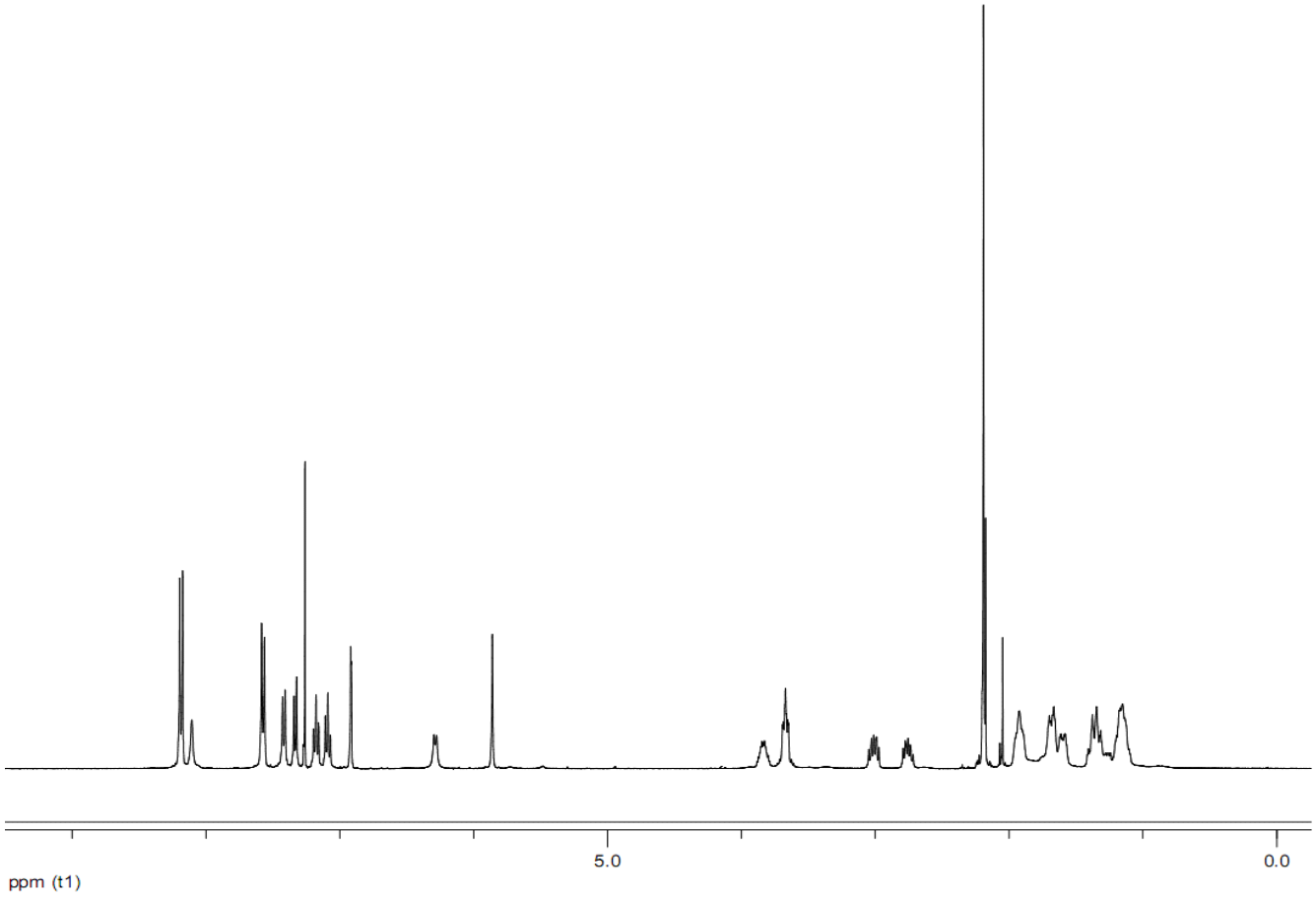
m.p. 125-126 °C.

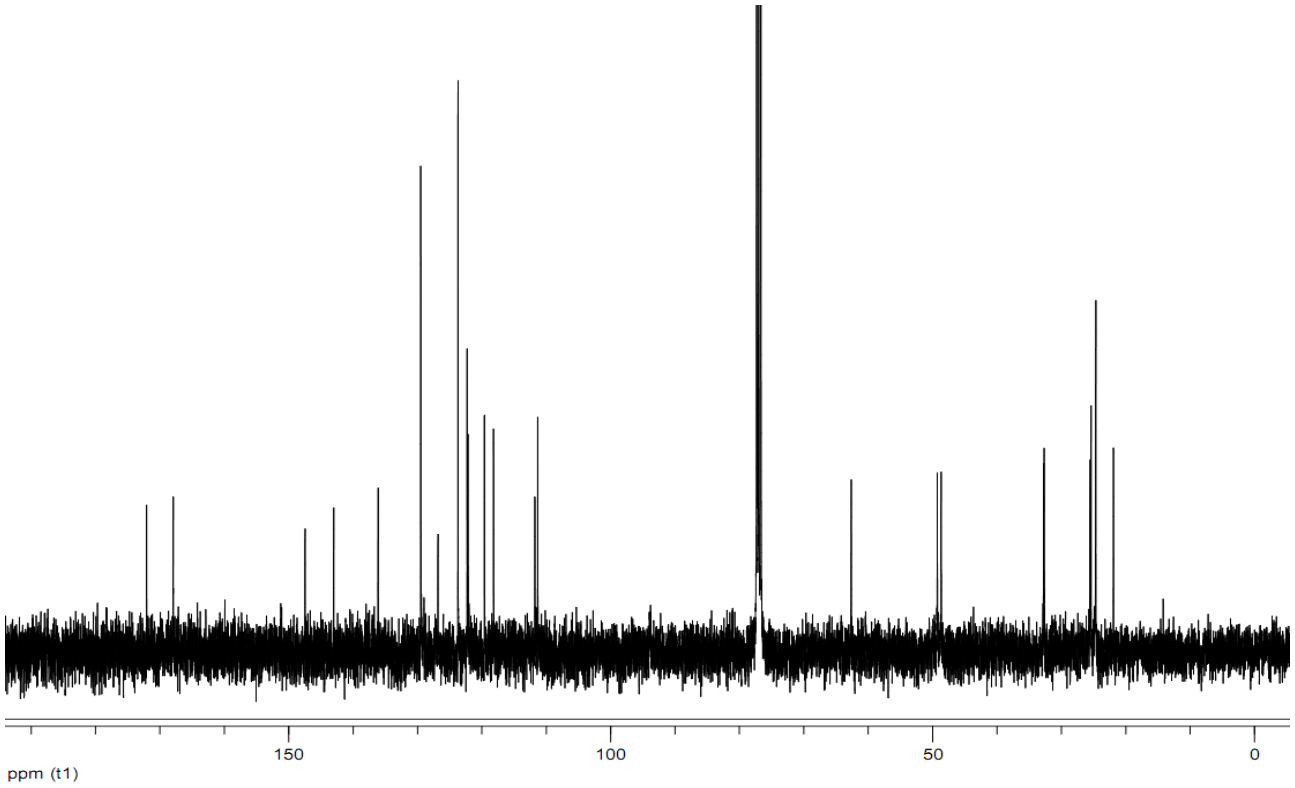
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.8 Hz, 2H), 8.04 (br s, 1H), 7.58 (d, *J* = 8.8 Hz, 2H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.18 (t, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.8 Hz, 1H), 6.92 (d, *J* = 2.0 Hz, 1H), 6.26 (br d, *J* = 7.3 Hz, 1H), 5.85 (s, 1H), 3.88-3.79 (m, 1H), 3.69-3.65 (m, 2H), 3.05-2.97 (m, 1H), 2.79-2.72 (m, 1H), 2.19 (s, 3H), 1.96-1.89 (m, 2H), 1.71-1.66 (m, 2H), 1.62-1.58 (m, 1H), 1.42-1.31 (m, 2H), 1.21-1.10 (m, 3H).

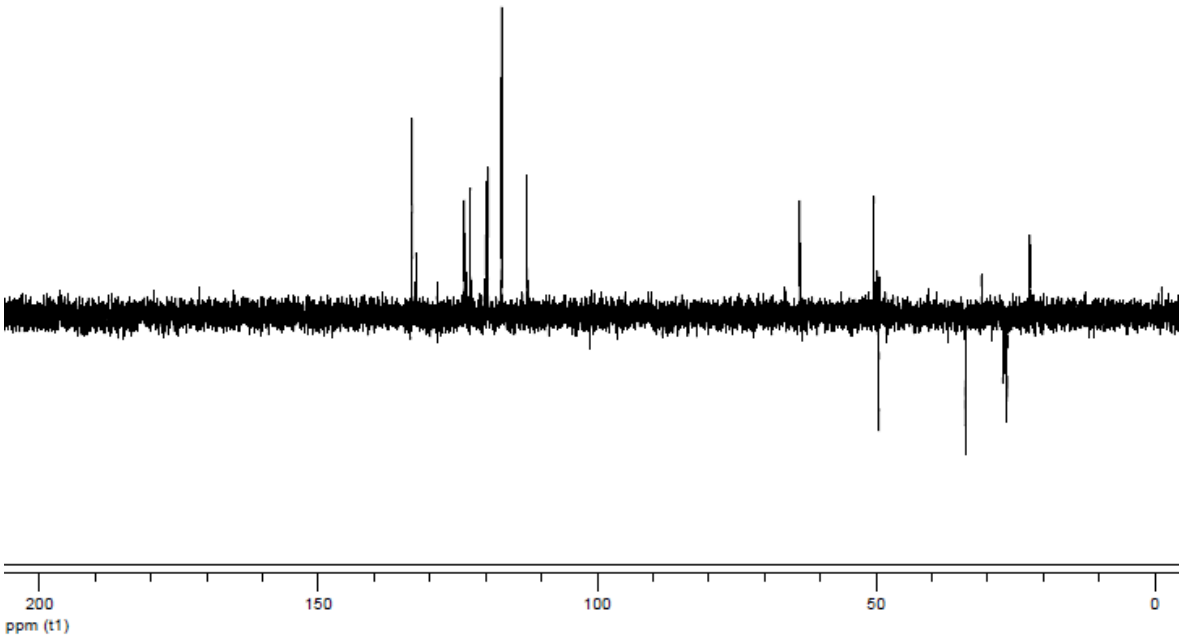
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.1, 168.0, 147.5, 143.0, 136.1, 129.5 (2), 126.8, 123.7 (2), 122.3, 122.1, 119.6, 118.2, 111.8, 111.3, 62.6, 48.7, 48.3, 32.8, 32.7, 25.5, 25.4, 24.7 (2), 21.9.

IR (thin film) 3296, 3061, 2936, 2855, 1652, 1628, 1456, 1352 cm^{-1}.

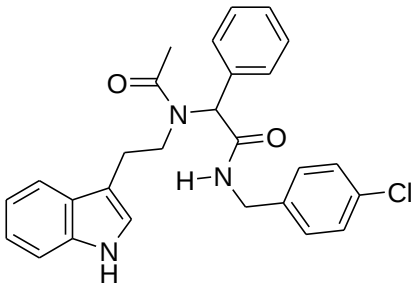
HRMS Calculated for $C_{26}H_{30}N_4O_4$ 462.2267, found 462.2272.







2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-(4-chlorobenzyl)-2-phenylacetamide



$C_{27}H_{26}ClN_3O_2$
MW = 459.97 g/mol

1g

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 μL, 2.0 mmol), *p*-chlorobenzyl isocyanide (255 μL, 2.0 mmol) and benzaldehyde (200 μL, 2.0 mmol). Reaction time: 4 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 70:30) as eluant gave the desired product (93%) as a white solid.

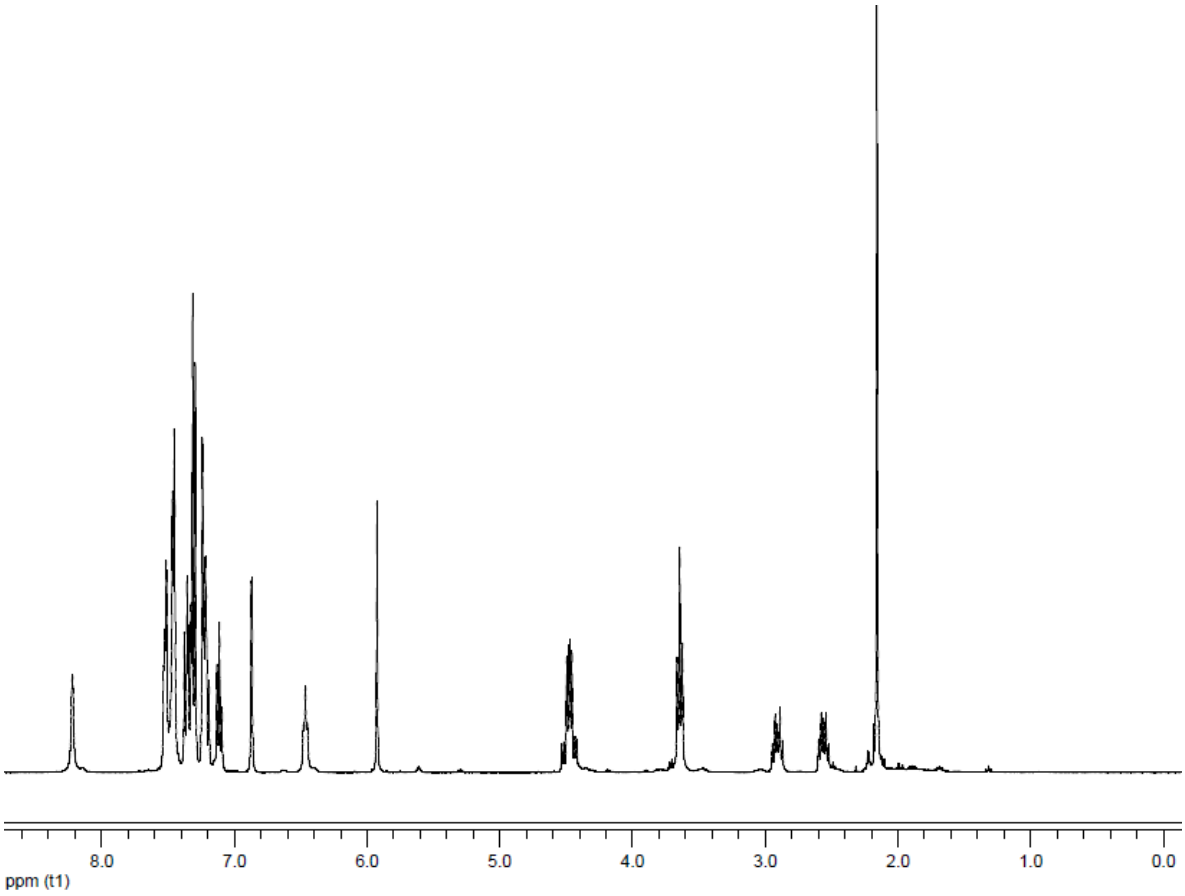
m.p. 180-181 °C.

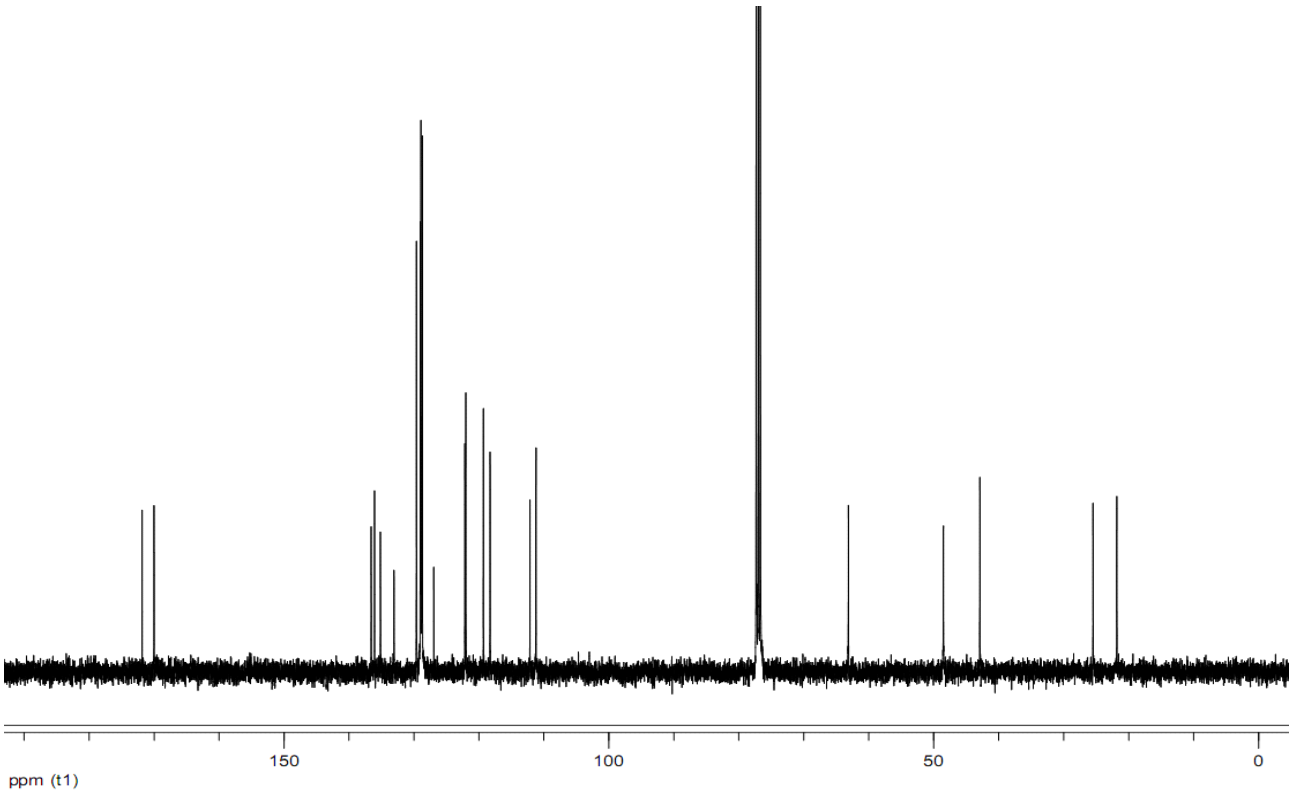
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (br s, 1H), 7.53-7.51 (m, 2H), 7.49-7.45 (m, 3H), 7.37 (d, *J* = 7.9 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.30 (d, *J* = 7.9 Hz, 2H), 7.24-7.19 (m, 3H), 7.11 (t, *J* = 7.9 Hz, 1H), 6.87 (d, *J* = 2.0 Hz, 1H), 6.46 (br t, *J* = 5.7 Hz, 1H), 5.92 (s, 1H), 4.51 (dd, *J* = 15.2, 6.1 Hz, 1H), 4.44 (dd, *J* = 15.2, 6.1 Hz, 1H), 3.64 (t, *J* = 8.1 Hz, 2H), 2.91 (dt, *J* = 14.6, 7.6 Hz, 1H), 2.83 (dt, *J* = 14.6, 7.6 Hz, 1H), 2.16 (s, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.8, 170.0, 136.6, 136.1, 135.2, 133.1, 129.6 (2), 129.0 (2), 128.9 (2), 128.8, 128.7 (2), 126.9, 122.1, 122.0, 119.3, 118.3, 112.1, 111.2, 68.1, 48.5, 42.9, 25.5, 21.8.

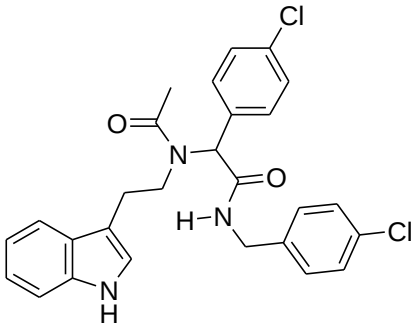
IR (thin film) 3280, 3059, 2925, 1665, 1629, 1564, 1453 cm^{-1}.

HRMS Calculated for $C_{27}H_{26}ClN_3O_2$ 459.1714, found 459.1702.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-(4-chlorobenzyl)-2-(4-chlorophenyl)acetamide



$C_{27}H_{25}Cl_2N_3O_2$
MW = 494.41 g/mol

1h

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), *p*-chlorobenzyl isocyanide (255 µL, 2.0 mmol) and *p*-chlorobenzaldehyde (281 mg, 2.0 mmol). Reaction time: 24 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 70:30) as eluant gave the desired product (88%) as a white solid.

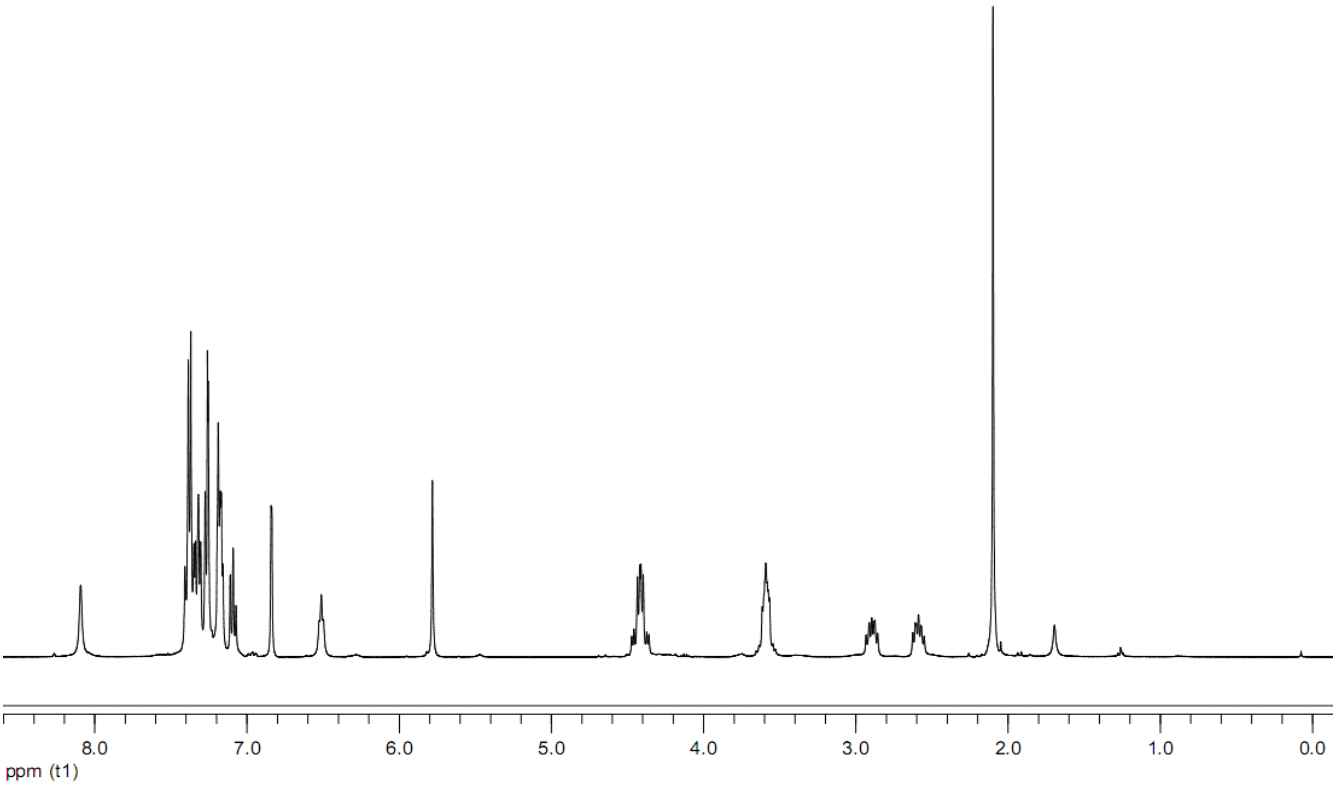
m.p. 190-191 °C.

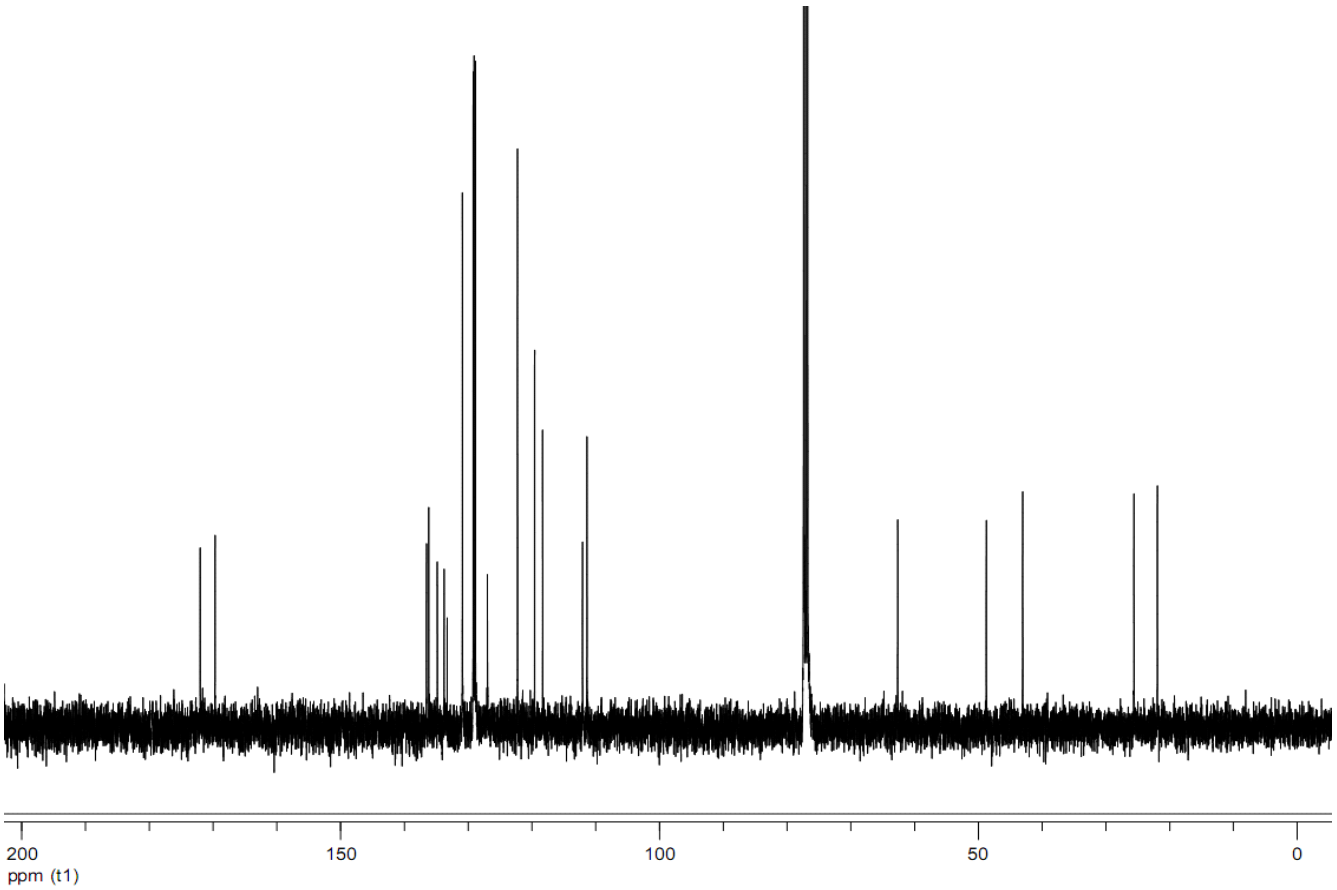
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (br s, 1H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.27-7.22 (m, 2H), 7.19 (d, *J* = 8.5 Hz, 2H), 7.11-7.08 (m, 3H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.76 (d, *J* = 1.8 Hz, 1H), 6.44 (br t, *J* = 5.3 Hz, 1H), 5.71 (s, 1H), 4.37 (dd, *J* = 15.2, 6.1 Hz, 1H), 4.31 (dd, *J* = 15.2, 6.1 Hz, 1H), 3.54-3.49 (m, 2H), 2.86-2.78 (m, 1H), 2.55-2.48 (m, 1H), 2.02 (s, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.9, 169.7, 136.4, 136.1, 134.7, 133.7, 133.2, 130.8 (2), 129.1 (2), 129.0 (2), 128.8 (2), 126.9, 122.2 (2), 119.5, 118.2, 112.0, 111.3, 62.5, 48.7, 43.0, 25.5, 21.8.

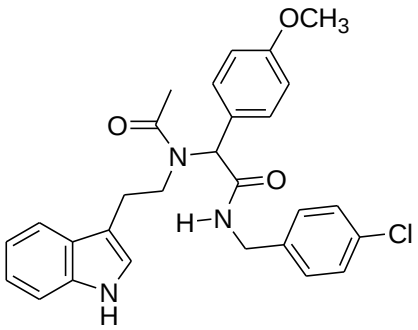
IR (thin film) 3292, 3058, 2926, 1668, 1623, 1525, 1456, 1356, 1014 cm^{-1}.

HRMS Calculated for $C_{27}H_{25}Cl_2N_3O_2$ 493.1324, found 493.1314.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-(4-chlorobenzyl)-2-(4-methoxyphenyl)acetamide



$C_{28}H_{28}ClN_3O_3$
MW = 489.99 g/mol

1i

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 μL, 2.1 mmol), *p*-chlorobenzyl isocyanide (255 μL, 2.0 mmol) and *p*-metoxybenzaldehyde (240 μL, 2.0 mmol). Reaction time: 24h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 70:30) as eluant gave the desired product (84%) as a white solid.

m.p. 209-210 °C.

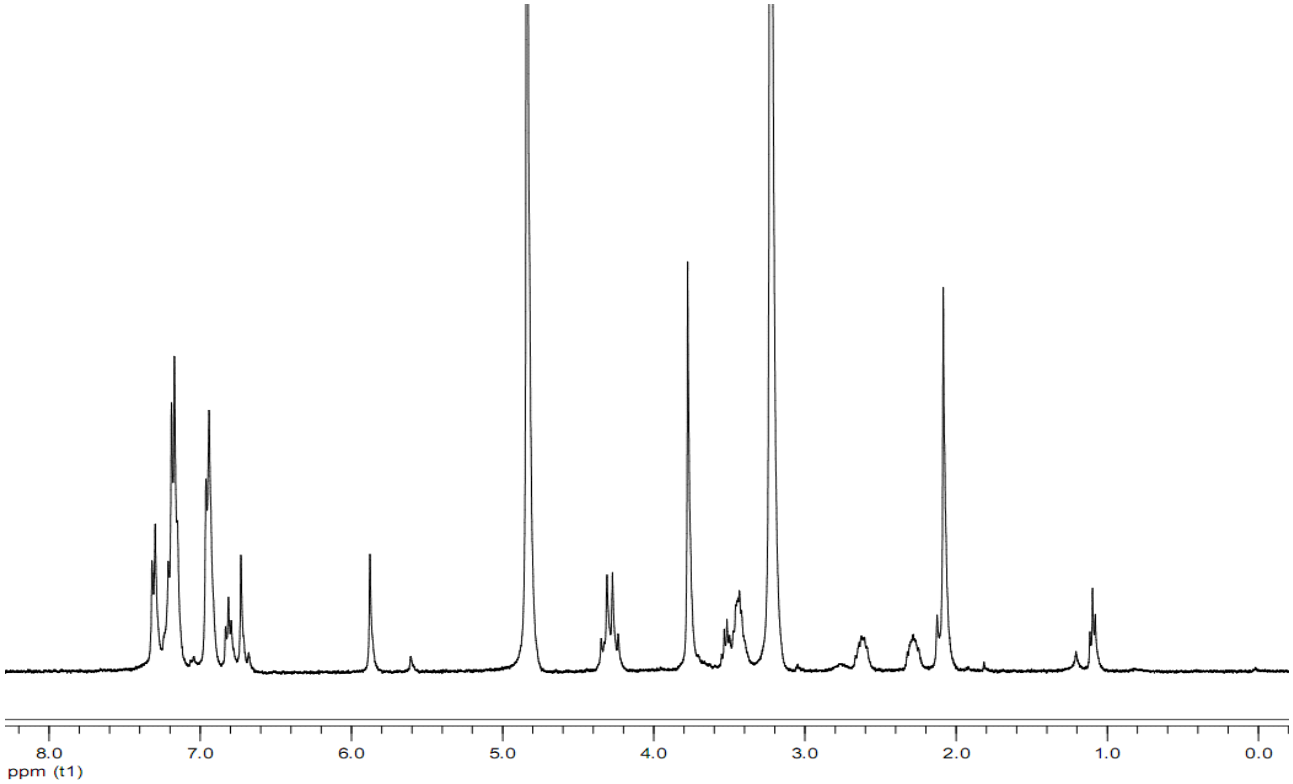
^{1}H NMR (400 MHz, CD_3OD) δ 7.31 (d, *J* = 8.6 Hz, 2H), 7.21-7.15 (m, 6H), 6.96-6.94 (m, 4H), 6.81 (t, *J* = 7.3 Hz, 2H), 6.73 (s, 1H), 5.88 (s, 1H), 4.34-4.24 (m, 2H), 3.47-3.40 (m, 2H), 3.78 (s, 3H), 2.67-2.59 (m, 1H), 2.32-2.25 (m, 1H), 2.10 (s, 3H).

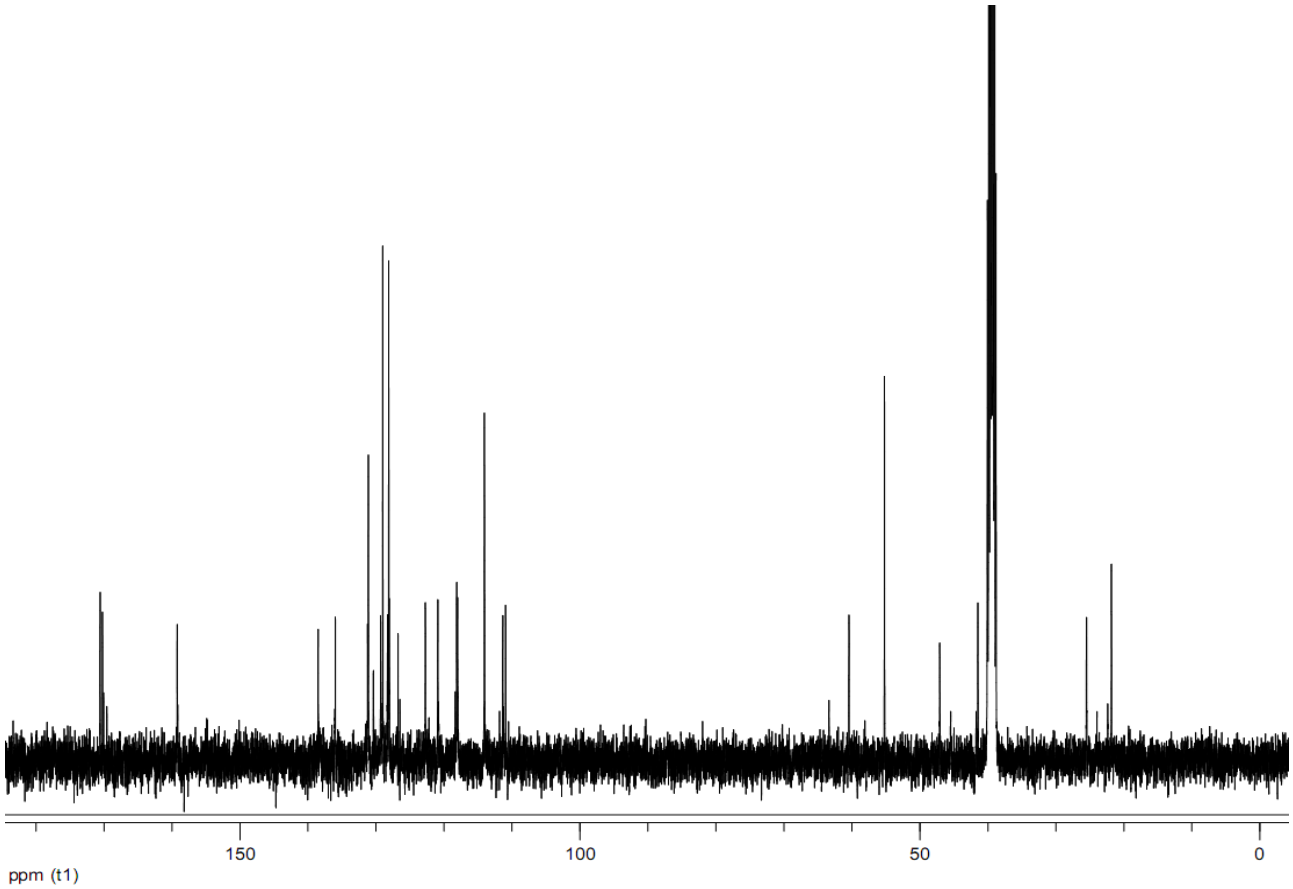
(mm123-DM/10)

^{13}C NMR (100.6 MHz, DMSO) δ 170.6, 170.21, 159.2, 138.5, 136.0, 131.2, 131.1, 129.3, 129.0 (2), 128.3, 128.1 (2), 128.0, 126.7, 122.7, 120.9, 118.1, 118.0, 114.0, 111.3, 110.9, 60.4, 55.2, 47.0, 41.5, 25.5, 21.8.

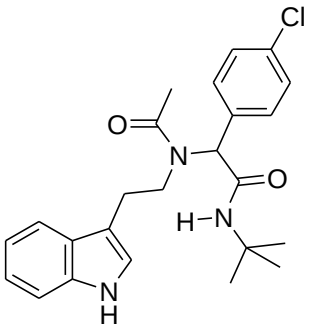
IR (thin film) 3300, 3061, 2924, 2852, 1660, 1625, 1515, 1458, 1354, 1280, 1030 cm^{-1}.

HRMS Calculated for $C_{28}H_{28}ClN_3O_3$ 489.1819, found 489.1819.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-tert-butyl-2-(4-chlorophenyl)acetamide



$C_{24}H_{28}ClN_3O_2$
MW = 425.95 g/mol

1j

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 µL, 2.0 mmol), *t*-butyl isocyanide (230 µL, 2.0 mmol) and *p*-chlorobenzaldehyde (281 mg, 2.0 mmol). Reaction time: 24 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 – 70:30) as eluant gave the desired product (94%) as a white solid.

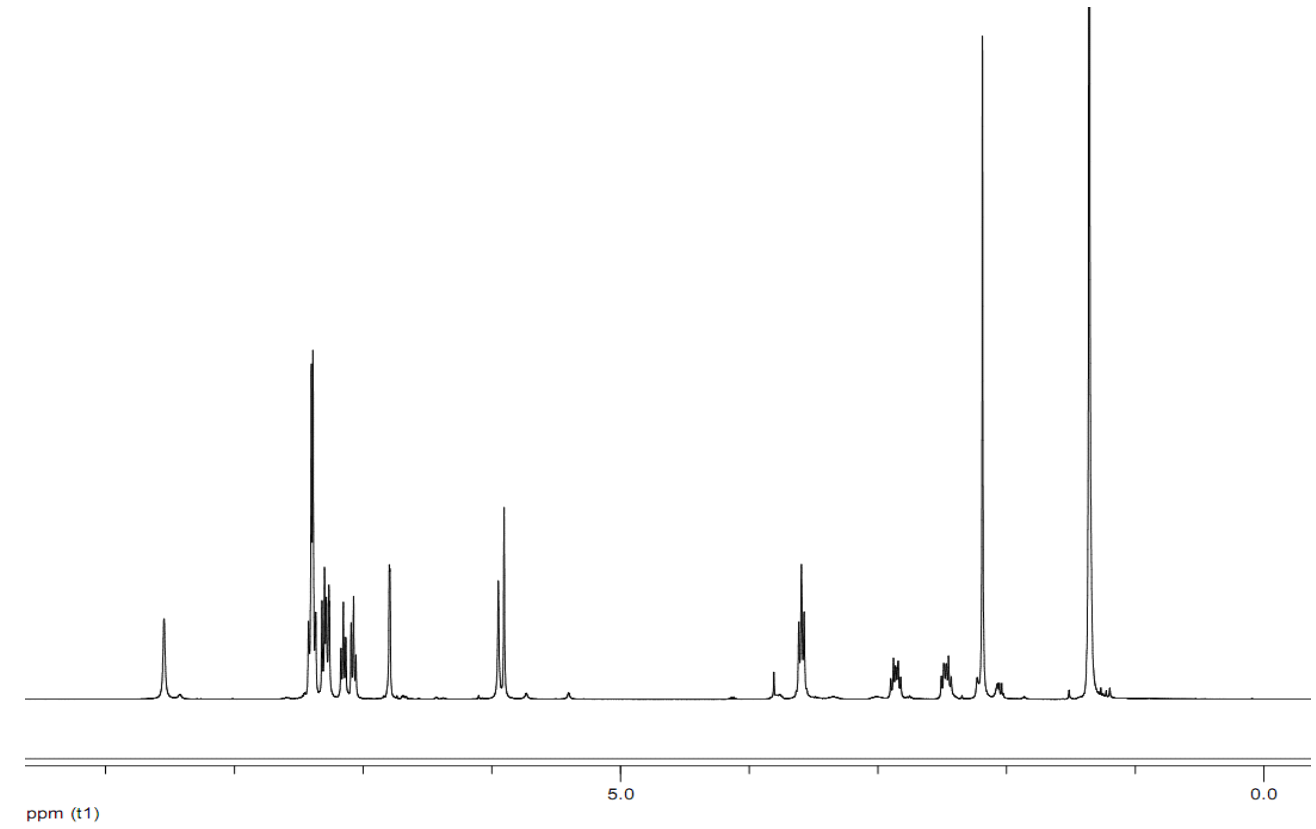
m.p. 177-178 °C.

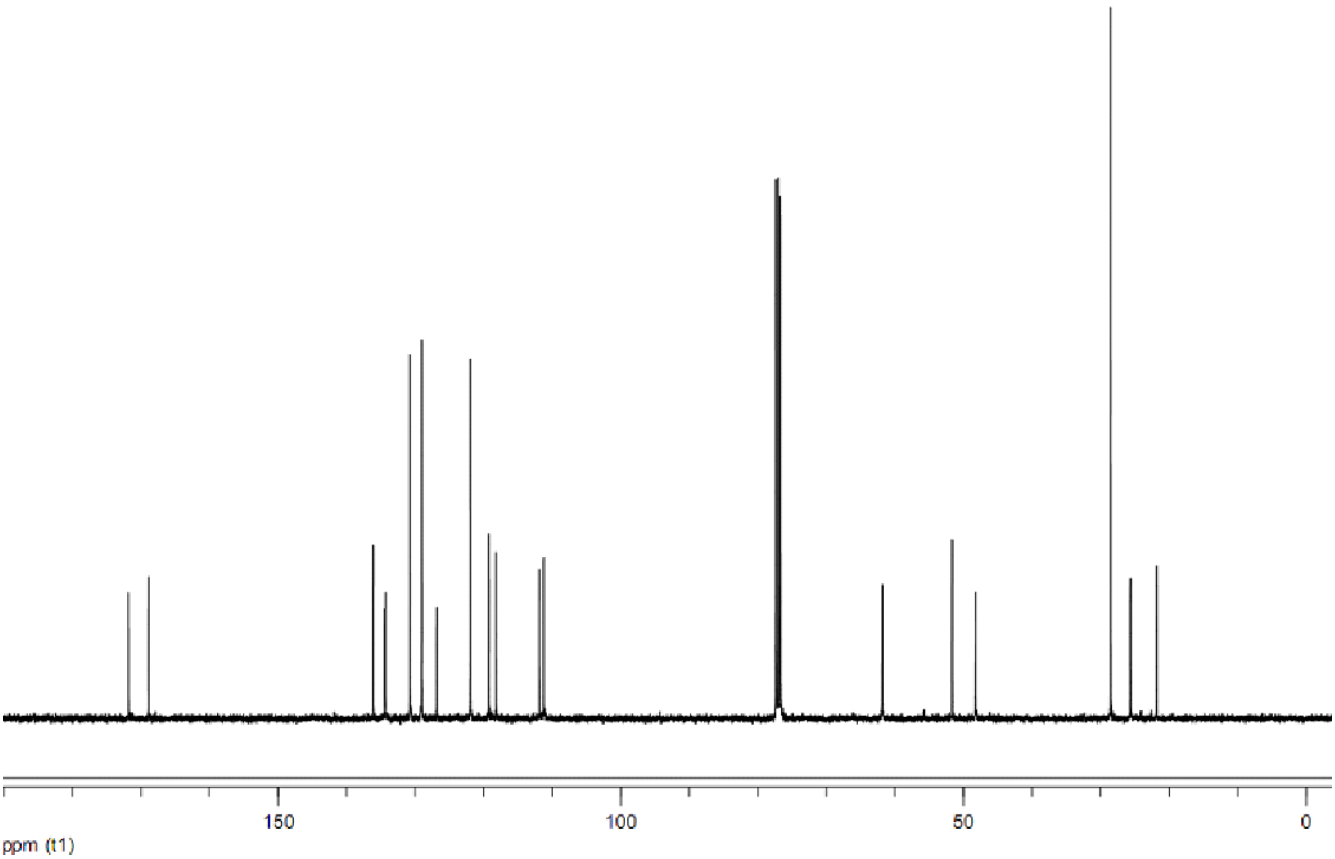
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 (br s, 1H), 7.41 (d, *J* = 8.8, 2H), 7.38 (d, *J* = 8.8, 2H), 7.31 (d, *J* = 7.7, 1H), 7.28 (d, *J* = 7.7, 1H), 7.15 (t, *J* = 7.7 Hz, 1H), 7.07 (t, *J* = 7.7 Hz, 1H), 6.79 (d, *J* = 1.8 Hz, 1H), 5.95 (br s, 1H), 5.90 (s, 1H), 3.59 (t, *J* = 8.0 Hz, 2H), 2.86 (dt, *J* = 14.8, 8.0 Hz, 1H), 2.47 (dt, *J* = 14.8, 8.0 Hz, 1H), 2.19 (s, 3H), 1.36 (s, 9H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.8, 168.8, 136.1, 134.4, 134.3, 130.8 (2), 129.0 (2), 126.8, 121.9 (2), 119.2, 118.2, 111.9, 111.3, 61.8, 51.6, 48.2, 28.5 (3), 25.7, 21.8.

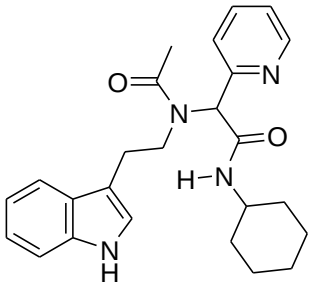
IR (thin film) 3303, 3061, 2971, 2931, 1675, 1629, 1542, 1492, 1367, 1230, 1019 cm^{-1}.

HRMS calculated for $C_{24}H_{28}ClN_3O_2$ 425.1870, found 425.1887.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-cyclohexyl-2-(pyridin-2-yl)acetamide



$C_{25}H_{30}N_4O_2$
MW = 418.53 g/mol

1k

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 μL, 2.0 mmol), cyclohexyl isocyanide (250 μL, 2.0 mmol) and pyridine-2-carbaldehyde (190 μL, 2.0 mmol). Reaction time: 20 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 90:10) as eluant gave the desired product (93%) as a beige solid.

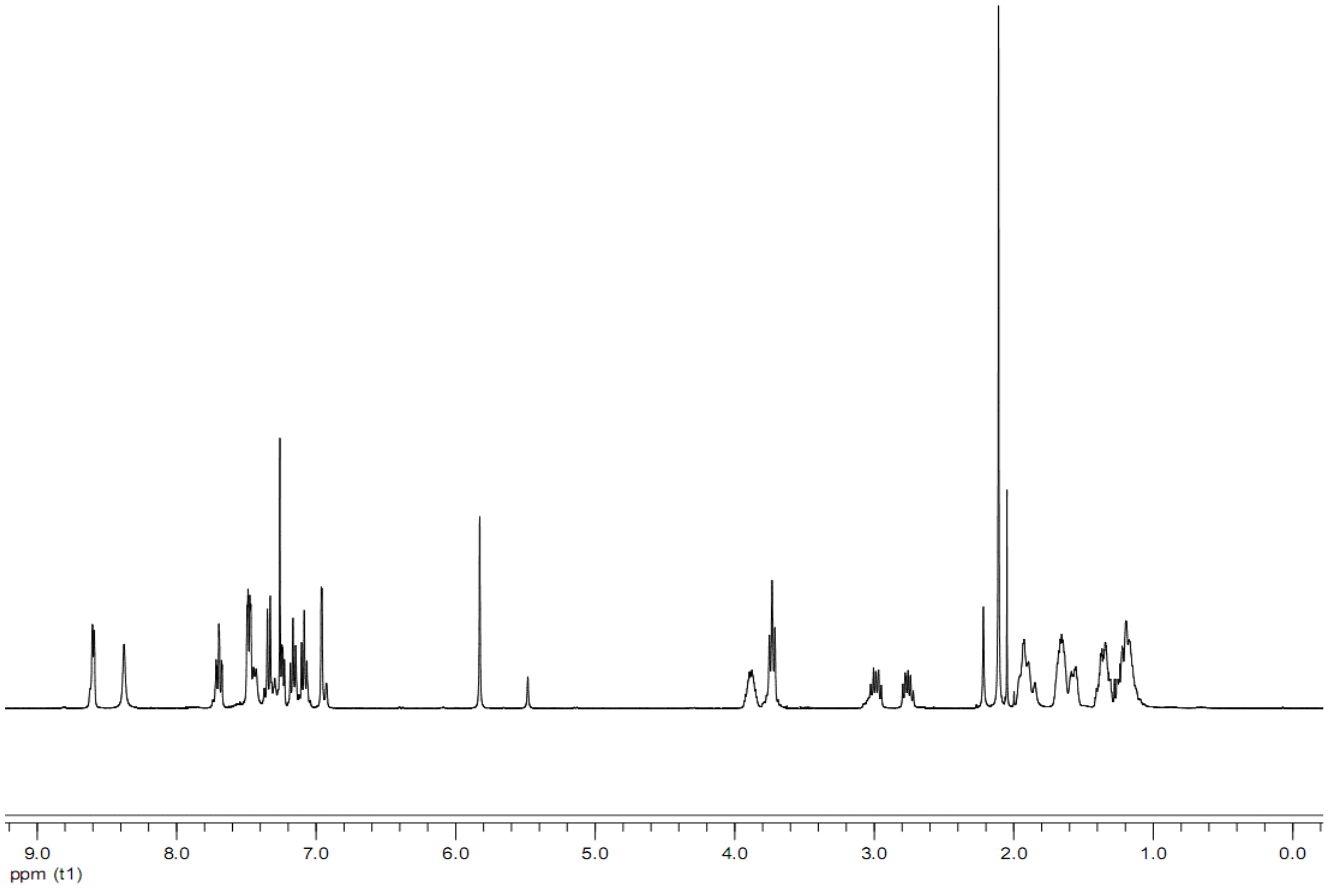
m.p. 178-179 °C.

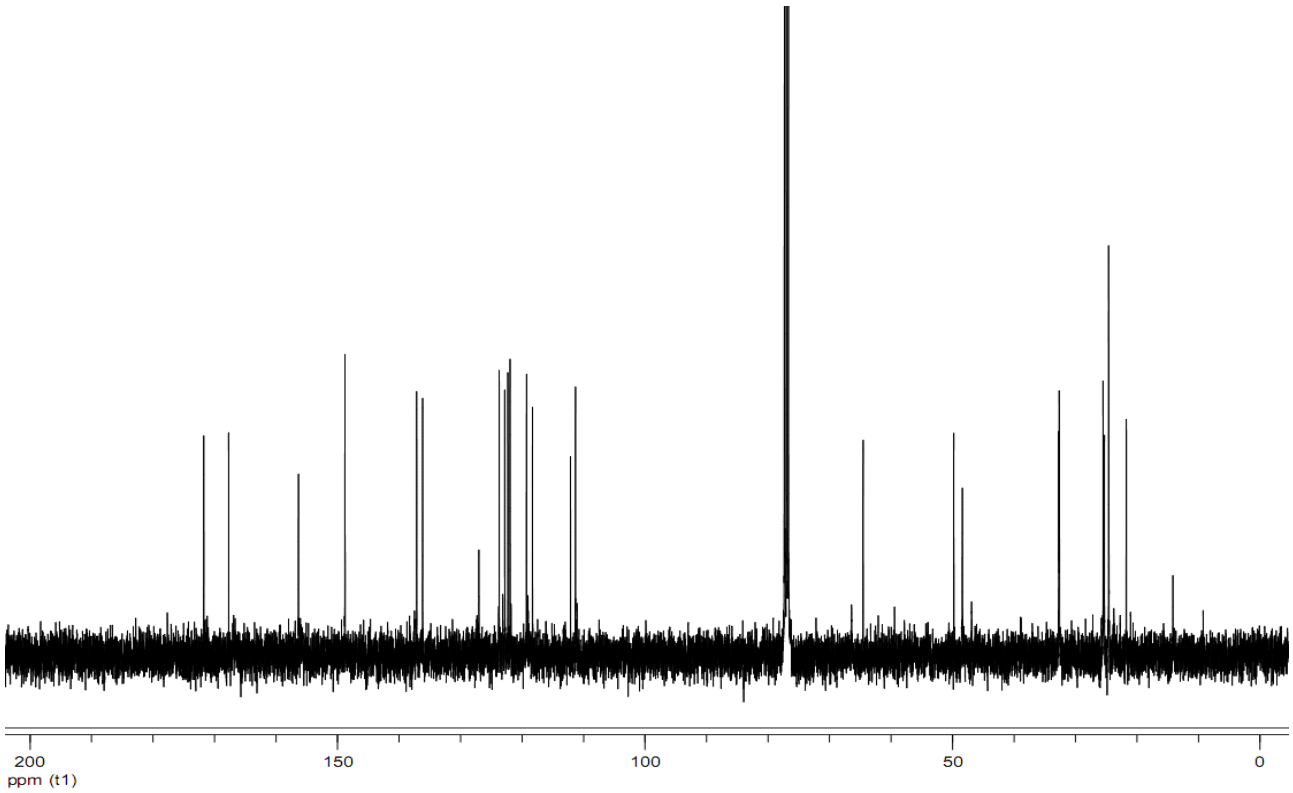
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 4.9 Hz, 1H), 8.38 (br s, 1H), 7.70 (td, *J* = 7.6, 1.7 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 1H) 7.48 (d, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.24 (dd, *J* = 7.6, 4.9 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 2.0 Hz, 1H), 5.82 (s, 1H), 3.93-3.84 (m, 1H), 3.73 (t, *J* = 8.0 Hz, 2H), 2.99 (dt, *J* = 15.2, 7.6 Hz, 1H), 2.76 (dt, *J* = 15.2, 7.6 Hz, 1H), 2.11 (s, 3H), 1.96-1.85 (m, 2H), 1.70-1.63 (m, 2H), 1.60-1.54 (m, 1H), 1.42-1.30 (m, 2H), 1.24-1.13 (m, 3H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.7, 167.7, 156.3, 148.8, 137.1, 136.2, 127.0, 123.7, 122.8, 122.3, 121.9, 119.3, 118.3, 112.1, 111.3, 64.5, 49.8, 48.4, 32.7, 32.6, 25.5, 25.3, 24.6 (2), 21.7.

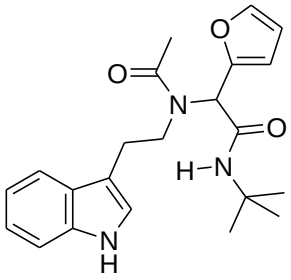
IR (thin film) 3284, 3055, 2932, 2858, 1652, 1629, 1547, 1464 cm^{-1}.

HRMS Calculated for $C_{25}H_{30}N_4O_2$ 418.2369, found 418.2384.





2-{N-[2-(1H-indol-3-yl)ethyl]acetamido}-N-tert-butyl-2-(furan-2-yl)acetamide



$C_{22}H_{27}N_3O_3$
MW = 381.47g/mol

1l

General procedure using tryptamine (320 mg, 2.0 mmol), acetic acid (120 μL, 2.0 mmol), *t*-butyl isocyanide (230 μL, 2.0 mmol) and furfural (170 μL, 2.0 mmol). Reaction time: 24 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (30:70 – 70:30) as eluant gave the desired product (93%) as a white solid.

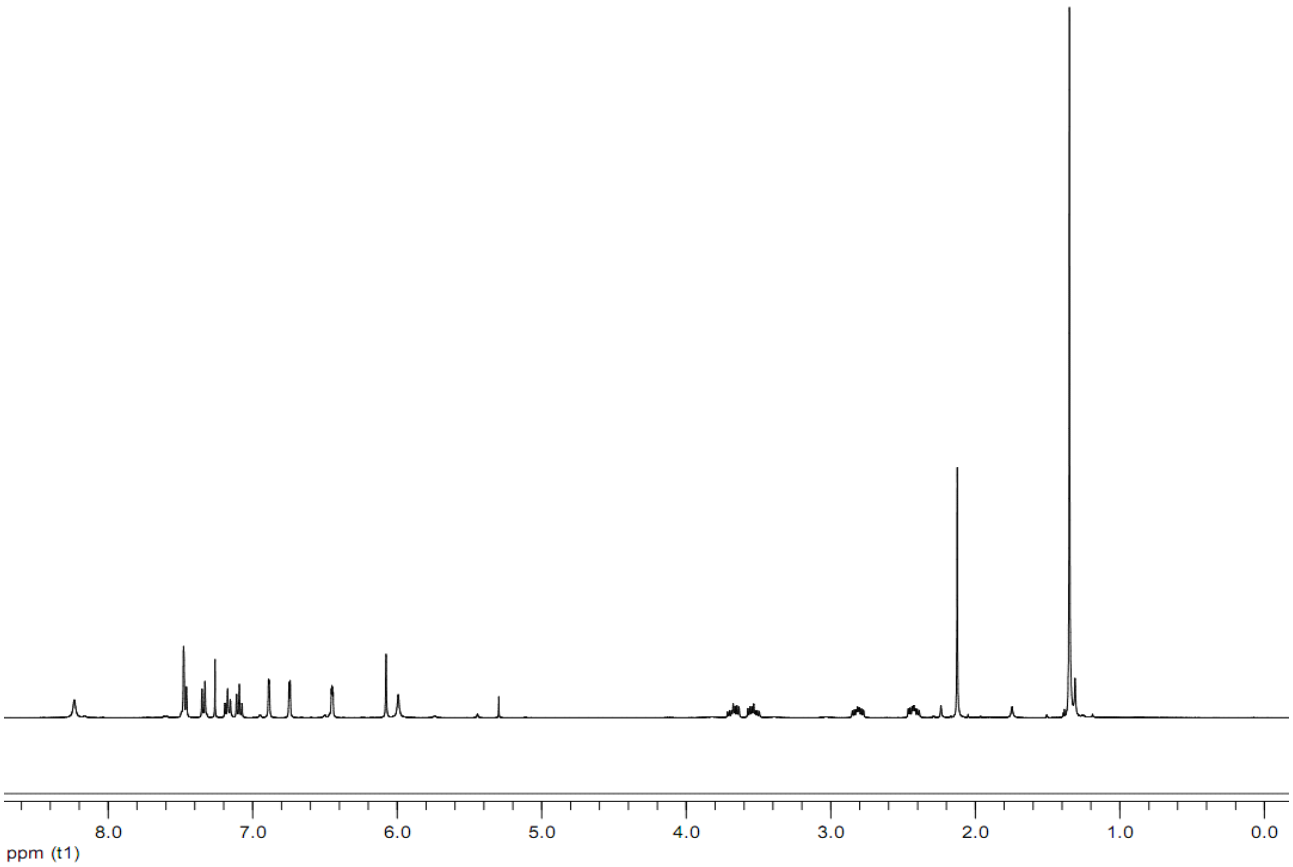
m.p. 192-194 °C.

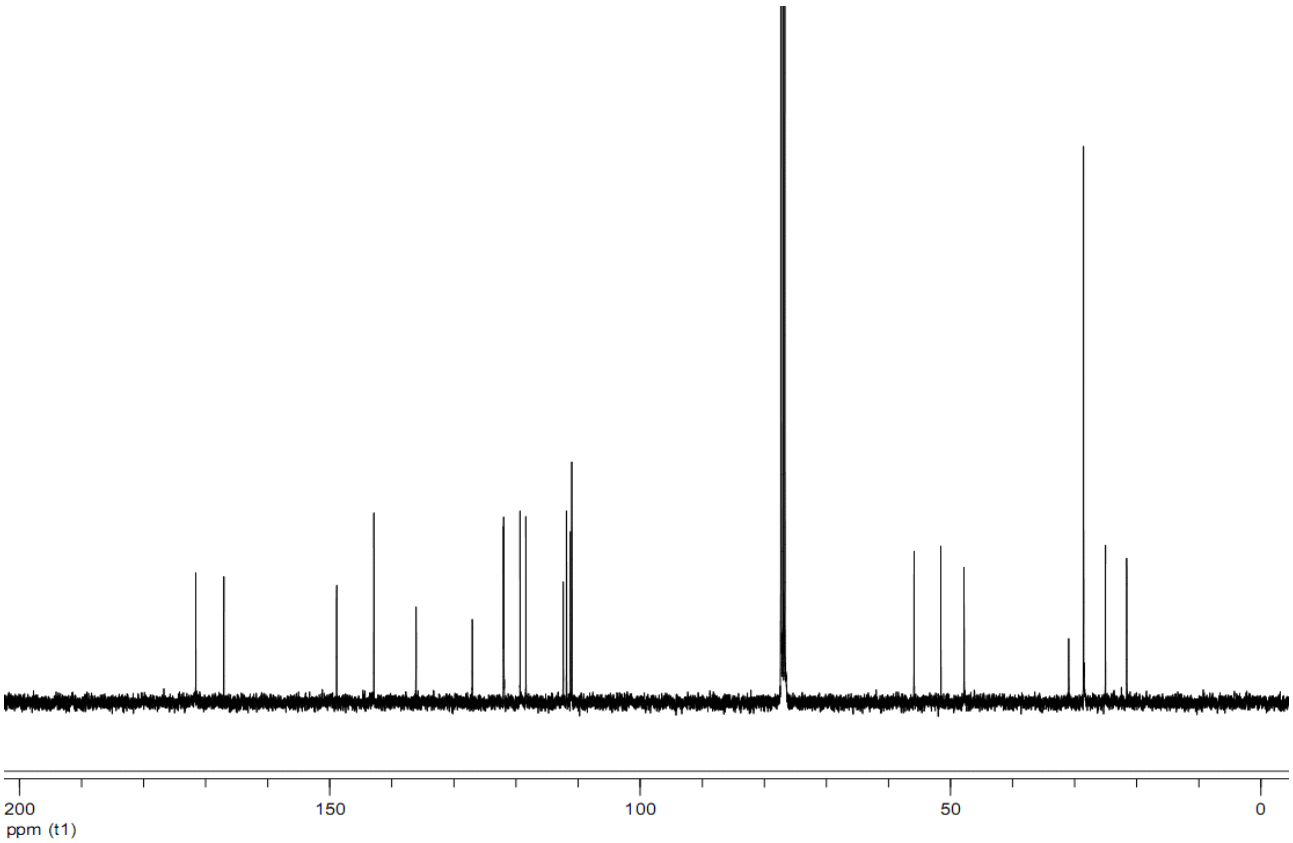
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (br s, 1H), 7.47 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 1H), 7.17 (t, *J* = 7.3 Hz, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 6.89 (d, *J* = 2.0 Hz, 1H), 6.74 (d, *J* = 3.3 Hz, 1H), 6,45 (dd, *J* = 3.3, 2.0 Hz, 1H), 6.08 (s, 1H), 5.99 (br s, 1H), 3.71-3.64 (m, 1H), 3.57-3.49 (m,1H), 2.81 (ddd, *J* = 14.4, 10.6, 5.4 Hz, 1H), 2.43 (ddd, *J* = 14.4, 10.6, 5.4 Hz, 1H), 2.13 (s, 3H), 1.35 (s, 9H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 171.6, 167.1, 148.9, 142.9, 136.1, 127.0, 122.0 (2), 119.3, 118.4, 112.4, 111.8, 111.2, 111.0, 55.9, 51.5, 47.8, 28.6 (3), 25.0, 21.6.

IR (thin film) 3315, 2971, 2929, 1675, 1625, 1541, 1461, 1395 cm^{-1}.

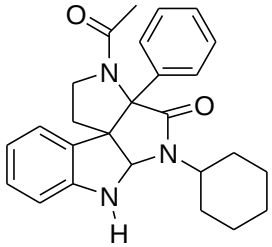
HRMS calcd for $C_{22}H_{27}N_3O_3$ 381.2052, found 381.2071.





General Procedure for the Radical Oxidative Reaction

To a solution of Ugi adduct (1 equiv) in anhydrous THF (0.22 M) was added copper acetate (1 equiv) at 0 °C and then DBU (1 equiv). The mixture was heated to reflux with a potassium hydroxide trap, until completion of the reaction checked with TLC analysis (6-27 h). Then, the reaction mixture was cooled at room temperature and the solvent was removed under reduced pressure. The crude residue was purified by flash column chromatography on silica gel using a gradient of EtOAc in petroleum ether as eluant.



$C_{26}H_{29}N_3O_2$
MW = 415.53 g/mol

2a

General procedure using of Ugi adduct (320 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 µL, 0.43 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (60:40 - 80:20) as eluant gave the desired product (77%) as a white solid.

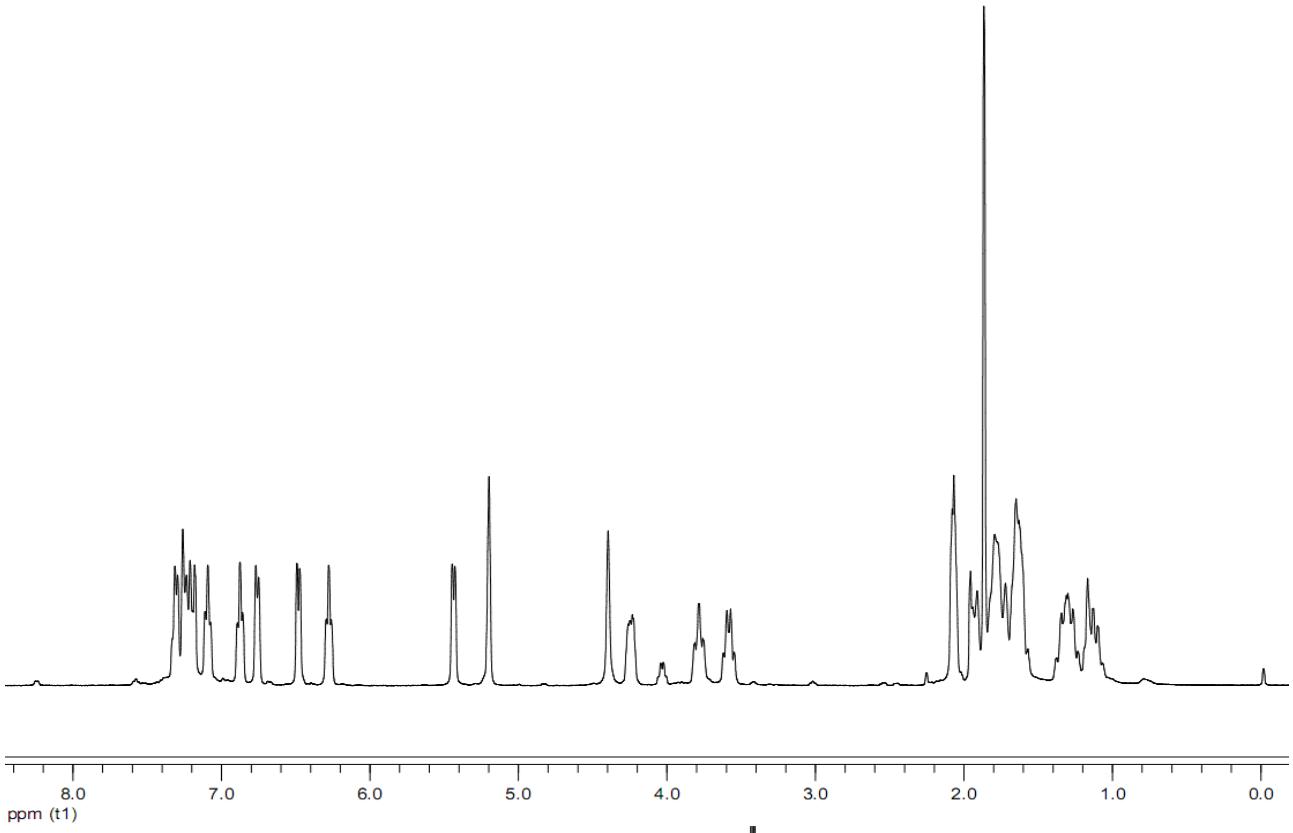
m.p. 268-270 °C.

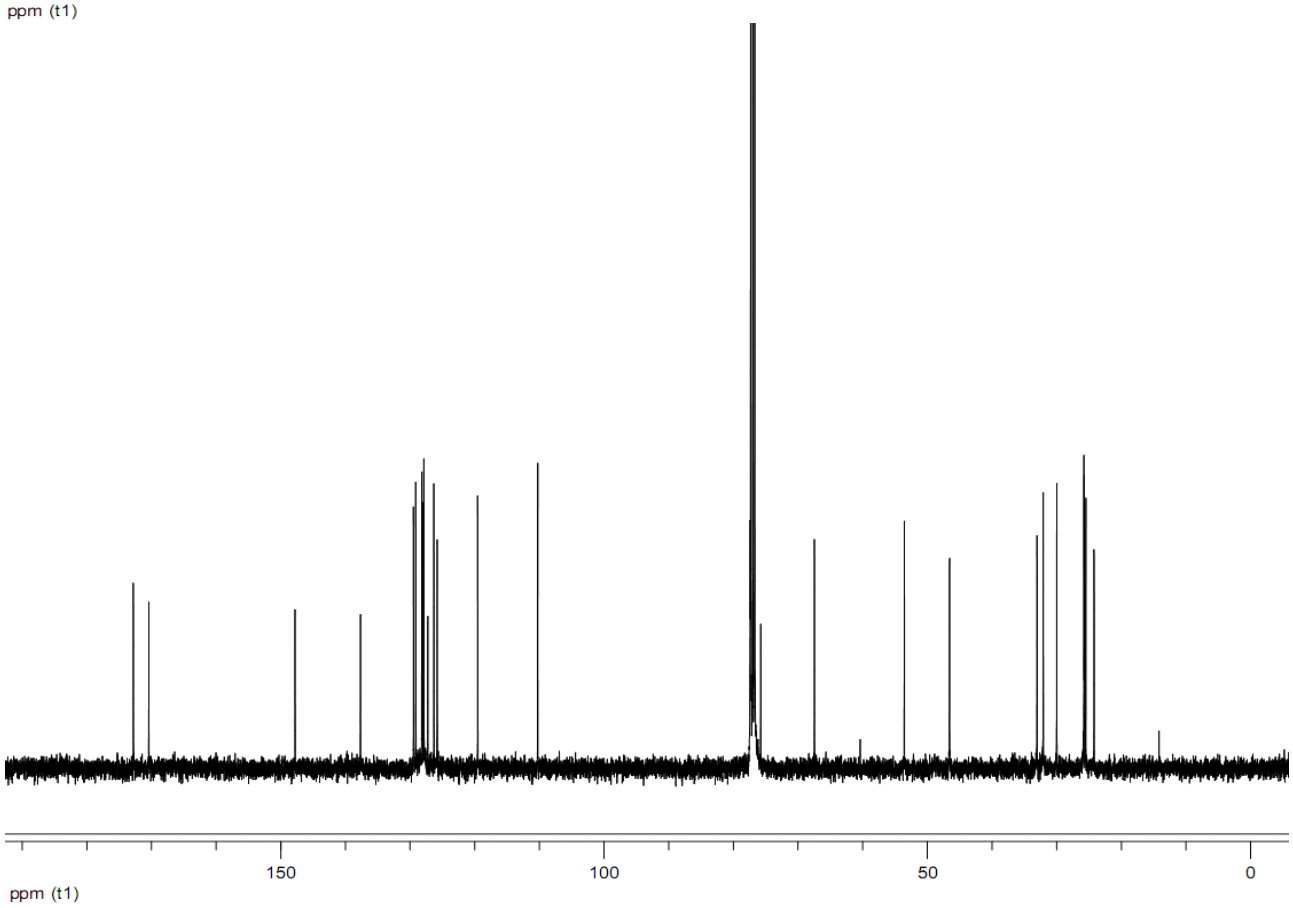
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 6.8 Hz, 1H), 7.28-7.20 (m, 2H), 7.11 (t, *J* = 6.8 Hz, 1H), 6.89 (t, *J* = 6.8 Hz, 1H), 6.77 (d, *J* = 7.5 Hz, 1H), 6.50 (d, *J* = 7.5 Hz, 1H), 6.29 (t, *J* = 6.8 Hz, 1H), 5.45 (d, *J* = 6.8 Hz, 1H), 5.22 (s, 1H), 4.41 (s, 1H), 4.28-4.24 (m, 1H), 3.83-3.77 (m, 1H), 3.64-3.56 (m, 1H), 2.11-2.07 (m, 2H), 1.98-1.92 (m, 1H), 1.88 (s, 3H), 1.84-1.74 (m, 3H), 1.69-1.64 (m, 3H), 1.39-1.25 (m, 2H), 1.21-1.09 (m, 1H).

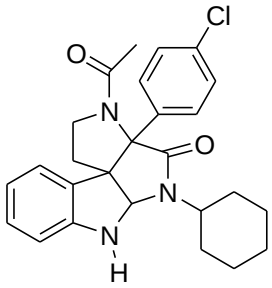
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.8, 170.4, 147.8, 137.7, 129.5, 129.1, 128.2, 128.1, 127.7, 127.3, 126.3, 125.8, 119.5, 110.3, 77.4, 75.8, 67.5, 53.5, 46.6, 33.0, 32.1, 30.0, 25.8, 25.7, 25.5, 24.2.

IR (thin film) 3347, 3053, 2933, 1676, 1622, 1487, 1382, 1259, 1179, 1058 cm^{-1}.

HRMS Calculated for $C_{26}H_{29}N_3O_2$ 415.2260, found 415.2244.







$C_{26}H_{28}ClN_3O_2$
MW = 449.97 g/mol

2c

General procedure using of Ugi adduct (195 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 – 70:30) as eluant gave the desired product (64%) as a white solid.

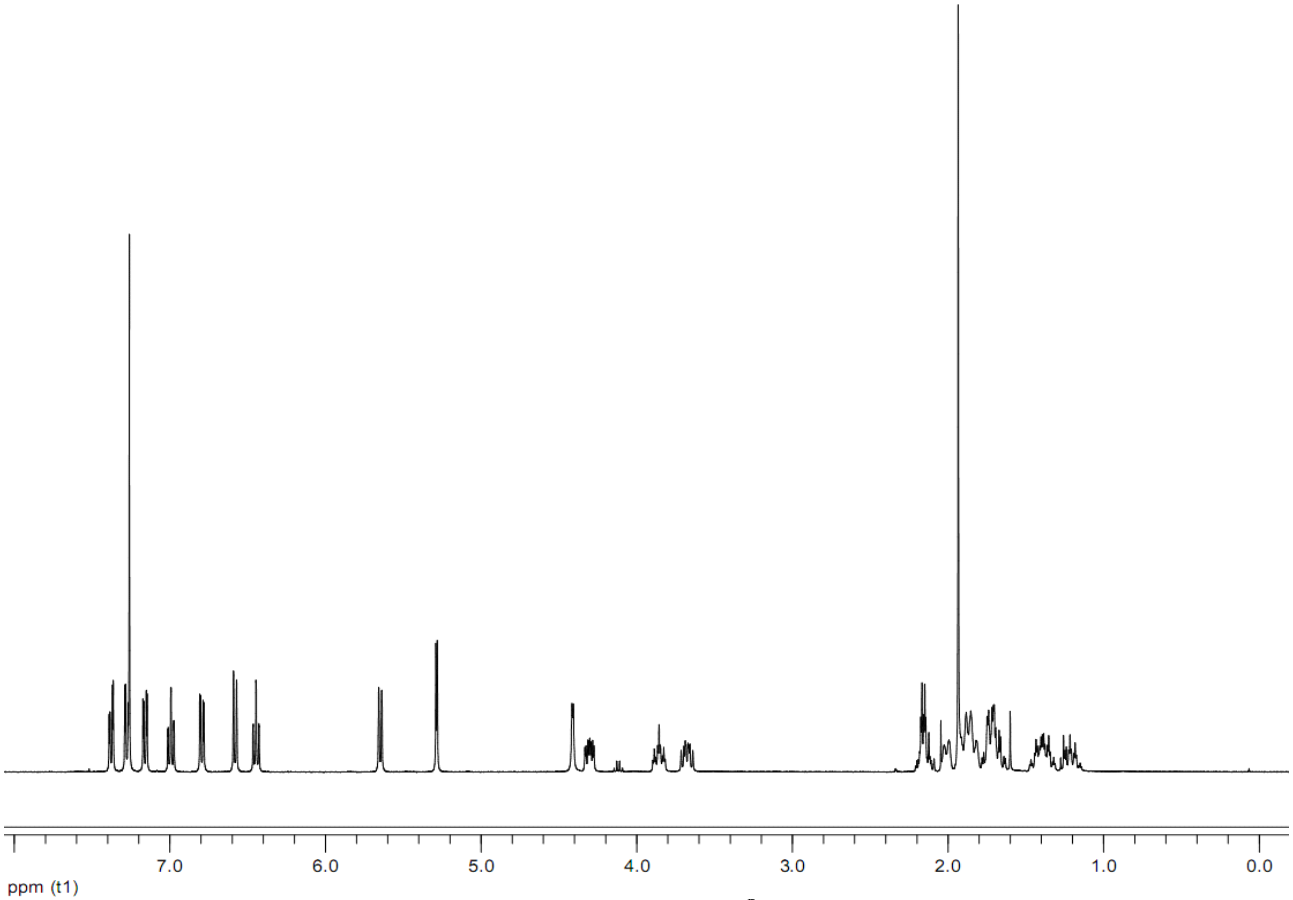
m.p. 298 - 290 °C.

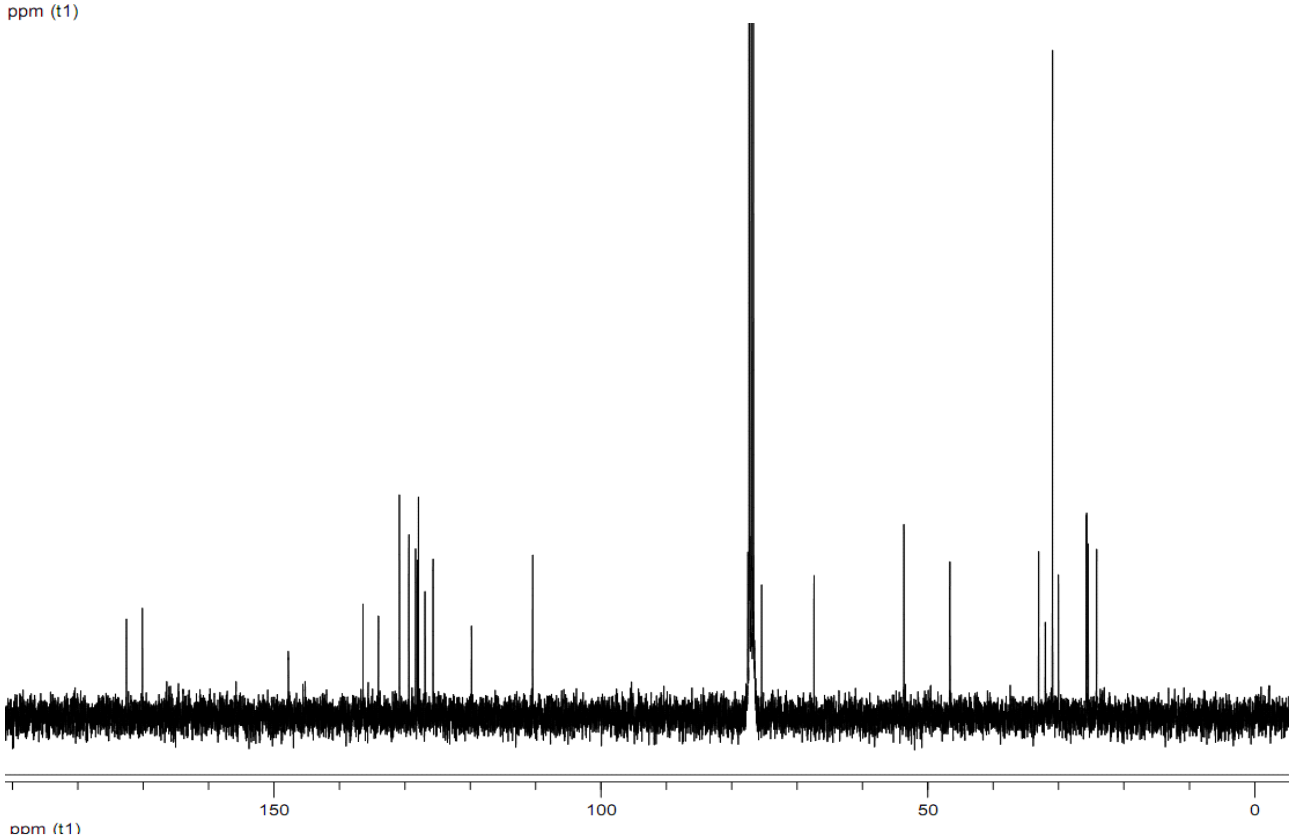
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.28 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.16 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.99 (td, *J* = 7.7, 1.0 Hz, 1H), 6.79 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.58 (d, *J* = 7.7 Hz, 1H), 6.45 (td, *J* = 7.7, 1.0 Hz, 1H), 6.65 (d, *J* = 7.7 Hz, 1H), 5.29 (d, *J* = 3.4 Hz, 1H), 4.41 (br d, *J* = 3.4 Hz, 1H), 4.30 (ddd, *J* = 12.3, 7.4, 3.3 Hz, 1H), 3.86 (tt, *J* = 12.3, 3.3 Hz, 1H), 3.68 (ddd, *J* = 12.3, 7.4, 3.3 Hz, 1H), 2.20-2.14 (m, 2H), 2.03-1.99 (m, 1H), 1.93 (s, 3H), 1.91-1.88 (m, 3H), 1.78-1.63 (m, 3H), 1.48-1.31 (m, 2H), 1.27-1.14 (m, 1H).

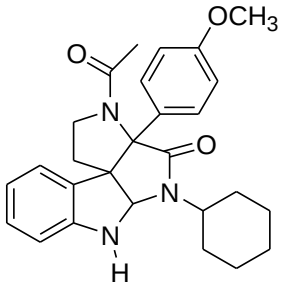
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.6, 170.1, 147.8, 136.4, 134.0, 130.8, 129.4, 128.3, 128.1, 127.9, 126.9, 125.7, 119.8, 110.4, 77.5, 75.4, 67.4, 53.7, 46.6, 33.0, 32.0, 30.0, 25.8, 25.7, 25.5, 24.2.

IR (thin film) 3320, 3056, 2934, 2856, 1676, 1624, 1545, 1492, 1458, 1354 cm^{-1}.

HRMS Calculated for $C_{26}H_{28}ClN_3O_2$ 449.1870, found 449.1870.







$C_{27}H_{31}N_3O_3$
MW = 445.55 g/mol

2d

General procedure using of Ugi adduct (193 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 7.5 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 70:30) as eluant gave the desired product (53%) as a white solid.

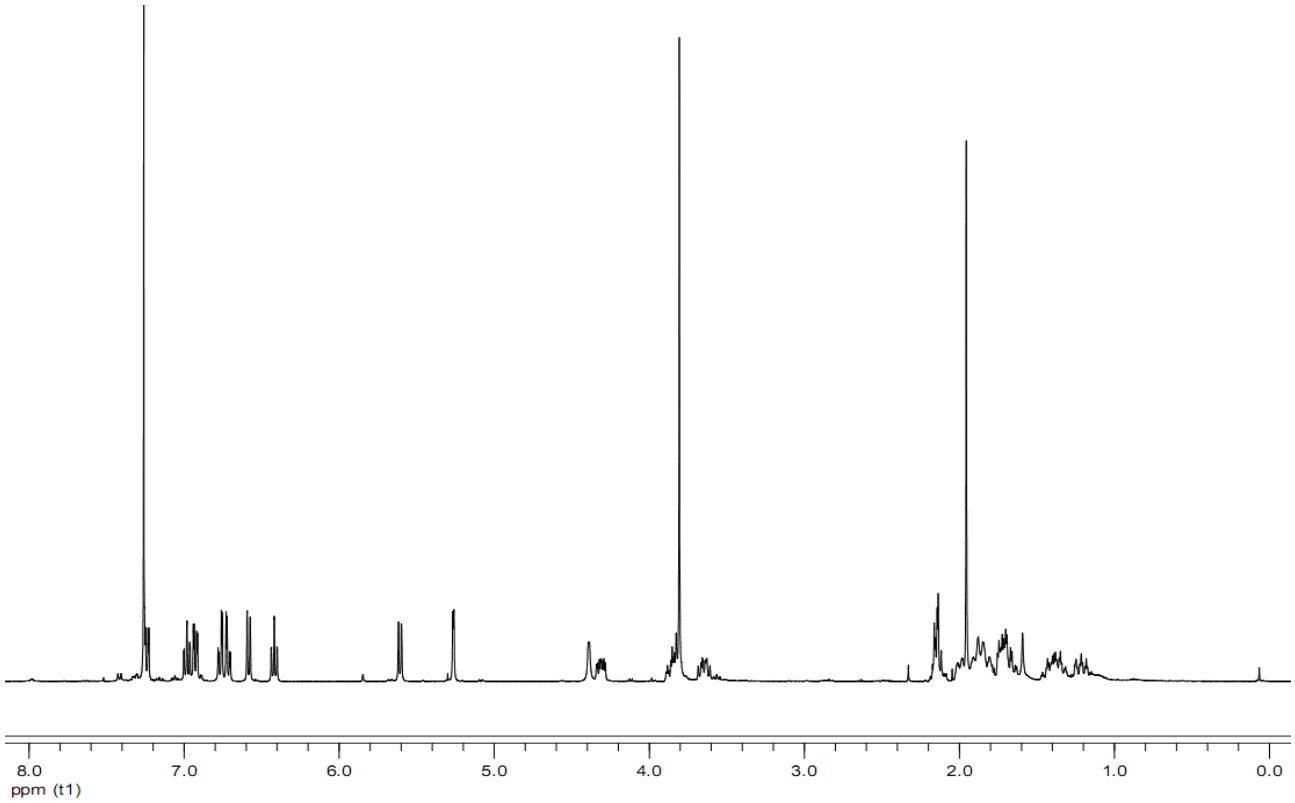
m.p. 231-232 °C.

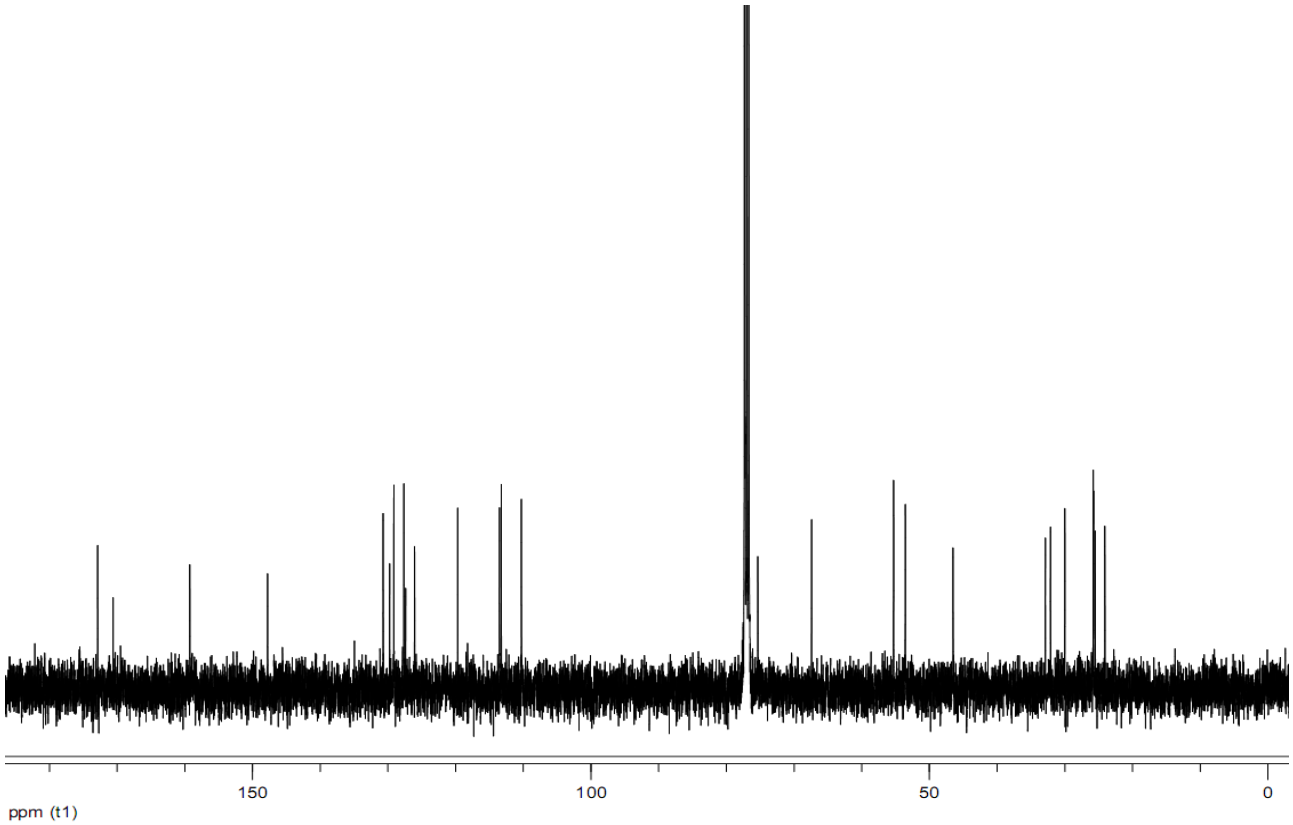
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.98 (td, *J* = 7.5, 1.0 Hz, 1H), 6.93 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.77 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.71 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.58 (d, *J* = 7.5 Hz, 1H), 6.42 (td, *J* = 7.5, 1.0 Hz, 1H), 5.61 (d, *J* = 7.5 Hz, 1H), 5.26 (d, *J* = 2.7 Hz, 1H), 4.39 (br d, *J* = 2.7 Hz, 1H), 4.31 (ddd, *J* = 10.2, 7.4, 3.2 Hz, 1H), 3.89-3.83 (m, 1H), 3.81 (s, 3H), 3.65 (ddd, *J* = 12.1, 10.2, 7.4 Hz, 1H), 2.17-2.12 (m, 2H), 2.01-1.98 (m, 1H), 1.98 (s, 3H), 1.92-1.78 (m, 3H), 1.75-1.69 (m, 3H), 1.47-1.31 (m, 2H), 1.26-1.14 (m, 1H).

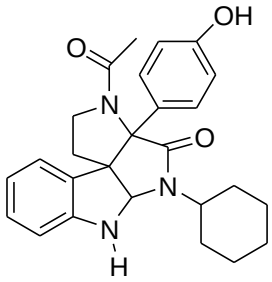
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.9, 170.6, 159.2, 147.8, 130.7, 129.7, 129.1, 127.6, 127.4, 126.1 119.7, 113.5, 113.2, 110.3, 77.2, 75.3, 67.4, 55.3, 53.6, 46.5, 32.9, 32.1, 30.0, 25.8, 25.7, 25.5, 24.1.

IR (thin film) 3320, 3051, 2932, 2855, 1683, 1610, 1487, 1472, 1450, 1340, 1250, 1034 cm^{-1}.

HRMS Calculated for $C_{27}H_{31}N_3O_3$ 445.2365, found 445.2383.







$C_{26}H_{29}N_3O_3$
MW = 431.53 g/mol

2e

General procedure using of Ugi adduct (187 mg, 0.432 mmol), copper acetate (86 mg, 0.432 mmol) and DBU (65 μL, 0.432 mmol). Reaction time: 7h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:70 - 70:30) as eluant gave the desired product (77%) as a white solid.

m.p. 312-313 °C.

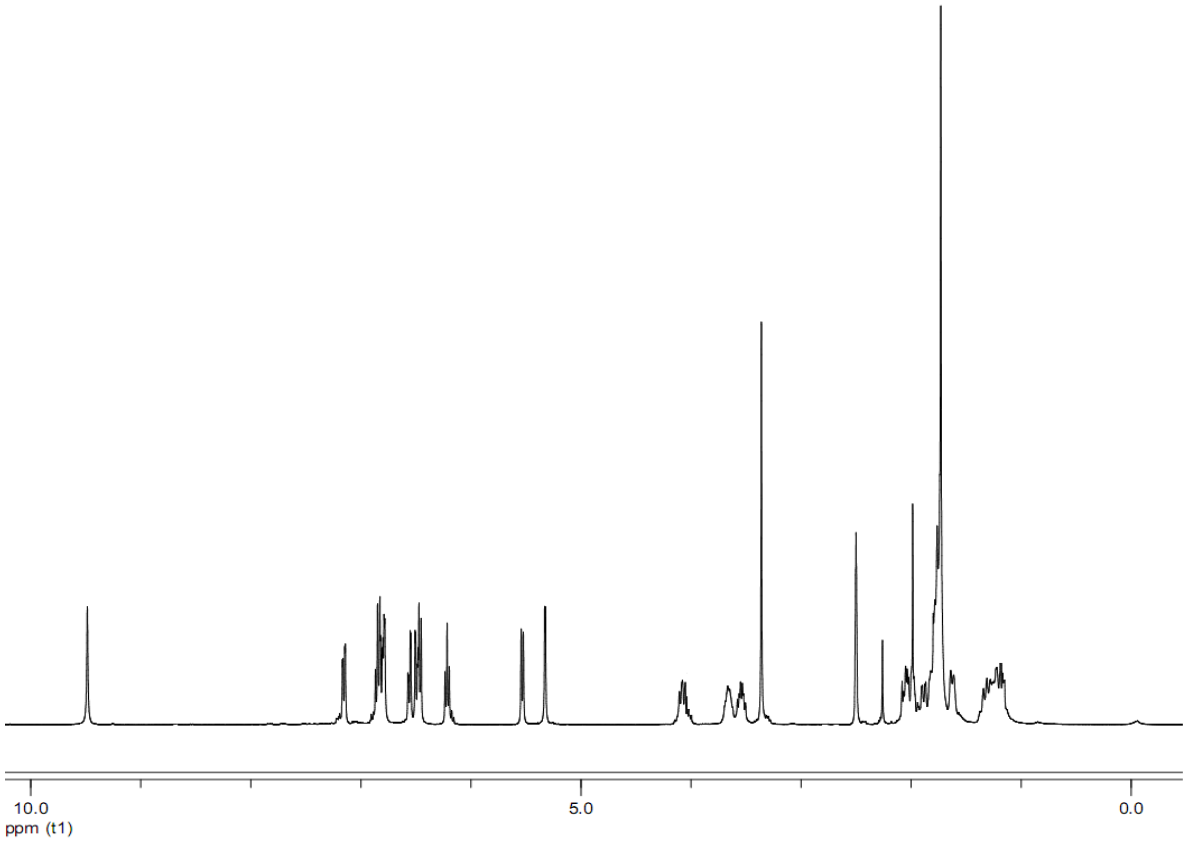
(mm133/10)

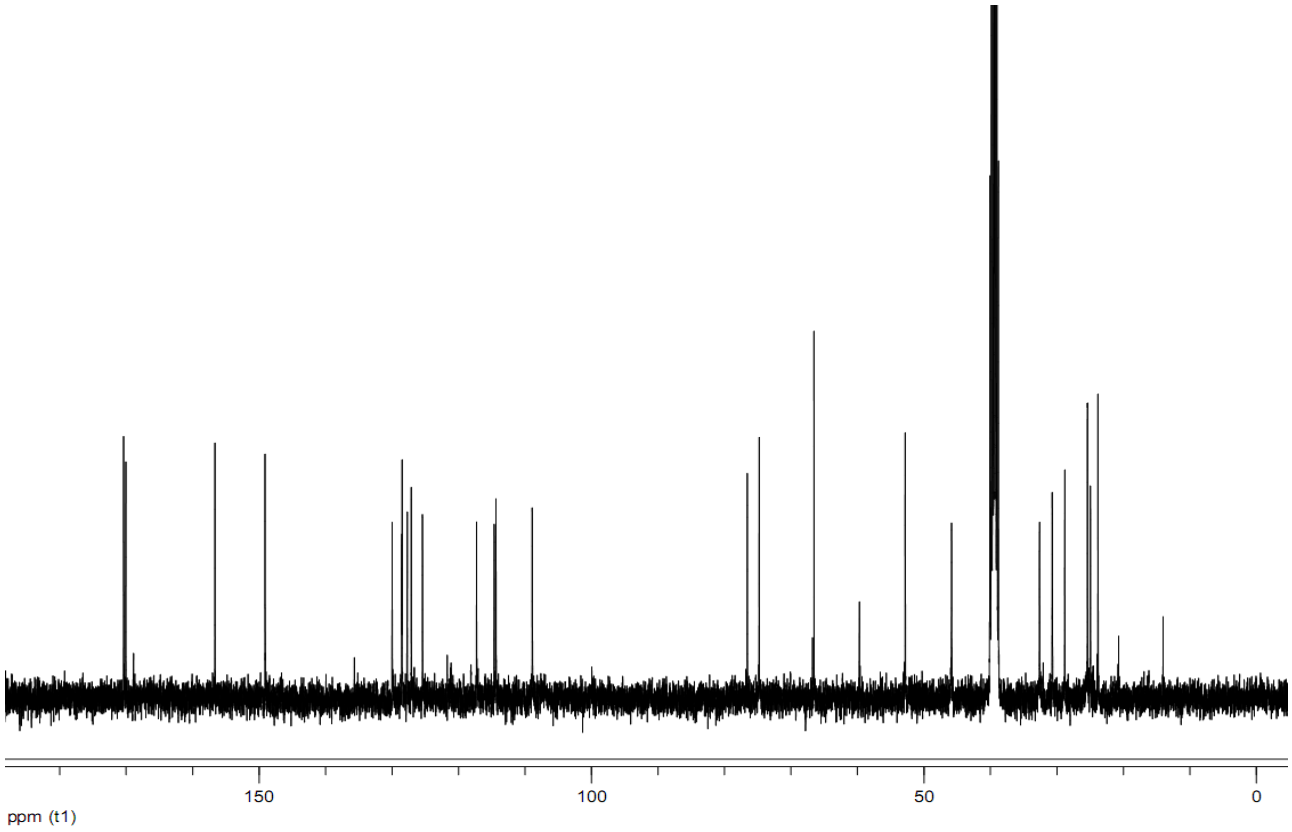
^{1}H NMR (400 MHz, DMSO) δ 9.49 (s, 1H), 7.15 (dd, *J* = 8.5, 2.4 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.82 (d, *J* = 2.4 Hz, 1H), 6.79 (dd, *J* = 6.4, 2.4 Hz, 1H), 6.56 (dd, *J* = 8.5, 2.4 Hz, 1H), 6.49 (dd, *J* = 8.5, 2.4 Hz, 1H), 6.46 (d, *J* = 7.6 Hz, 1H), 6.22 (t, *J* = 7.6 Hz, 1H), 5.53 (d, *J* = 7.6 Hz, 1H), 5.33 (d, *J* = 3.0 Hz, 1H), 4.11-4.05 (m, 1H), 3.70-3.62 (m, 1H), 3.54 (td, *J* = 16.1, 11.4, 6.5 Hz, 1H), 2.08-1.94 (m, 2H), 1.94-1.87 (m, 1H), 1.80-1.73 (m, 6H), 1.73 (s, 3H), 1.62 (m, 1H), 1.37-1.22 (m, 2H).

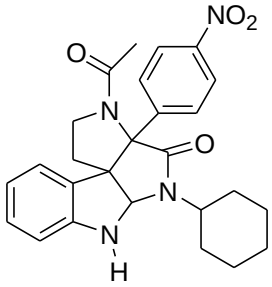
^{13}C NMR (100.6 MHz, DMSO) δ 170.4, 170.1, 156.6, 149.1, 130.0, 128.5, 128.5, 127.7, 127.1, 125.4, 117.3, 114.6, 114.4, 108.9, 76.6, 74.8, 66.5, 52.8, 45.8, 32.6, 30.7, 30.6, 28.8, 25.4, 25.0, 23.8.

IR (thin film) 3300, 2932, 2857, 1656, 1613, 1461, 1394, 1343, 1238 cm^{-1}.

HRMS Calculated for $C_{26}H_{29}N_3O_3$ 431.2209, found 431.2191.







$C_{26}H_{28}N_4O_4$
MWt = 460.53 g/mol

2f

General procedure using of Ugi adduct (200 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 4 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 – 70:30) as eluant gave the desired product (76%) as a yellow solid.

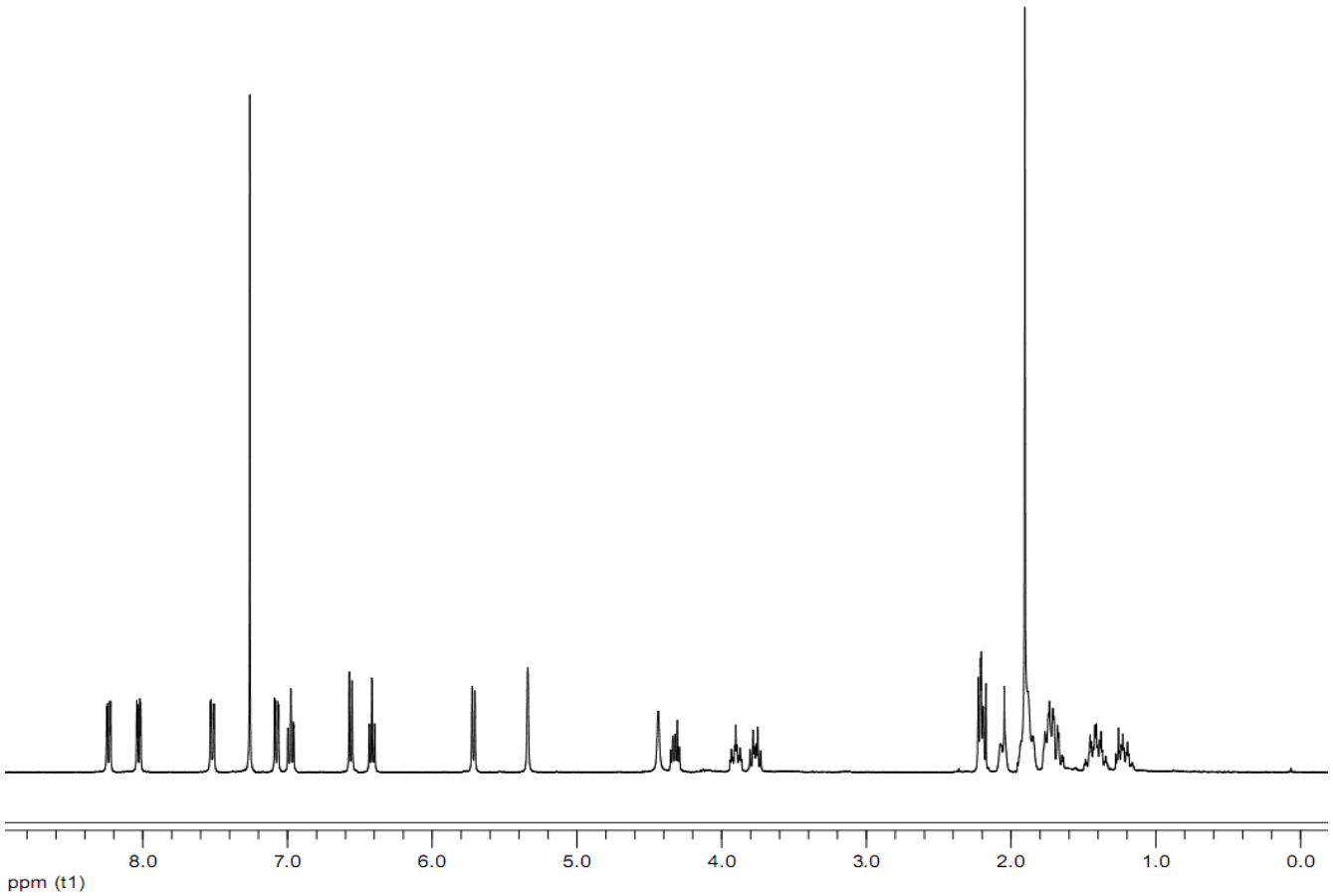
m.p. 291-292 °C.

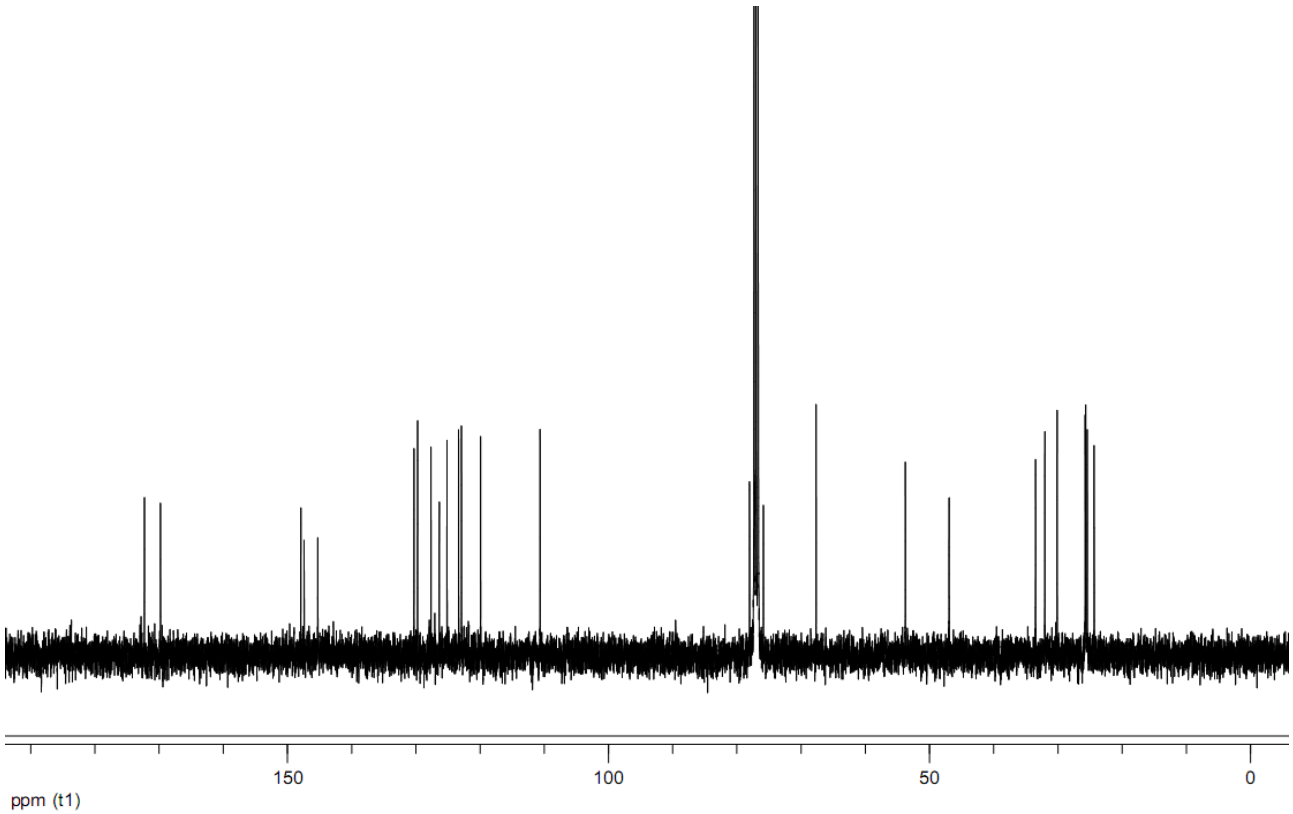
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 8.6, 2.2 Hz, 1H), 8.03 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.52 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.08 (dd, *J* = 8.6, 2.2 Hz, 1H), 6.98 (td, *J* = 7.6, 0.8 Hz, 1H), 6.56 (d, *J* = 7.6 Hz, 1H), 6.42 (td, *J* = 7.6, 0.8 Hz, 1H), 5.72 (d, *J* = 7.6 Hz, 1H), 5.34 (s, 1H), 4.44 (br s, 1H), 4.35-4.29 (m, 1H), 3.92 (tt, *J* = 12.3, 3.5 Hz, 1H), 3.77 (dt, *J* = 12.3, 8.5 Hz, 1H), 2.23-2.19 (m, 2H), 2.07-2.03 (m, 1H), 1.93-1.85 (m, 3H), 1.91 (s, 3H), 1.77-1.64 (m, 3H), 1.49-1.34 (m, 2H), 1.28-1.16 (m, 1H).

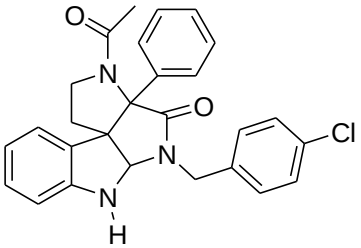
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.3, 169.8, 147.9, 147.4, 145.3, 130.3, 129.7, 127.6, 126.4, 125.1, 123.3, 122.9, 119.9, 110.7, 78.0, 75.9, 67.7, 53.8, 46.9, 33.5, 32.0, 30.1, 25.7 (2), 25.4, 24.3.

IR (thin film) 3322, 2938, 2858, 1684, 1632, 1519, 1488, 1384, 1345 cm^{-1}.

HRMS Calculated for $C_{26}H_{28}N_4O_4$ 460.2111, found 460.2115.







$C_{27}H_{24}ClN_3O_2$
MW = 457.95 g/mol

2g

General procedure using of Ugi adduct (199 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 6 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 - 70:30) as eluant gave the desired product (66%) as a white solid.

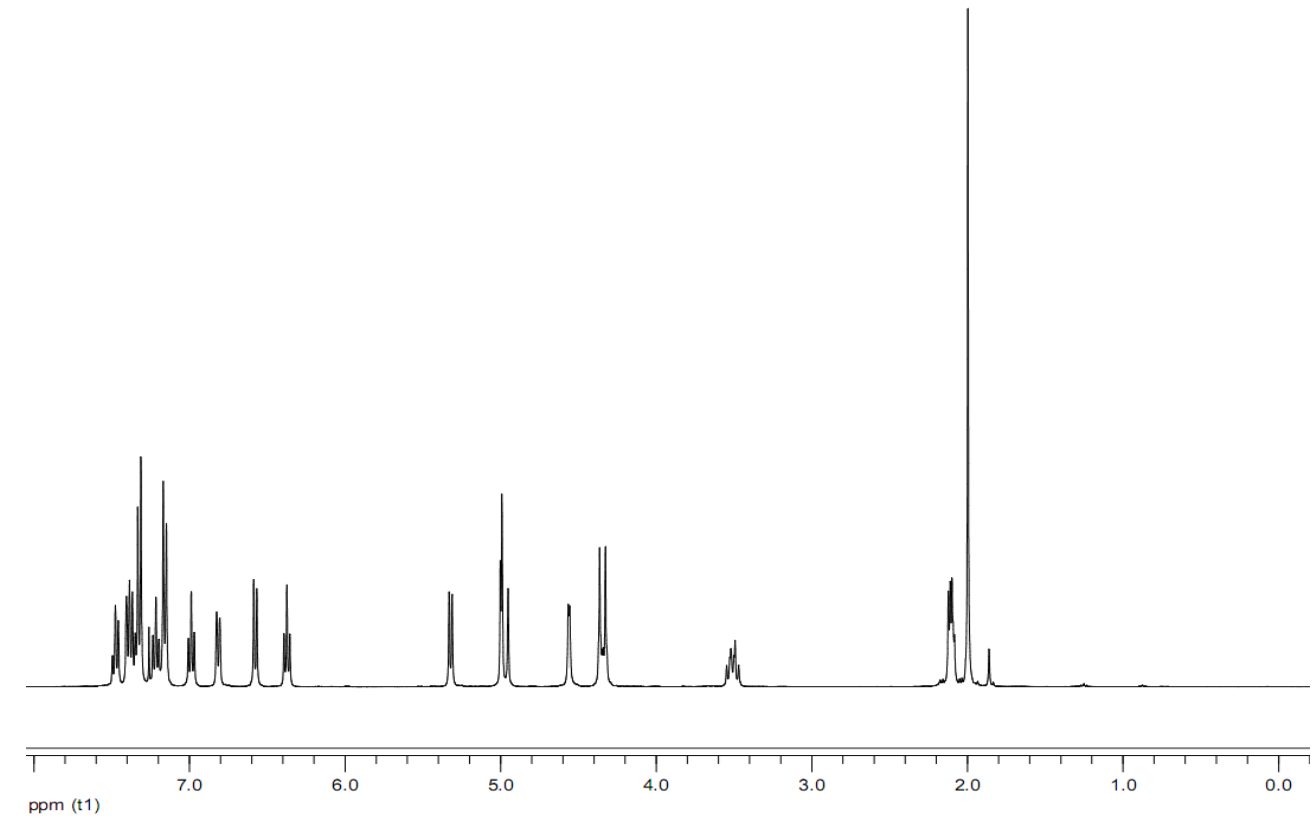
m.p. 198-200 °C.

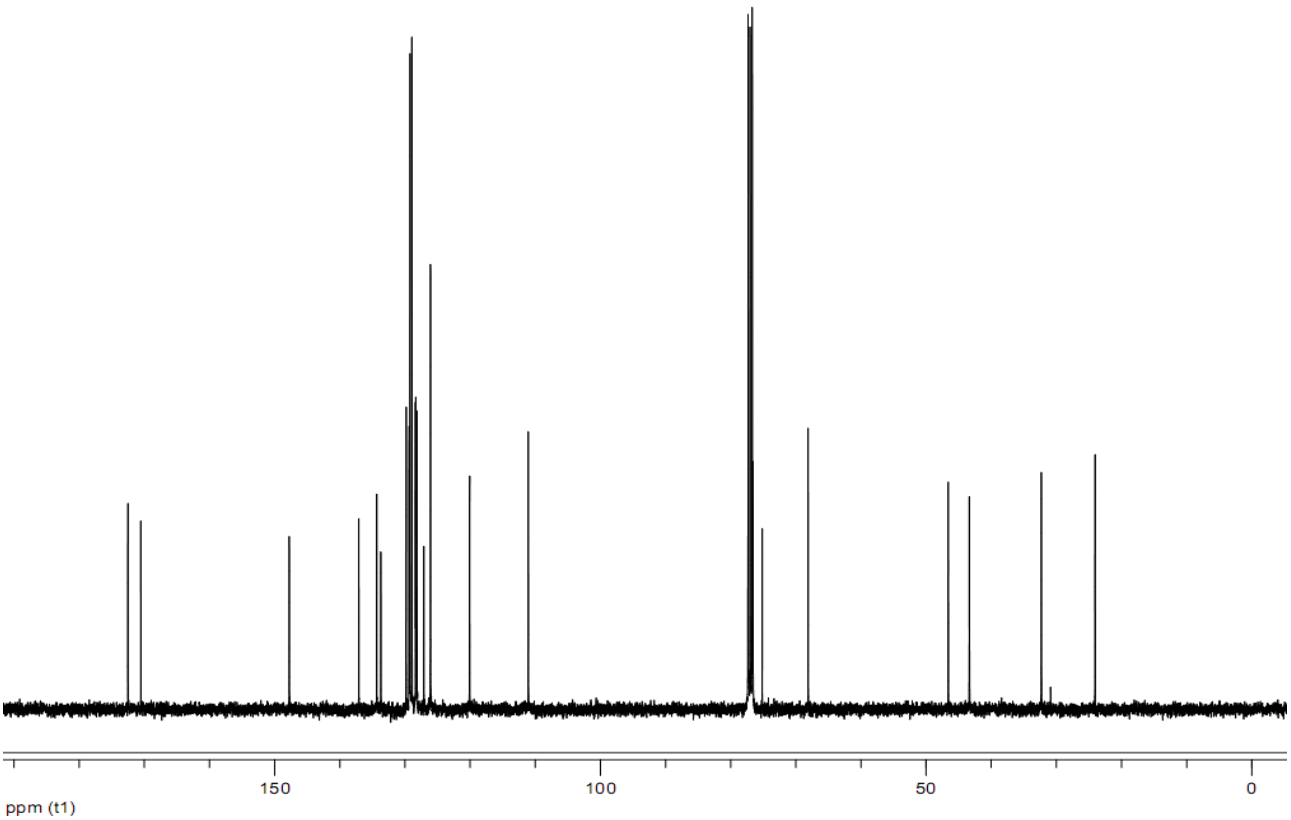
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (t, *J* = 7.7 Hz, 1H), 7.39 (d, J = 7.7 Hz, 1H), 7.36 (d, J = 7.7 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.21 (t, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 8.2 Hz, 2H), 6.99 (t, *J* = 7.7 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 6.58 (d, *J* = 7.7 Hz, 1H), 6.37 (t, *J* = 7.7 Hz, 1H), 5.32 (d, *J* = 7.7 Hz, 1H), 5.00 (d, *J* = 3.7 Hz, 1H), 4.95 (d, *J* = 15.5 Hz, 1H), 4.56 (d, *J* = 3.7 Hz, 1H), 4.37-4.32 (m, 2H), 3.51 (dt, *J* = 11.5, 8.6 Hz, 1H), 2.12-2.08 (m, 2H), 2.00 (s, 3H).

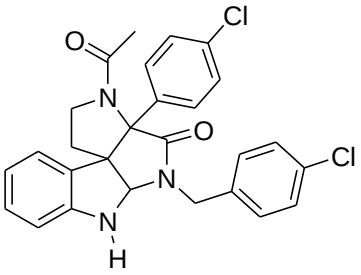
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.5, 170.5, 147.7, 137.1, 134.3, 133.7, 129.8, 129.3, 129.2 (2), 128.9 (2), 128.4, 128.3, 128.1, 127.1, 126.1 (2), 120.1, 111.1, 76.6, 75.1, 68.1, 46.6, 43.3, 32.3, 24.0.

IR (thin film) 3337, 2875, 1699, 1652, 1492, 1390, 1343 cm^{-1}.

HRMS Calculated for $C_{27}H_{24}ClN_3O_2$ 457.1557, found 457.1558.







$C_{27}H_{23}Cl_2N_3O_2$
MW = 492.40 g/mol

2h

General procedure using of Ugi adduct (214 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 4 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 70:30) as eluant gave the desired product (75%) as a white solid.

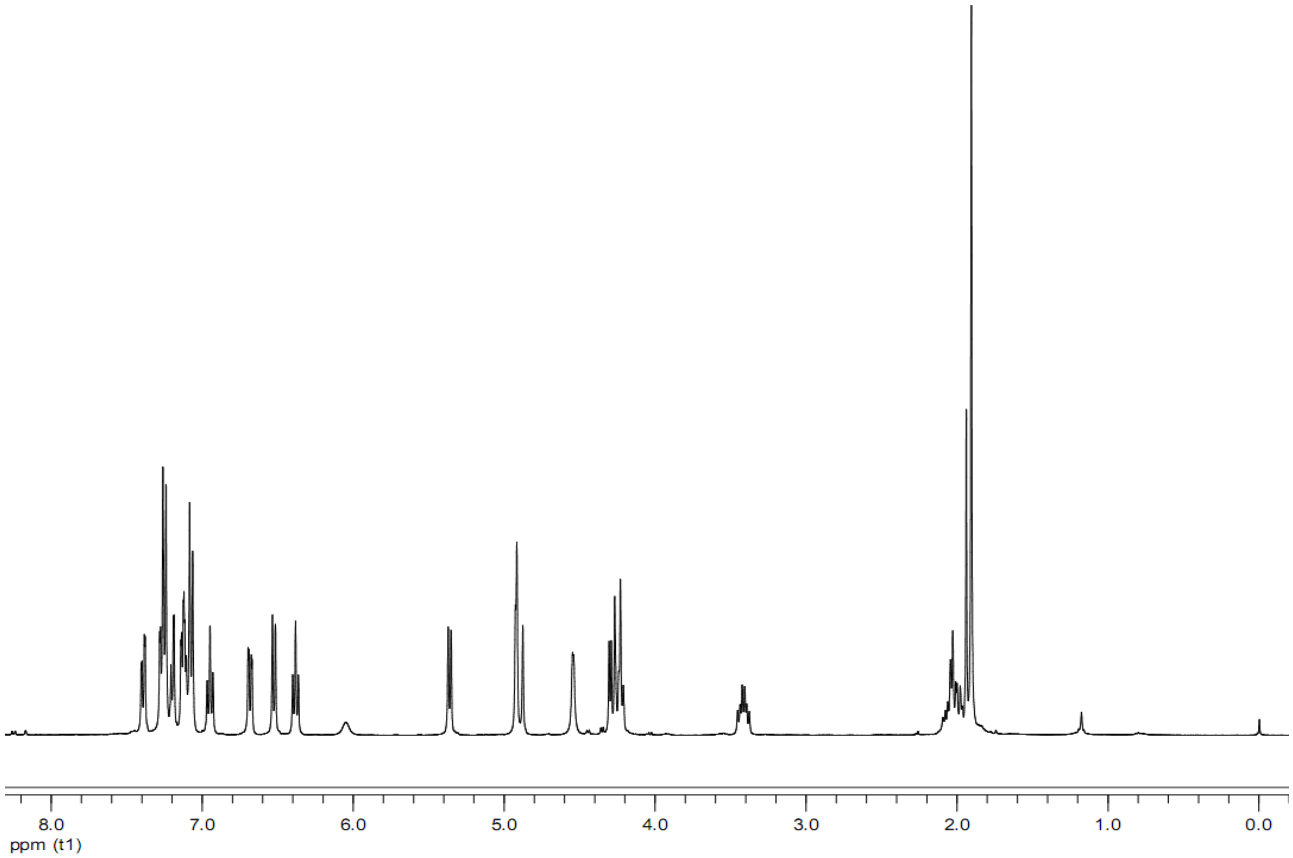
m.p. 231-233 °C.

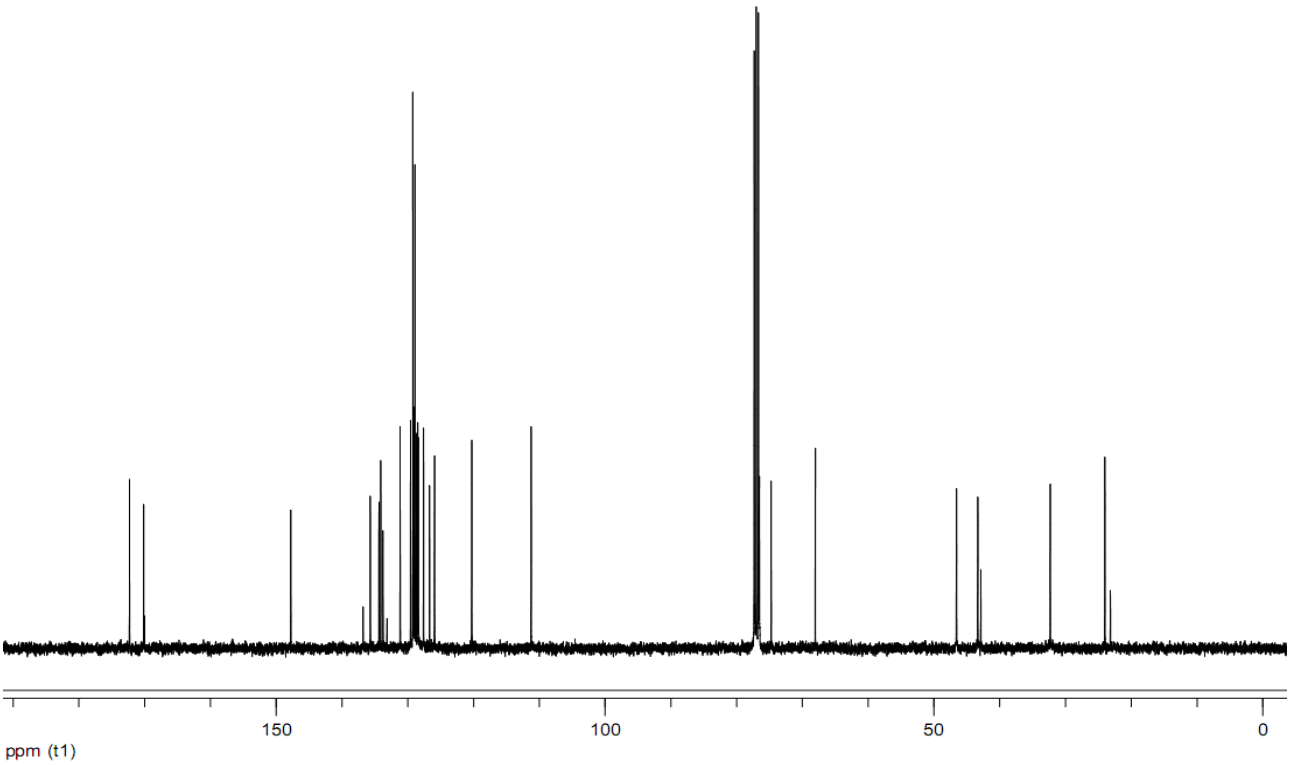
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.28-7.25 (m, 3H), 7.14-7.11 (m, 1H), 7.07 (d, *J* = 8.3 Hz, 2H), 6.95 (t, *J* = 7.8, 1H), 6.68 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.53 (d, *J* = 7.8 Hz, 1H), 6.38 (t, *J* = 7.8 Hz, 1H), 5.36 (d, *J* = 7.8 Hz, 1H), 4.92 (d, *J* = 3.0, 1H), 4.90 (d, *J* = 15.9, 1H), 4.55 (br d, *J* = 3.0 Hz, 1H), 4.31-4.21 (m, 2H); 3.42 (td, *J* = 12.2, 6.8 Hz, 1H), 2.09-1.96 (m, 2H), 1.91 (s, 3H).

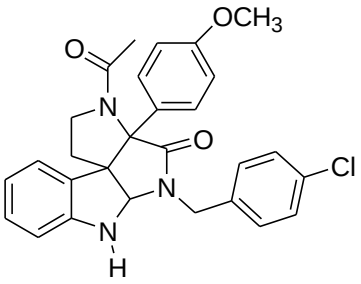
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.3, 170.1, 147.8, 135.7, 134.4, 134.1, 133.7, 131.2, 129.6, 129.2 (2), 128.9 (2), 128.5, 128.4, 127.6, 126.7, 125.9, 120.3, 111.2, 76.5, 74.7, 68.0, 46.6, 43.3, 32.3, 24.0.

IR (thin film) 3319, 3052, 2928, 1696, 1628, 1065 cm^{-1}.

HRMS Calculated for $C_{27}H_{23}Cl_2N_3O_2$ 491.1167, found 491.1164.







$C_{28}H_{26}ClN_3O_3$
MW = 487.98 g/mol

2i

General procedure using of Ugi adduct (212 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 5 h. Purification by flash chromatography using a gradient of etyl acetate in petroleum ether (50:50 - 70:30) as eluant gave the desired product (59%) as a white solid.

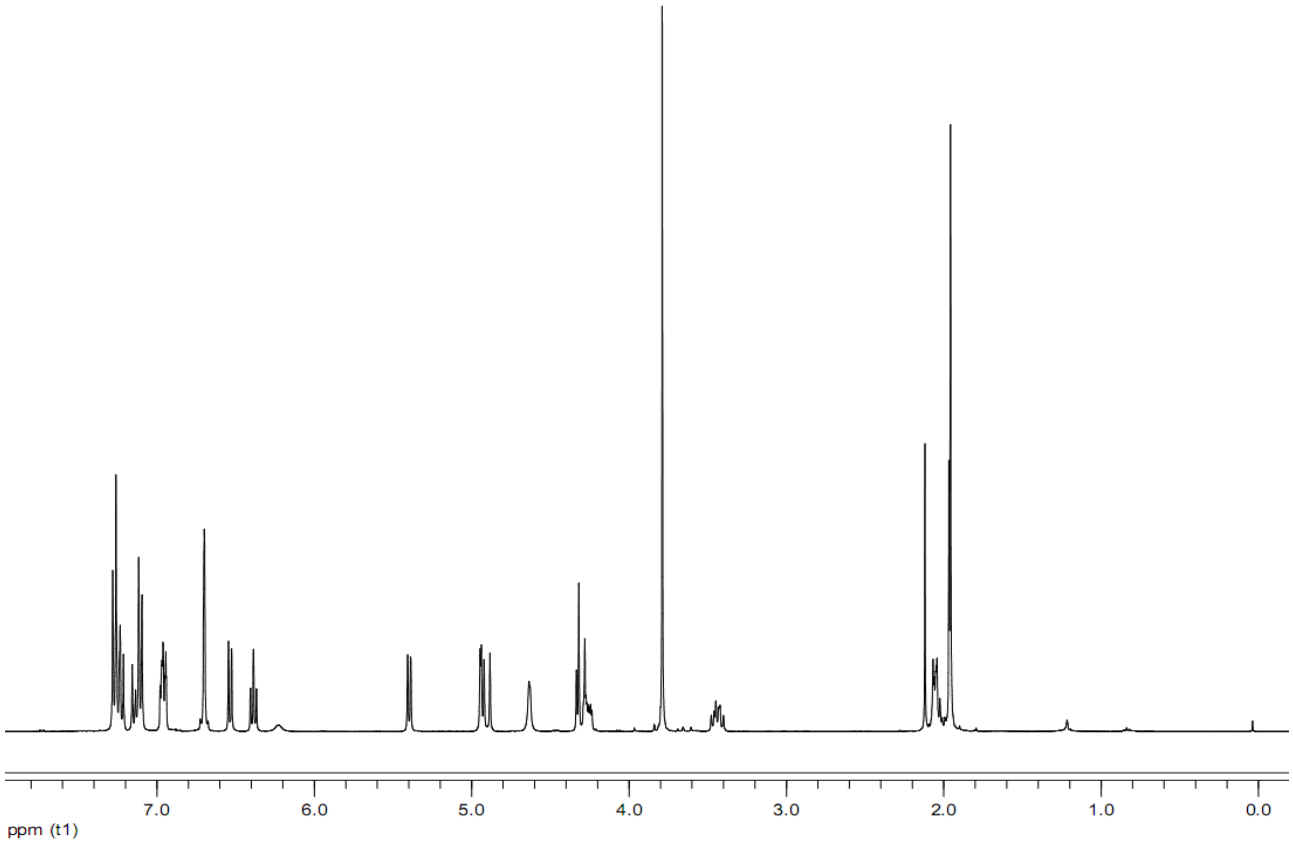
m.p. 234-236 °C.

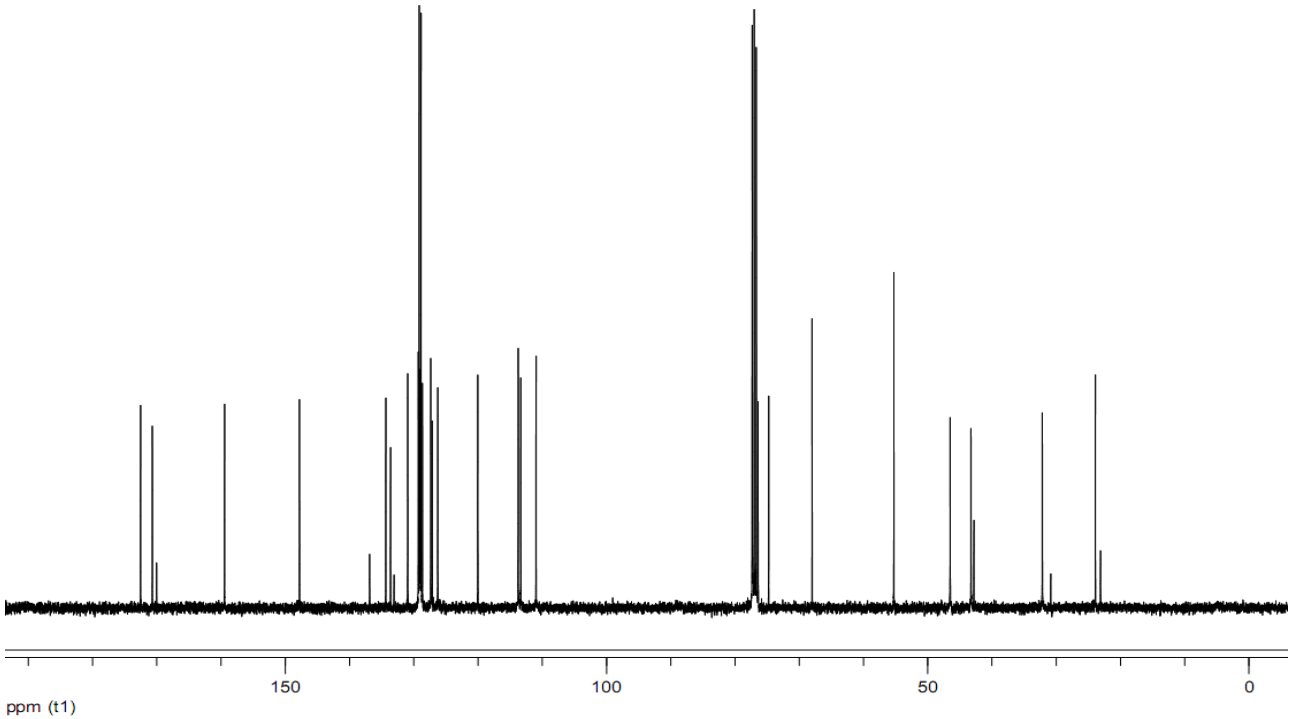
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.19 (m, 3H), 7.07 (d, *J* = 8.1 Hz, 2H), 6.95-6.90 (m, 2H), 6.66 (br, 2H), 6.50 (d, *J* = 7.5 Hz, 1H), 6.35 (t, *J* = 7.5, 1H), 5.36 (d, *J* = 7.5 Hz, 1H), 4.91 (d, *J* = 3.0 Hz, 1H), 4.87 (d, *J* = 15.2 Hz, 1H), 4.58 (br d, *J* = 3.0 Hz, 1H), 4.26 (d, *J* = 15.2 Hz, 1H), 4.26-4.19 (m, 1H), 3.75 (s, 3H), 3.40 (td, *J* = 11.7, 7.7 Hz, 1H), 2.05-1.98 (m, 2H), 1.92 (s, 3H).

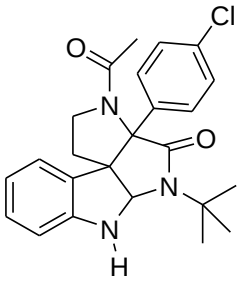
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.5, 170.7, 159.4, 147.8, 134.3, 133.6, 130.9, 129.3, 129.1 (2), 129.0, 128.8 (2), 127.3, 127.1, 126.2, 120.0, 113.7, 113.5, 111.0, 76.3, 74.7, 68.0, 55.3, 46.5, 43.2, 32.2, 23.9.

IR (thin film) 3319, 3051, 2936, 1699, 1632, 1515, 1494, 1386, 1342, 1253, 1038 cm^{-1}.

HRMS Calculated for $C_{28}H_{26}ClN_3O_3$ 487.1663, found 487.1655.







$C_{24}H_{26}ClN_3O_2$
MW = 423.94 g/mol

2j

General procedure using of Ugi adduct (284 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 5 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 70:30) as eluant gave the desired product (66%) as a white solid.

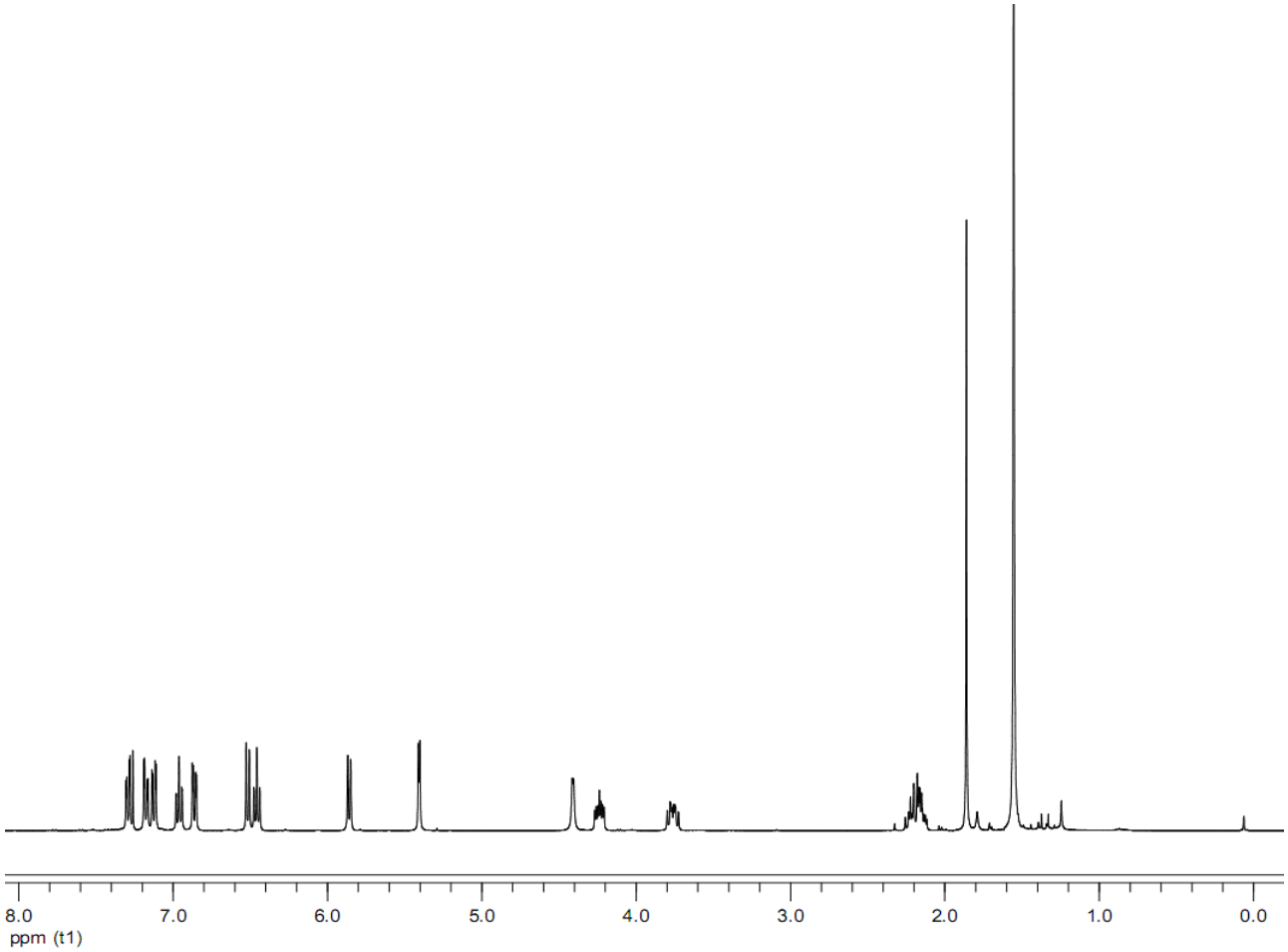
m.p. 269-270 °C.

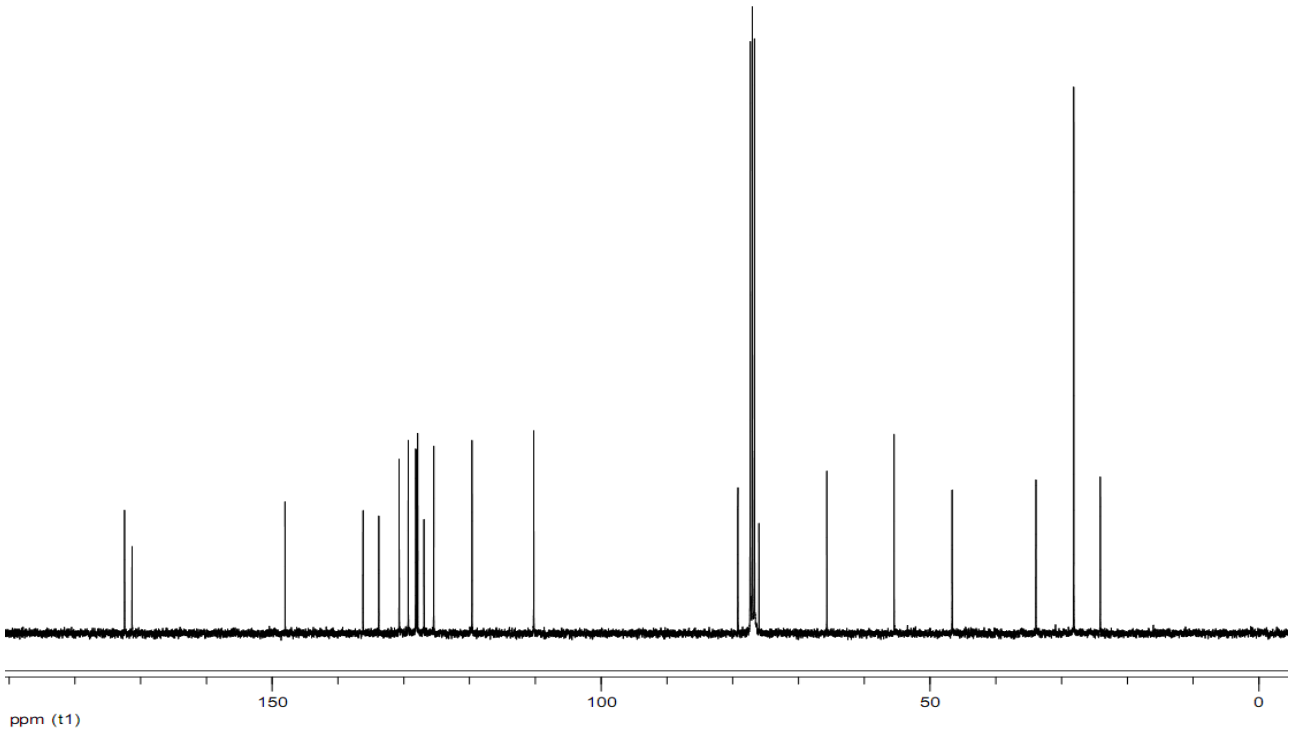
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.17 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.12 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.96 (td, *J* = 7.7, 1.1 Hz, 1H), 6.86 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.51 (d, *J* = 7.7 Hz, 1H), 6.46 (t, *J* = 7.7, 1H), 5.86 (d, *J* = 7.7 Hz, 1H), 5.41 (d, *J* = 3.6 Hz, 1H), 4.41 (br d, *J* = 3.6, 1H), 4.24 (ddd, *J* = 12.2, 7.5, 4.0 Hz, 1H), 3.76 (ddd, *J* = 12.2, 9.4, 7.5 Hz, 1H), 2.26-2.12 (m, 2H), 1.86 (s, 3H), 1.55 (s, 9H).

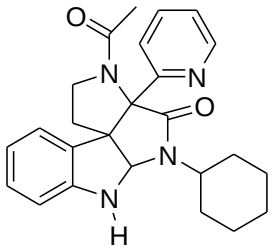
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.4, 171.3, 148.0, 136.2, 133.8, 130.7, 129.3, 128.2, 128.1, 127.9, 126.9, 125.4, 119.6, 110.2, 79.2, 76.0, 65.7, 55.5, 46.6, 33.9, 28.1 (3), 24.0.

IR (thin film) 3331, 3057, 2975, 2874, 1692, 1625, 1490, 1390 cm^{-1}.

HRMS Calculated for $C_{24}H_{26}ClN_3O_2$ 423.1714, found 423.1717.







$C_{25}H_{28}N_4O_2$
MW = 416.52 g/mol

2k

General procedure using of Ugi adduct (187 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 27 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 - 90:10) as eluant gave the desired product (46%) as a white solid.

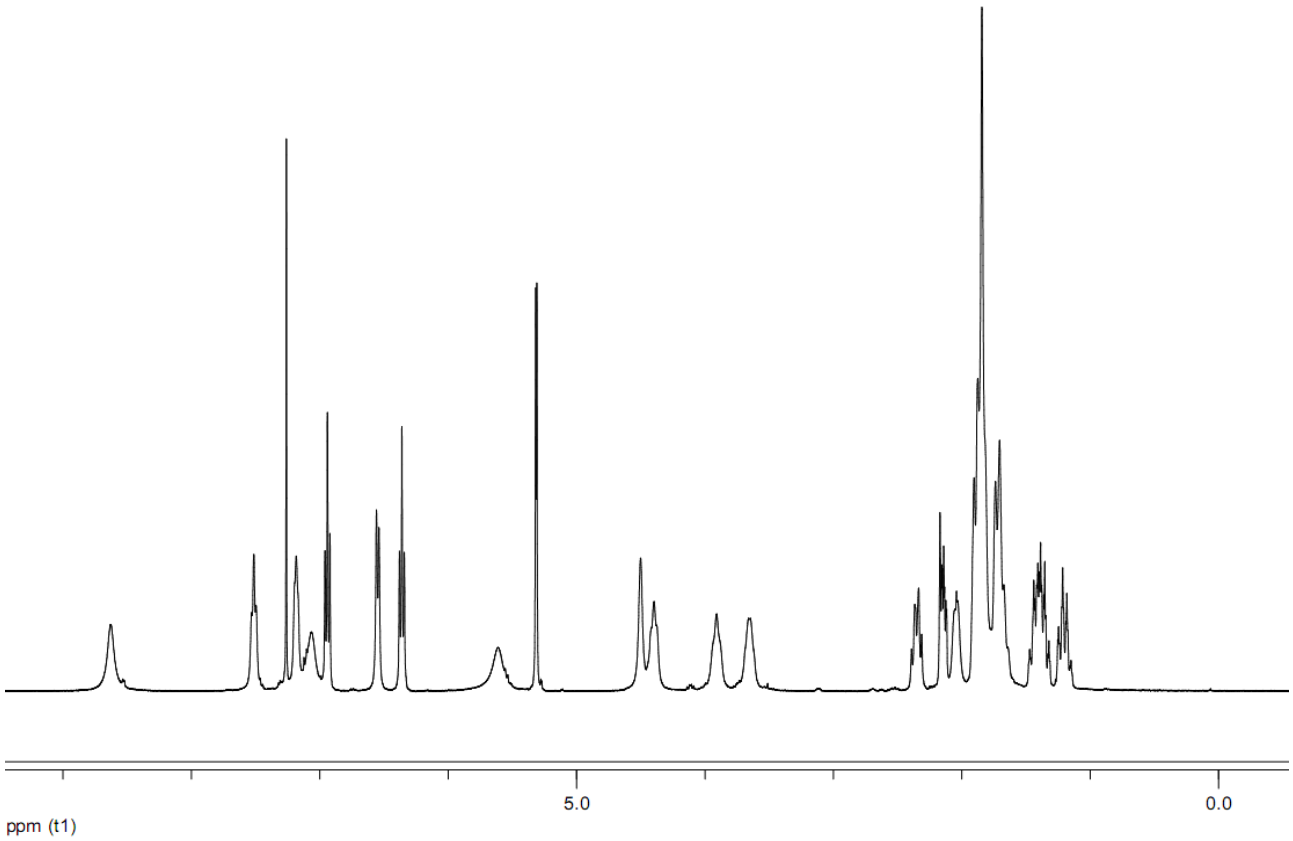
m.p. 270-271 °C.

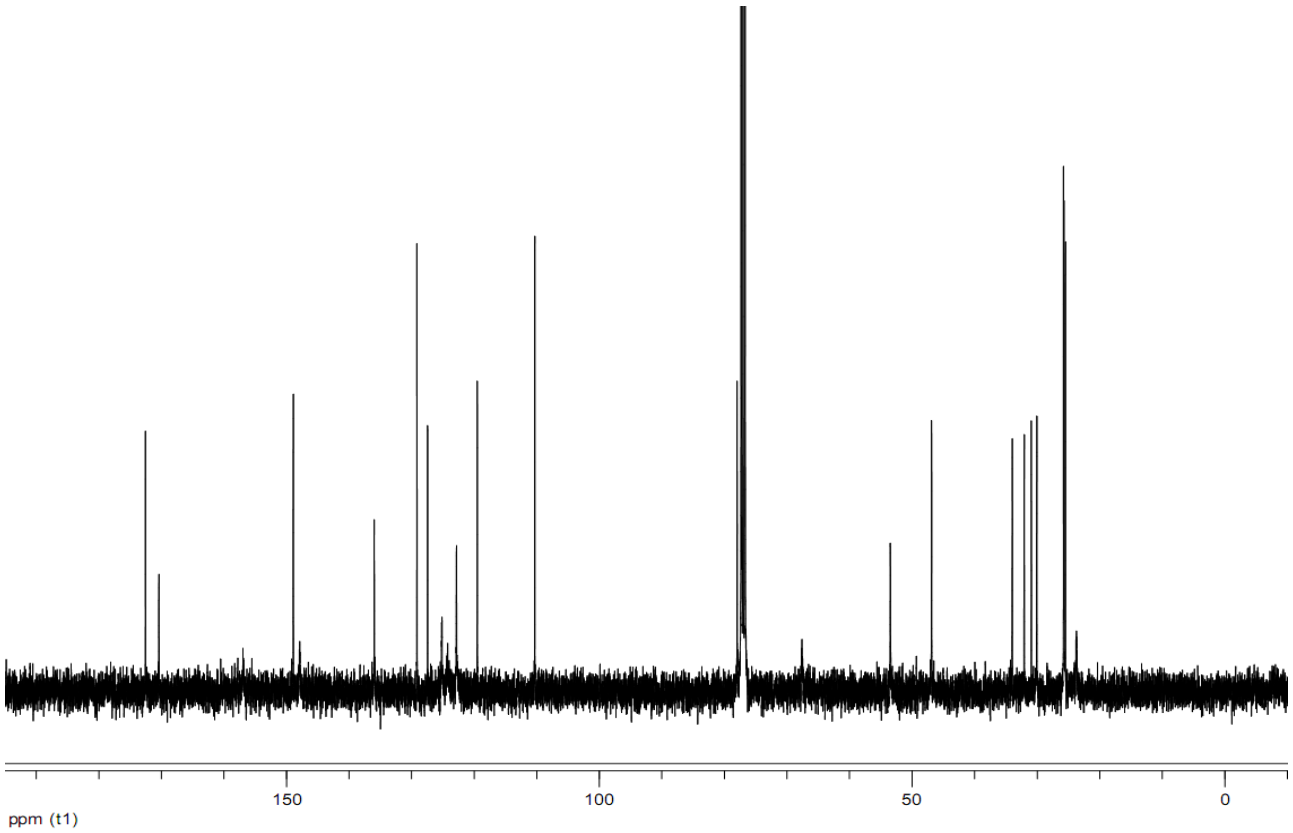
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (br s, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.20-7.17 (m, 1H), 7.07 (br s, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.55 (d, *J* = 7.5 Hz, 1H), 6.36 (t, *J* = 7.5 Hz, 1H), 5.61 (br, 1H), 5.31 (d, *J* = 3.8 Hz, 1H), 4.50 (br s, 1H), 4.40 (br t, 1H), 3.91 (br, 1H), 3.65 (br, 1H), 2.39-2.31 (m, 1H), 2.17-2.12 (m, 1H), 2.06-2.03 (m, 1H), 1.84 (s, 3H), 1.74-1.64 (m, 3H), 1.90-1.81 (m, 3H), 1.48-1.31 (m, 2H), 1.25-1.15 (m, 1H).

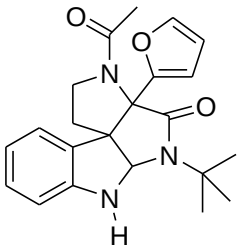
^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.5, 170.4, 148.9, 147.91, 136.0, 129.1 (2), 127.4, 125.1, 122.8, 119.5, 110.3 (2), 78.0, 77.2, 67.6, 53.5, 46.9, 34.0, 32.1, 30.9, 30.1, 25.8, 25.7, 25.5.

IR (thin film) 3315, 3053, 2932, 1687, 1625, 1487, 1431, 1339 cm^{-1}.

HRMS Calculated for $C_{25}H_{28}N_4O_2$ 416.2212, found 416.2209.







$C_{22}H_{25}N_3O_3$
MW = 379.45 g/mol

2l

General procedure using of Ugi adduct (165 mg, 0.43 mmol), copper acetate (86 mg, 0.43 mmol) and DBU (65 μL, 0.43 mmol). Reaction time: 4 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (50:50 – 70:30) as eluant gave the desired product (37%) as a white solid.

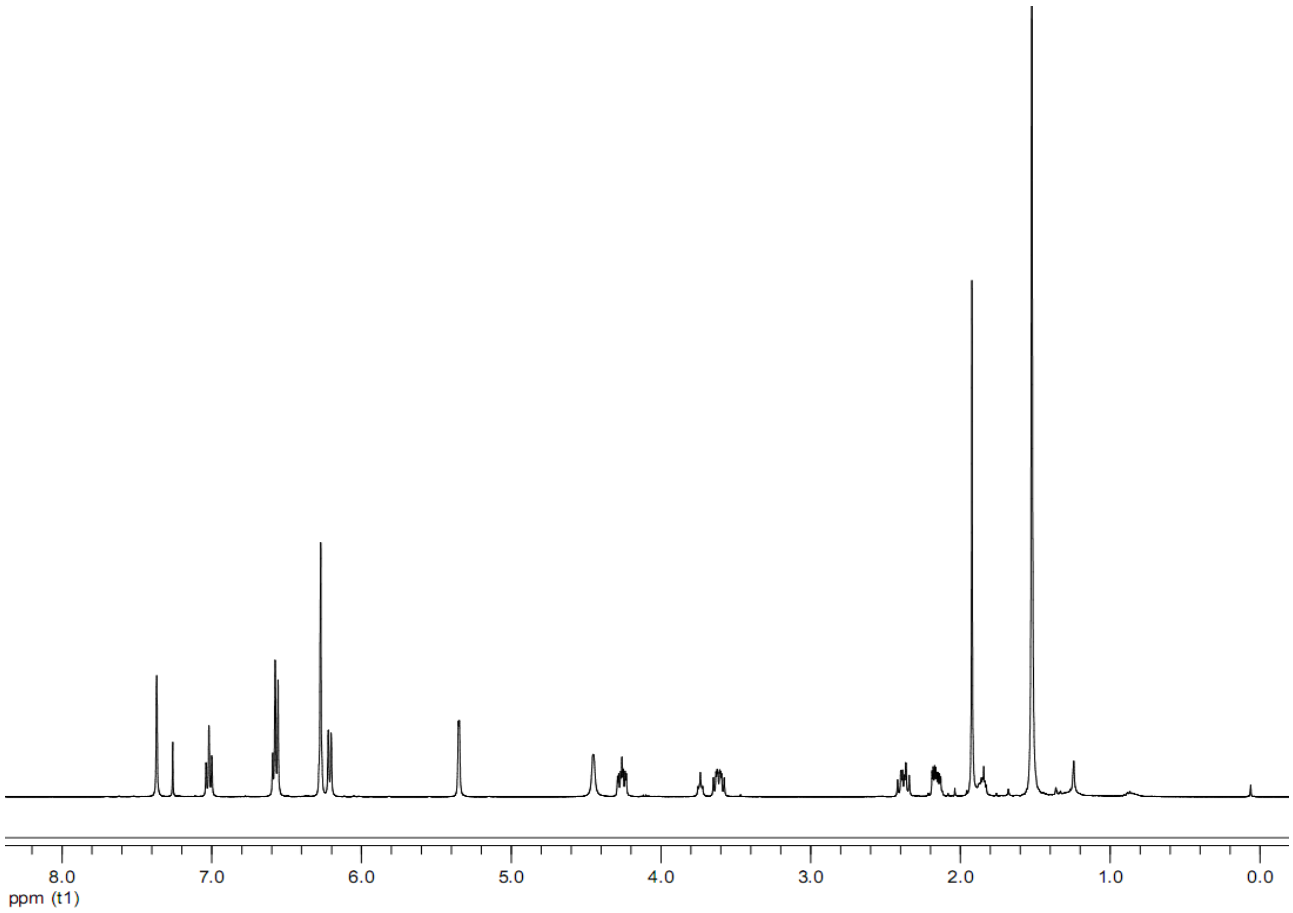
m.p. 238-239 °C.

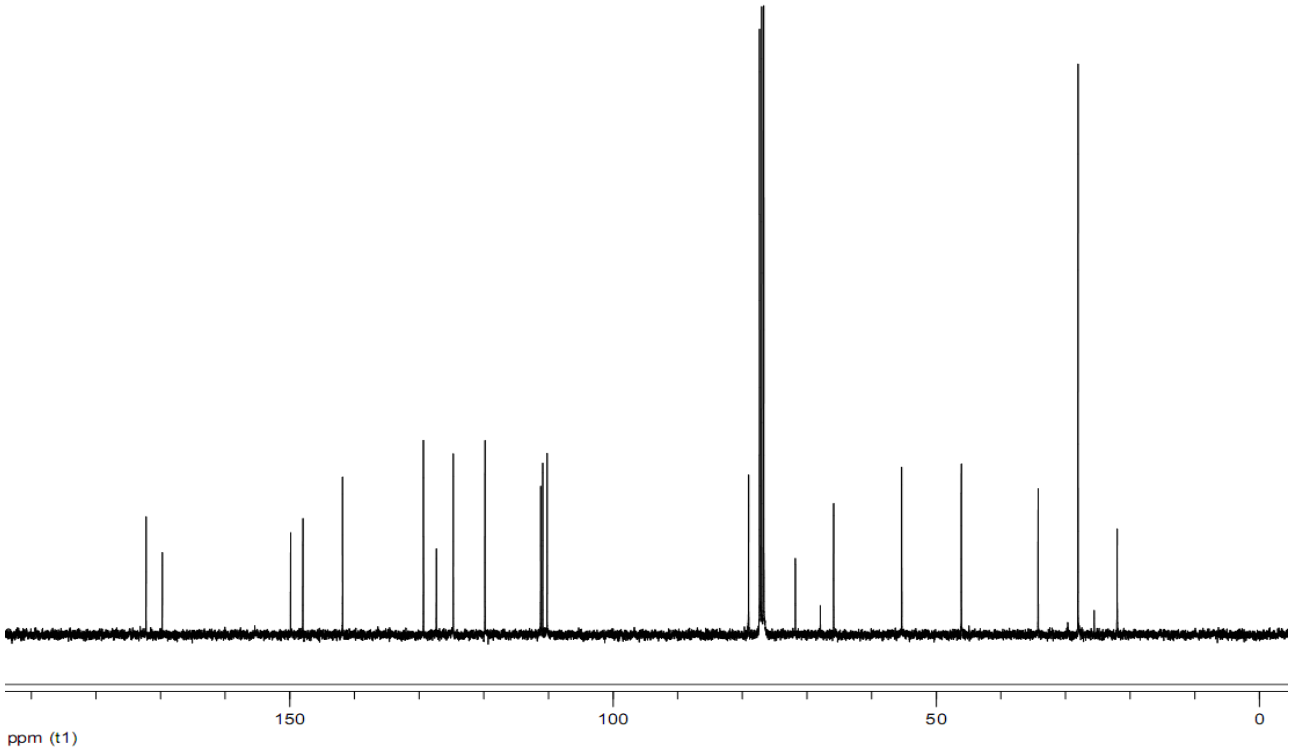
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.02 (t, *J* = 7.3 Hz, 1H), 6.58 (t, *J* = 7.3 Hz, 1H), 6.57 (d, *J* = 7.3 Hz, 1H), 6.27 (br s, 2H), 6.21 (d, *J* = 7.3 Hz, 1H), 5.35 (d, *J* = 2.5 Hz, 1H), 4.45 (s, 1H), 4.26 (ddd, *J* = 11.9, 8.5, 3.4 Hz, 1H), 3.61 (ddd, *J* = 11.9, 9.9, 6.8 Hz, 1H), 2.38 (dt, *J* = 13.1, 8.5 Hz, 1H), 2.19-2.13 (m, 1H), 1.92 (s, 3H), 1.52 (s, 9H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.2, 169.7, 149.9, 148.0, 141.8, 129.3, 127.3, 124.7, 119.8, 111.2, 110.9, 110.2, 79.0, 71.8, 65.9, 55.3, 46.1, 34.2, 28.1 (3), 22.0.

IR (thin film) 3331, 2971, 2927, 1695, 1626, 1489, 1472, 1393, 1331 cm^{-1}.

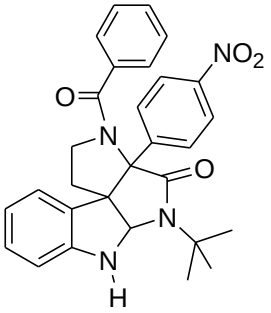
HRMS Calculated for $C_{22}H_{25}N_3O_3$ 376.1896, found 397.1889.





General Procedure for the One Pot Reaction

To a solution of tryptamine (1 equiv) in anhydrous methanol (5 M) was added successively under stirring, acid (1.0 equiv), isocyanide (1 equiv) and benzaldehyde (1 equiv). The mixture was stirred at room temperature for 6-24 h under nitrogen atmosphere. To this mixture was added anhydrous THF to a concentration (0.2 M), copper acetate (1 equiv) at 0 °C and then DBU (1 equiv). The reaction mixture was heated to reflux with a potassium hydroxide trap, until completion of the reaction checked with TLC analysis. Then, the reaction mixture was cooled at room temperature and the solvent was removed under reduced pressure. The crude residue was purified by flash column chromatography on silica gel using a gradient of EtOAc in petroleum ether as eluant.



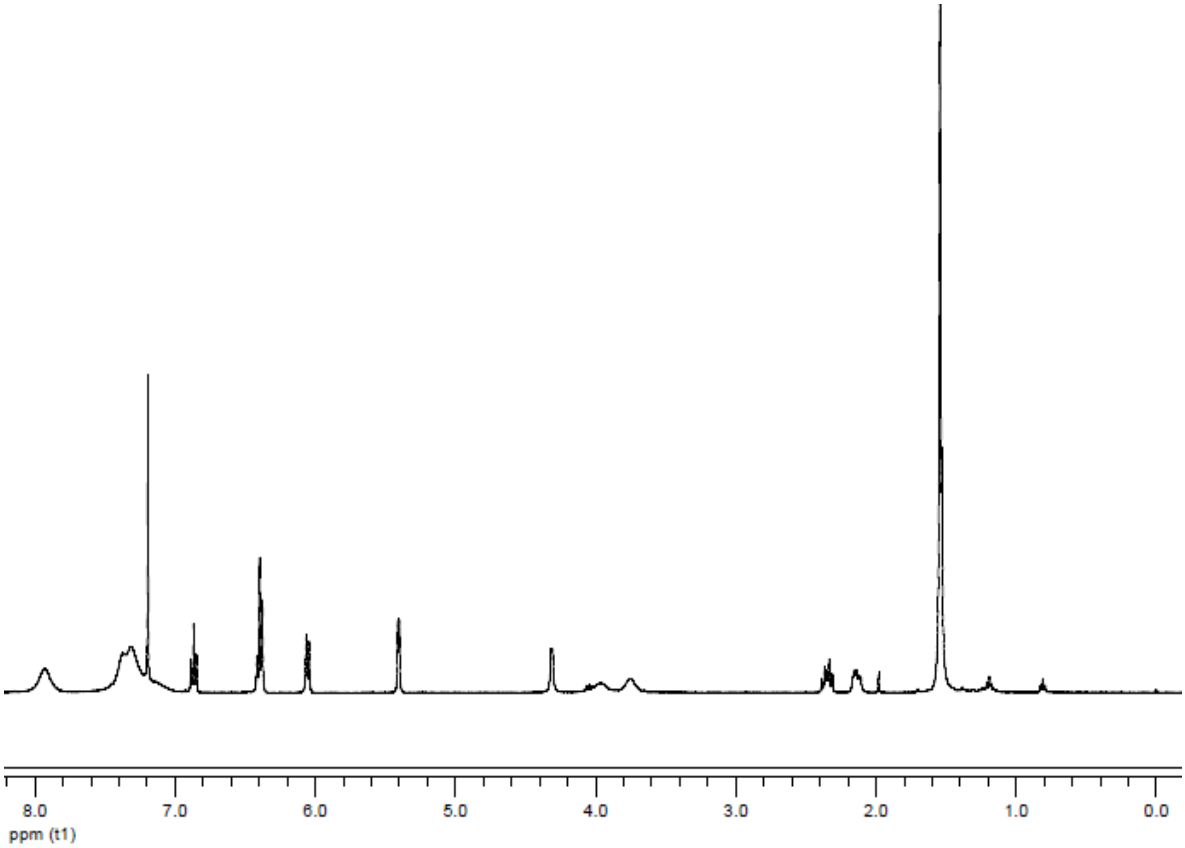
$C_{29}H_{28}N_4O_4$
MW = 496.56 g/mol

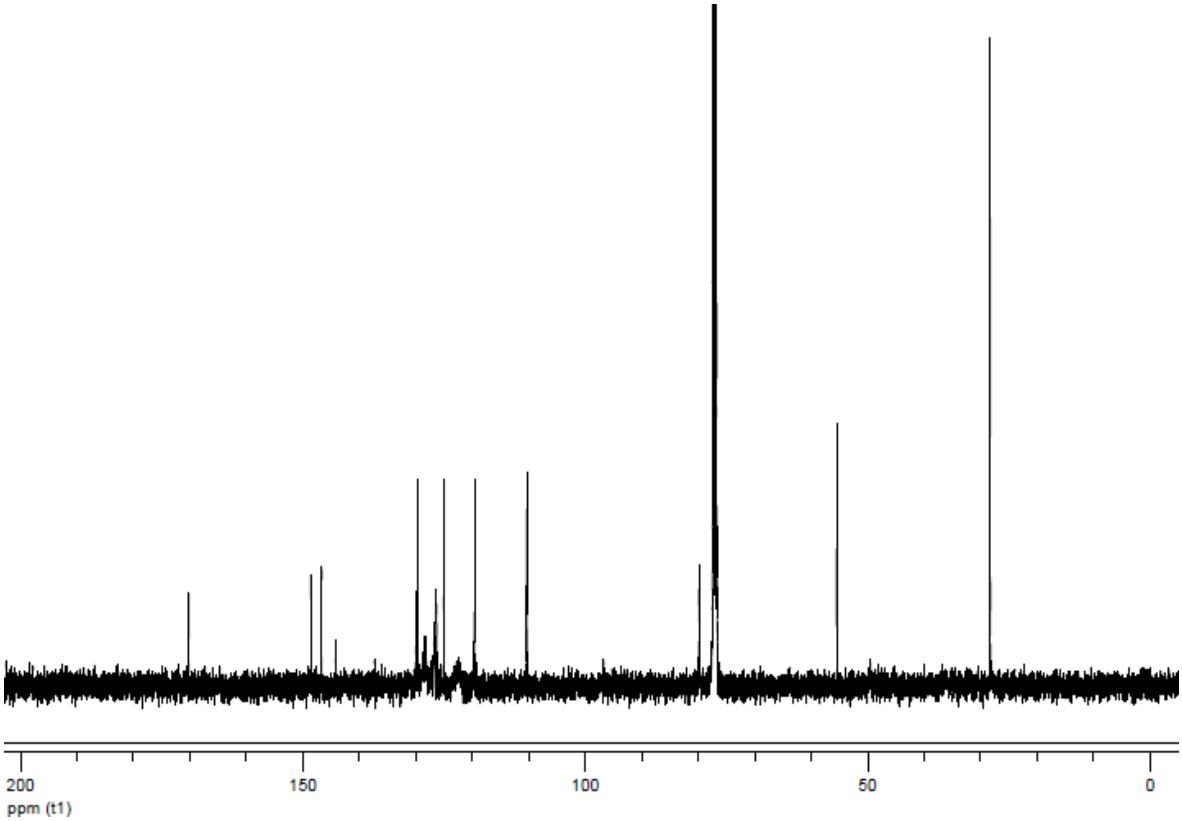
General procedure was followed using tryptamine (160 mg, 1 mmol), benzoic acid (122 g, 1 mmol), *t*-butyl isocyanide (120 μL, 1 mmol) and *p*-nitrobenzaldehyde (151 mg, 1 mmol). Reaction time: 24 h. After were added anhydrous THF (4.4 mL), copper acetate (200 mg, 1 mmol) and DBU (150 μL, 1 mmol). Reaction time: 22 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 90:10) as eluant gave the desired product (62%) as a yellow solid.

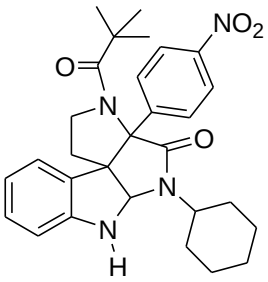
m.p. 332-333 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (br, 2H), 7.48-7.34 (m, 7H), 6.93 (t, *J* = 7.6 Hz, 1H), 6.46 (t, *J* = 7.6 Hz, 1H), 6.45 (d, *J* = 7.6 Hz, 1H), 6.12 (d, *J* = 7.6 Hz, 1H), 5.47 (d, *J* = 3.7 Hz, 1H), 4.38 (d, *J* = 3.7 Hz, 1H), 4.04 (br, 1H), 3.81 (br, 1H), 2.42 (dt, *J* = 13.4, 8.5 Hz, 1H), 2.23-2.17 (m, 1H), 1.61 (s, 9H).

IR (thin film) 3319, 2975, 2360, 2341, 1686, 1653, 1518, 1380, 1344 cm^{-1}.







$C_{29}H_{34}N_4O_4$
MW = 502.61 g/mol

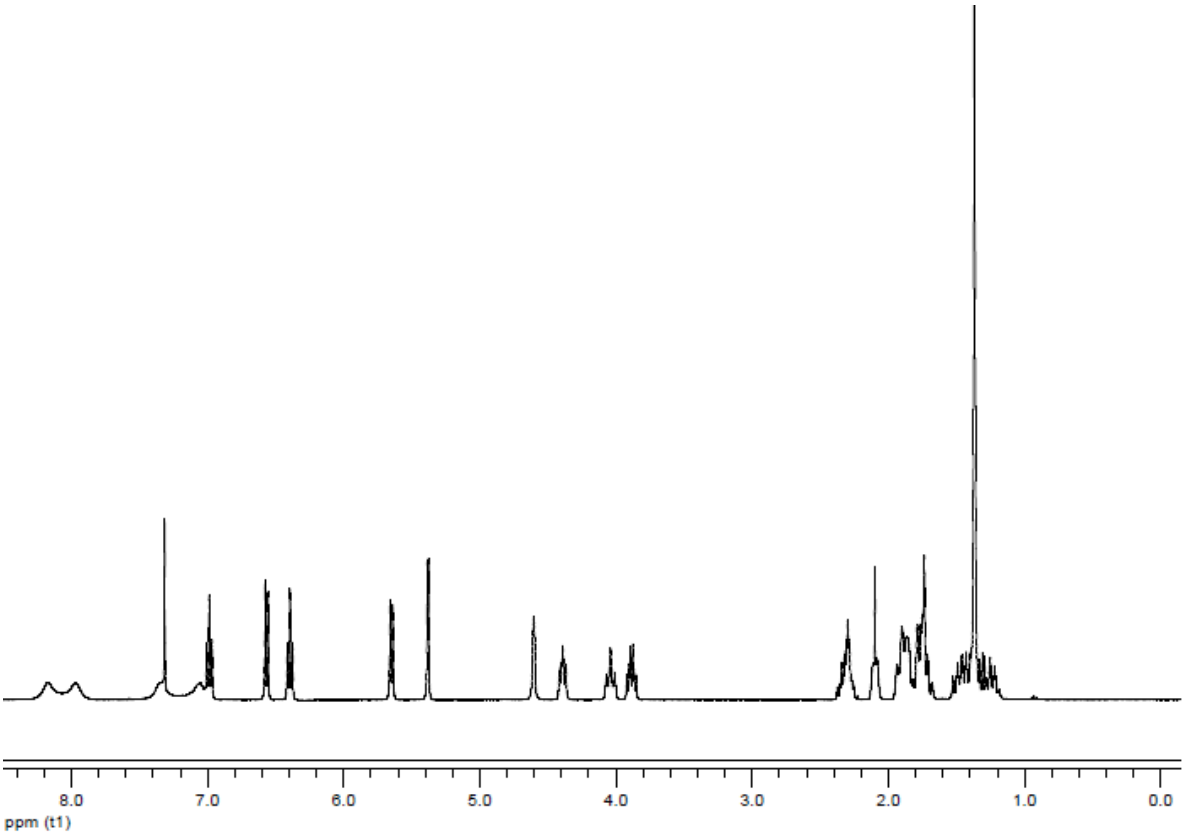
General procedure was followed using tryptamine (160 mg, 1 mmol), *t*-butylic acid (102 mg, 1 mmol), cyclohexyl isocyanide (130 μL, 1 mmol) and *p*-nitrobenzaldehyde (151 mg, 1 mmol). Reaction time: 3 days. After were added anhydrous THF (4.4 mL), copper acetate (200 mg, 1 mmol) and DBU (150 μL, 1 mmol). Reaction time: 22 h. Purification by flash chromatography using a gradient of ethyl acetate in petroleum ether (40:60 - 70:30) as eluant gave the desired product (43%) as a yellow solid.

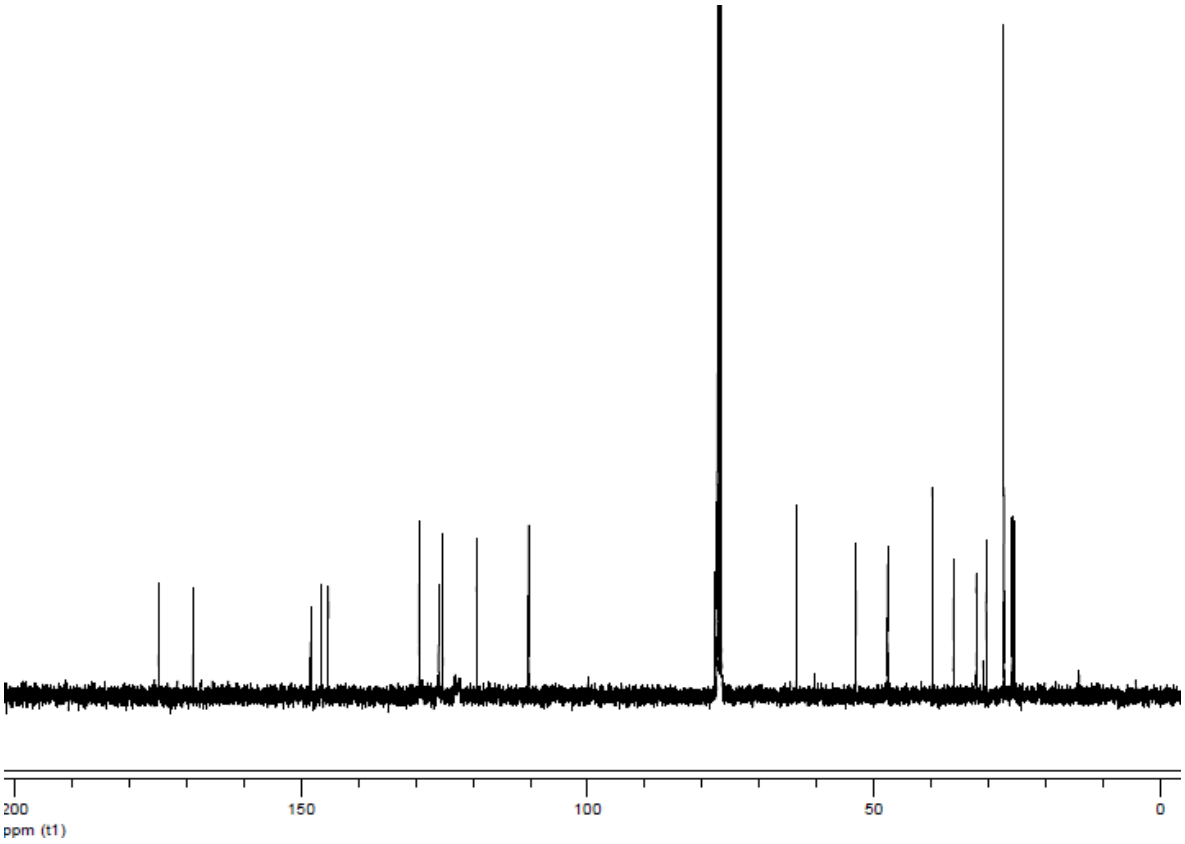
m.p. 311-312 °C.

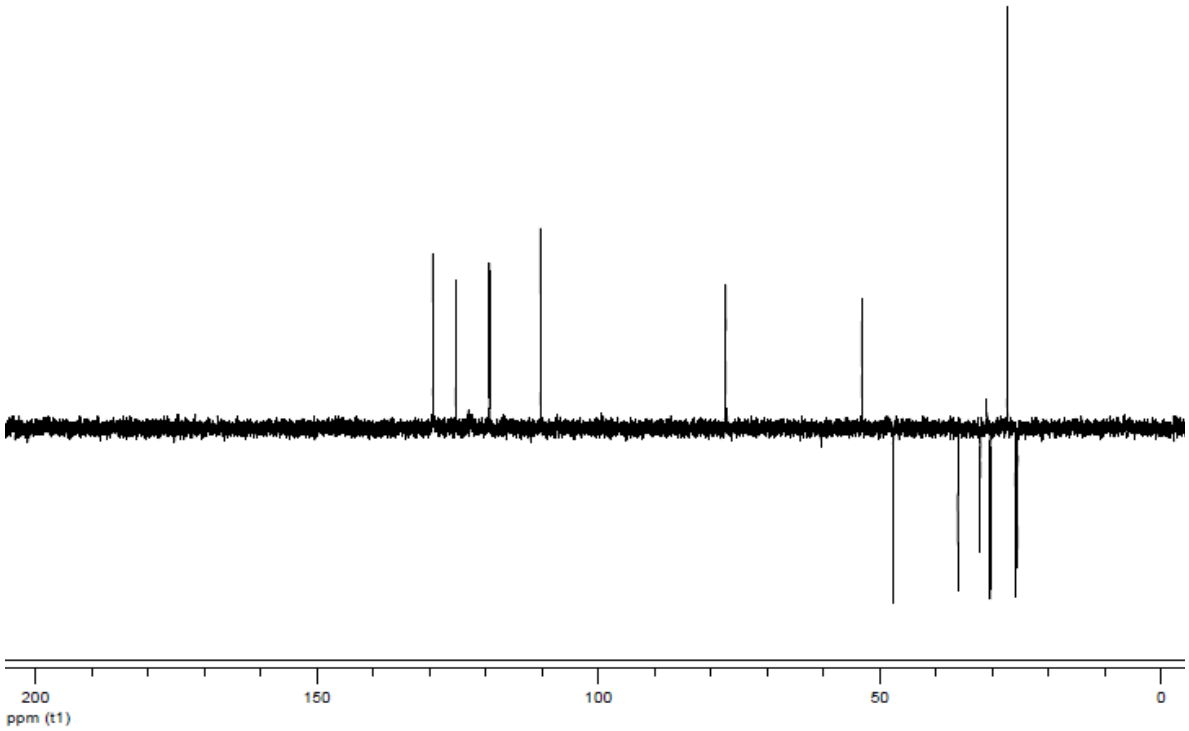
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (br s, 1H), 7.91 (br s, 1H), 7.29 (br s, 1H), 7.00 (br s, 1H), 6.91 (t, *J* = 7.7 Hz, 1H), 6.51 (d, *J* = 7.7 Hz, 1H), 6.34 (t, *J* = 7.7 Hz, 1H), 5.59 (d, *J* = 7.7 Hz, 1H), 5.33 (d, *J* = 3.5 Hz, 1H), 4.55 (br s, 1H), 4.39 (ddd, *J* = 10.1, 7.6, 2.5 Hz, 1H), 3.99 (tt, *J* = 12.0, 3.5 Hz, 1H), 3.83 (td, *J* = 10.1, 6.4 Hz, 1H), 2.32-2.20 (m, 2H), 2.06-2.03 (m, 1H), 1.89-1.78 (m, 3H), 1.74-1.65 (m, 3H), 1.47-1.34 (m, 2H), 1.31 (s, 9H), 1.24-1.12 (m, 1H).

^{13}C NMR (100.6 MHz, $CDCl_3$) δ 172.4, 168.8, 148.3, 146.5, 145.2, 129.4 (2), 126.0 (2), 125.3, 122.6 (2), 119.3, 110.3, 77.8, 77.4, 63.5, 53.2, 47.6, 39.8, 36.1, 32.1, 30.3, 27.3 (3), 25.9, 25.7, 25.5.

IR (thin film) 3318, 2933, 2856, 2360, 2341, 1684, 1518, 1344 cm^{-1}.







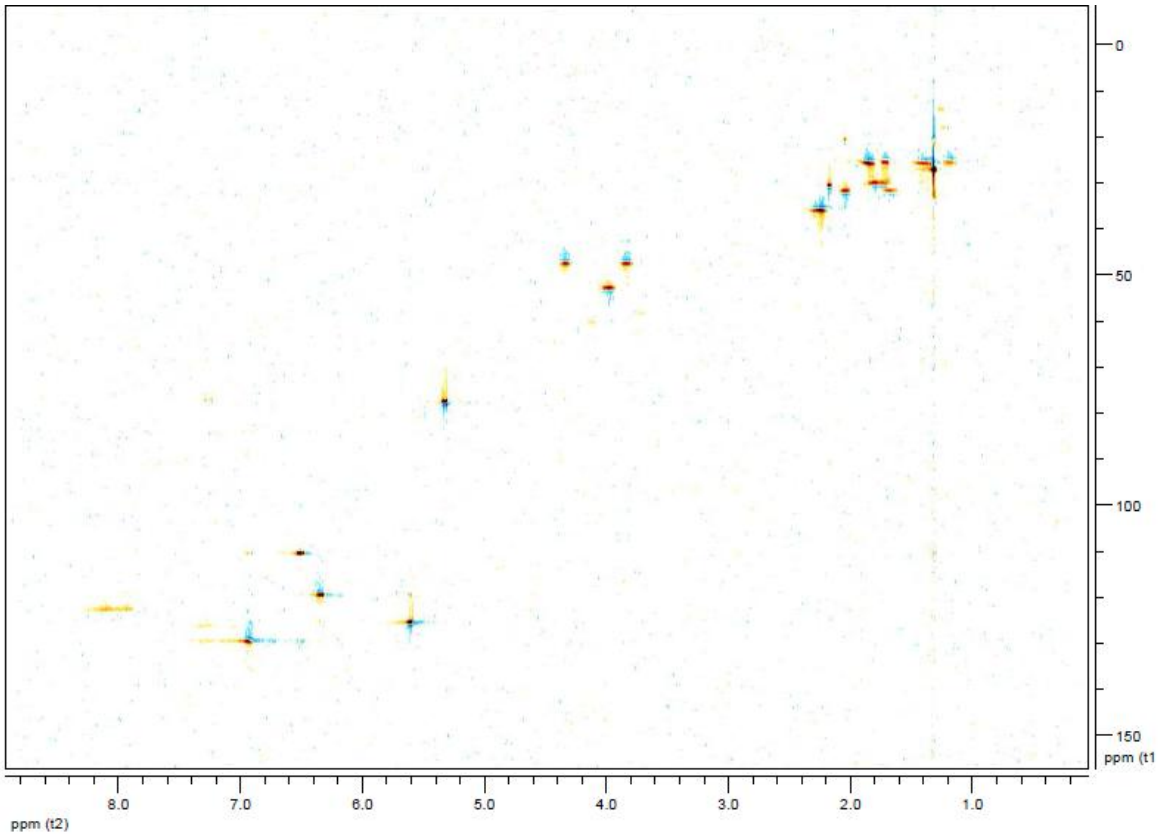


Table 1. Crystal data for mm77

Compound	mm77
Molecular formula	$C_{26}H_{28}N_4O_4$
Molecular weight	460.52
Crystal habit	pale yellow plate
Crystal dimensions(mm)	0.22x0.20x0.09
Crystal system	orthorhombic
Space group	Pbca
a(Å)	13.237(1)
b(Å)	14.763(1)
c(Å)	24.055(1)
α(°)	90.00
β(°)	90.00
γ(°)	90.00
V(Å^3)	4700.8(5)
Z	8
d(g-cm^{-3})	1.301
F(000)	1952
μ(cm^{-1})	0.089
Absorption corrections	multi-scan ; 0.9806 min, 0.9920 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	27.48
HKL ranges	-14 17 ; -19 19 ; -21 31
Reflections measured	33090
Unique data	5373
Rint	0.0481
Reflections used	4184
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	311
Reflections / parameter	13
wR2	0.1322
R1	0.0555
Weights a, b	0.0619 ; 2.7883
GoF	1.031
difference peak / hole (e Å^{-3})	0.287(0.042) / -0.319(0.042)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for mm77

atom	x	y	z	U(eq)
O(1)	2851(1)	3535(1)	2454(1)	38(1)
O(2)	1279(1)	886(1)	1912(1)	50(1)
O(3)	4732(1)	4117(2)	-172(1)	80(1)
O(4)	4166(3)	2827(2)	-414(1)	133(1)
N(1)	-19(1)	5156(1)	2270(1)	36(1)
N(2)	1277(1)	4145(1)	2597(1)	28(1)
N(3)	1133(1)	2393(1)	1851(1)	32(1)
N(4)	4179(2)	3467(2)	-110(1)	61(1)
C(1)	66(1)	5439(1)	1724(1)	32(1)
C(2)	-171(1)	6282(1)	1507(1)	39(1)
C(3)	2(2)	6436(1)	951(1)	44(1)
C(4)	395(2)	5768(2)	609(1)	44(1)
C(5)	605(1)	4911(1)	828(1)	38(1)
C(6)	444(1)	4749(1)	1385(1)	30(1)
C(7)	480(1)	3880(1)	1721(1)	28(1)
C(8)	292(1)	4225(1)	2324(1)	29(1)
C(9)	1981(1)	3686(1)	2309(1)	28(1)
C(10)	1492(1)	3337(1)	1761(1)	27(1)
C(11)	21(1)	2316(1)	1822(1)	39(1)
C(12)	-303(1)	3178(1)	1537(1)	37(1)
C(13)	1393(1)	4451(1)	3177(1)	30(1)
C(14)	2245(2)	5128(2)	3251(1)	53(1)
C(15)	2299(2)	5428(2)	3860(1)	71(1)
C(16)	2419(2)	4623(3)	4248(1)	79(1)
C(17)	1571(2)	3959(2)	4170(1)	63(1)
C(18)	1486(2)	3645(2)	3567(1)	47(1)
C(19)	1700(2)	1631(1)	1879(1)	37(1)
C(20)	2828(2)	1673(2)	1855(1)	48(1)
C(21)	2169(1)	3409(1)	1256(1)	27(1)
C(22)	2050(2)	2809(1)	812(1)	40(1)
C(23)	2696(2)	2836(1)	363(1)	48(1)
C(24)	3442(2)	3484(1)	352(1)	40(1)
C(25)	3546(1)	4123(1)	766(1)	36(1)
C(26)	2907(1)	4076(1)	1221(1)	30(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for mm77

O(1)-C(9)	1.224(2)	O(2)-C(19)	1.237(2)
O(3)-N(4)	1.215(3)	O(4)-N(4)	1.195(3)
N(1)-C(1)	1.382(2)	N(1)-C(8)	1.441(2)
N(1)-H(1N)	0.90(2)	N(2)-C(9)	1.344(2)
N(2)-C(8)	1.464(2)	N(2)-C(13)	1.475(2)
N(3)-C(19)	1.354(2)	N(3)-C(11)	1.477(2)
N(3)-C(10)	1.489(2)	N(4)-C(24)	1.478(3)
C(1)-C(2)	1.387(3)	C(1)-C(6)	1.397(2)
C(2)-C(3)	1.376(3)	C(2)-H(2)	0.9500
C(3)-C(4)	1.385(3)	C(3)-H(3)	0.9500
C(4)-C(5)	1.397(3)	C(4)-H(4)	0.9500
C(5)-C(6)	1.379(3)	C(5)-H(5)	0.9500
C(6)-C(7)	1.517(2)	C(7)-C(12)	1.530(2)
C(7)-C(8)	1.558(2)	C(7)-C(10)	1.564(2)
C(8)-H(8)	1.0000	C(9)-C(10)	1.556(2)
C(10)-C(21)	1.512(2)	C(11)-C(12)	1.508(3)
C(11)-H(11A)	0.9900	C(11)-H(11B)	0.9900
C(12)-H(12A)	0.9900	C(12)-H(12B)	0.9900
C(13)-C(14)	1.517(3)	C(13)-C(18)	1.522(3)
C(13)-H(13)	1.0000	C(14)-C(15)	1.533(3)
C(14)-H(14A)	0.9900	C(14)-H(14B)	0.9900
C(15)-C(16)	1.520(5)	C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900	C(16)-C(17)	1.502(4)
C(16)-H(16A)	0.9900	C(16)-H(16B)	0.9900
C(17)-C(18)	1.527(3)	C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900	C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900	C(19)-C(20)	1.495(3)
C(20)-H(20A)	0.9800	C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800	C(21)-C(26)	1.391(2)
C(21)-C(22)	1.397(2)	C(22)-C(23)	1.378(3)
C(22)-H(22)	0.9500	C(23)-C(24)	1.375(3)
C(23)-H(23)	0.9500	C(24)-C(25)	1.378(3)
C(25)-C(26)	1.386(2)	C(25)-H(25)	0.9500
C(26)-H(26)	0.9500		

C(1)-N(1)-C(8)	110.5(2)	C(1)-N(1)-H(1N)	124(1)
C(8)-N(1)-H(1N)	119(1)	C(9)-N(2)-C(8)	115.2(1)
C(9)-N(2)-C(13)	124.8(1)	C(8)-N(2)-C(13)	119.5(1)
C(19)-N(3)-C(11)	119.5(2)	C(19)-N(3)-C(10)	127.4(1)
C(11)-N(3)-C(10)	112.5(1)	O(4)-N(4)-O(3)	123.8(2)
O(4)-N(4)-C(24)	117.6(2)	O(3)-N(4)-C(24)	118.5(2)
N(1)-C(1)-C(2)	127.6(2)	N(1)-C(1)-C(6)	111.3(2)
C(2)-C(1)-C(6)	121.0(2)	C(3)-C(2)-C(1)	118.5(2)
C(3)-C(2)-H(2)	120.8	C(1)-C(2)-H(2)	120.8
C(2)-C(3)-C(4)	121.4(2)	C(2)-C(3)-H(3)	119.3
C(4)-C(3)-H(3)	119.3	C(3)-C(4)-C(5)	119.8(2)
C(3)-C(4)-H(4)	120.1	C(5)-C(4)-H(4)	120.1
C(6)-C(5)-C(4)	119.4(2)	C(6)-C(5)-H(5)	120.3
C(4)-C(5)-H(5)	120.3	C(5)-C(6)-C(1)	119.8(2)
C(5)-C(6)-C(7)	131.2(2)	C(1)-C(6)-C(7)	108.5(2)
C(6)-C(7)-C(12)	113.4(1)	C(6)-C(7)-C(8)	102.3(1)
C(12)-C(7)-C(8)	112.5(2)	C(6)-C(7)-C(10)	119.5(1)
C(12)-C(7)-C(10)	104.6(1)	C(8)-C(7)-C(10)	104.2(1)

N(1)-C(8)-N(2)	111.8(1)	N(1)-C(8)-C(7)	105.9(1)
N(2)-C(8)-C(7)	104.4(1)	N(1)-C(8)-H(8)	111.5
N(2)-C(8)-H(8)	111.5	C(7)-C(8)-H(8)	111.5
O(1)-C(9)-N(2)	126.7(2)	O(1)-C(9)-C(10)	124.9(2)
N(2)-C(9)-C(10)	108.4(1)	N(3)-C(10)-C(21)	111.8(1)
N(3)-C(10)-C(9)	108.7(1)	C(21)-C(10)-C(9)	114.3(1)
N(3)-C(10)-C(7)	102.4(1)	C(21)-C(10)-C(7)	114.9(1)
C(9)-C(10)-C(7)	103.9(1)	N(3)-C(11)-C(12)	103.9(1)
N(3)-C(11)-H(11A)	111.0	C(12)-C(11)-H(11A)	111.0
N(3)-C(11)-H(11B)	111.0	C(12)-C(11)-H(11B)	111.0
H(11A)-C(11)-H(11B)	109.0	C(11)-C(12)-C(7)	104.3(1)
C(11)-C(12)-H(12A)	110.9	C(7)-C(12)-H(12A)	110.9
C(11)-C(12)-H(12B)	110.9	C(7)-C(12)-H(12B)	110.9
H(12A)-C(12)-H(12B)	108.9	N(2)-C(13)-C(14)	112.9(1)
N(2)-C(13)-C(18)	110.7(2)	C(14)-C(13)-C(18)	112.5(2)
N(2)-C(13)-H(13)	106.8	C(14)-C(13)-H(13)	106.8
C(18)-C(13)-H(13)	106.8	C(13)-C(14)-C(15)	109.7(2)
C(13)-C(14)-H(14A)	109.7	C(15)-C(14)-H(14A)	109.7
C(13)-C(14)-H(14B)	109.7	C(15)-C(14)-H(14B)	109.7
H(14A)-C(14)-H(14B)	108.2	C(16)-C(15)-C(14)	111.5(2)
C(16)-C(15)-H(15A)	109.3	C(14)-C(15)-H(15A)	109.3
C(16)-C(15)-H(15B)	109.3	C(14)-C(15)-H(15B)	109.3
H(15A)-C(15)-H(15B)	108.0	C(17)-C(16)-C(15)	110.8(2)
C(17)-C(16)-H(16A)	109.5	C(15)-C(16)-H(16A)	109.5
C(17)-C(16)-H(16B)	109.5	C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	108.1	C(16)-C(17)-C(18)	111.8(2)
C(16)-C(17)-H(17A)	109.3	C(18)-C(17)-H(17A)	109.3
C(16)-C(17)-H(17B)	109.3	C(18)-C(17)-H(17B)	109.3
H(17A)-C(17)-H(17B)	107.9	C(13)-C(18)-C(17)	110.8(2)
C(13)-C(18)-H(18A)	109.5	C(17)-C(18)-H(18A)	109.5
C(13)-C(18)-H(18B)	109.5	C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	108.1	O(2)-C(19)-N(3)	119.4(2)
O(2)-C(19)-C(20)	119.3(2)	N(3)-C(19)-C(20)	121.2(2)
C(19)-C(20)-H(20A)	109.5	C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5	C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5	H(20B)-C(20)-H(20C)	109.5
C(26)-C(21)-C(22)	118.8(2)	C(26)-C(21)-C(10)	121.0(2)
C(22)-C(21)-C(10)	120.2(2)	C(23)-C(22)-C(21)	120.7(2)
C(23)-C(22)-H(22)	119.6	C(21)-C(22)-H(22)	119.6
C(24)-C(23)-C(22)	118.8(2)	C(24)-C(23)-H(23)	120.6
C(22)-C(23)-H(23)	120.6	C(23)-C(24)-C(25)	122.3(2)
C(23)-C(24)-N(4)	118.5(2)	C(25)-C(24)-N(4)	119.2(2)
C(24)-C(25)-C(26)	118.4(2)	C(24)-C(25)-H(25)	120.8
C(26)-C(25)-H(25)	120.8	C(25)-C(26)-C(21)	120.8(2)
C(25)-C(26)-H(26)	119.6	C(21)-C(26)-H(26)	119.6

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for mm77

atom	U11	U22	U33	U23	U13	U12
O(1)	24(1)	55(1)	35(1)	-5(1)	-2(1)	10(1)
O(2)	62(1)	27(1)	60(1)	3(1)	29(1)	1(1)
O(3)	55(1)	142(2)	44(1)	11(1)	12(1)	-13(1)
O(4)	237(3)	77(2)	85(2)	-18(1)	104(2)	7(2)
N(1)	34(1)	36(1)	37(1)	-4(1)	1(1)	12(1)
N(2)	23(1)	34(1)	27(1)	-4(1)	-1(1)	4(1)
N(3)	30(1)	25(1)	42(1)	-1(1)	7(1)	0(1)
N(4)	78(2)	72(1)	32(1)	11(1)	16(1)	21(1)
C(1)	24(1)	32(1)	40(1)	-2(1)	-7(1)	0(1)
C(2)	36(1)	30(1)	52(1)	-4(1)	-9(1)	4(1)
C(3)	40(1)	36(1)	57(1)	10(1)	-12(1)	1(1)
C(4)	43(1)	51(1)	39(1)	9(1)	-6(1)	2(1)
C(5)	38(1)	42(1)	35(1)	-1(1)	-5(1)	6(1)
C(6)	25(1)	31(1)	34(1)	-2(1)	-7(1)	1(1)
C(7)	23(1)	29(1)	32(1)	-3(1)	-3(1)	1(1)
C(8)	22(1)	30(1)	33(1)	-2(1)	-1(1)	2(1)
C(9)	25(1)	31(1)	28(1)	-1(1)	2(1)	3(1)
C(10)	25(1)	24(1)	32(1)	-3(1)	0(1)	0(1)
C(11)	31(1)	32(1)	54(1)	-4(1)	1(1)	-6(1)
C(12)	29(1)	35(1)	47(1)	-9(1)	-8(1)	-3(1)
C(13)	27(1)	38(1)	26(1)	-5(1)	2(1)	2(1)
C(14)	49(1)	69(2)	40(1)	-19(1)	14(1)	-24(1)
C(15)	54(1)	109(2)	51(1)	-41(2)	16(1)	-37(2)
C(16)	46(1)	156(3)	34(1)	-31(2)	-8(1)	29(2)
C(17)	75(2)	84(2)	30(1)	7(1)	1(1)	24(2)
C(18)	56(1)	49(1)	37(1)	5(1)	5(1)	11(1)
C(19)	45(1)	30(1)	38(1)	4(1)	16(1)	6(1)
C(20)	43(1)	42(1)	58(1)	12(1)	15(1)	15(1)
C(21)	29(1)	26(1)	28(1)	-2(1)	-2(1)	4(1)
C(22)	55(1)	32(1)	32(1)	-6(1)	-1(1)	-6(1)
C(23)	77(2)	40(1)	27(1)	-8(1)	3(1)	3(1)
C(24)	51(1)	44(1)	26(1)	5(1)	7(1)	13(1)
C(25)	31(1)	43(1)	34(1)	5(1)	1(1)	1(1)
C(26)	27(1)	31(1)	32(1)	-3(1)	-1(1)	2(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for mm77

atom	x	y	z	U(eq)
H(1N)	-470(20)	5390(10)	2520(10)	43
H(2)	-446	6743	1737	47
H(3)	-151	7013	798	53
H(4)	520	5891	228	53
H(5)	857	4444	594	46
H(8)	-232	3854	2518	34
H(11A)	-276	2273	2199	47
H(11B)	-183	1778	1603	47
H(12A)	-990	3358	1656	44
H(12B)	-298	3104	1128	44
H(13)	754	4772	3279	36
H(14A)	2128	5661	3010	64
H(14B)	2894	4847	3140	64
H(15A)	2879	5844	3910	86
H(15B)	1675	5761	3958	86
H(16A)	3072	4318	4173	95
H(16B)	2427	4837	4638	95
H(17A)	928	4246	4284	76
H(17B)	1685	3427	4413	76
H(18A)	2091	3286	3467	57
H(18B)	886	3250	3526	57
H(20A)	3114	1268	2137	71
H(20B)	3052	2295	1926	71
H(20C)	3058	1482	1486	71
H(22)	1517	2378	819	48
H(23)	2627	2415	67	57
H(25)	4044	4584	739	43
H(26)	2975	4505	1513	36