Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supplementary Information for

Synthesis of polycyclic spiroindolines by highly diastereo-selective interrupted Ugi cascade reactions of 3-(2-isocyano-ethyl)indoles

Jordy M. Saya,[†] Barry Oppelaar,[†] Râzvan C. Cioc,[†] Gydo van der Heijden,[†] Christophe M. L. Vande Velde,[‡] Romano V. A. Orru,[†] Eelco Ruijter*[,†]

[†] Department of Chemistry & Pharmaceutical Sciences and Amsterdam Institute for Molecules, Medicines & Systems (AIMMS), Vrije Universiteit Amsterdam, De Boelelaan 1108, 1081 HZ Amsterdam, The Netherlands

[‡] Faculty of Applied Engineering, Advanced Reactor Technology, University of Antwerp, Groenenborgerlaan 171, 2020 Antwerpen, Belgium

E-mail: e.ruijter@vu.nl

Content

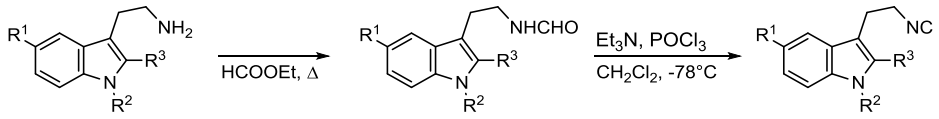
General information

Unless stated otherwise, all solvents and commercially available reagents were used as purchased. Dry dichloromethane was obtained by an Inert Solvent Purification System. All other solvents were used as purchased. 1-methyltryptamine[1], 2-methyltryptamine[2] and 1-methylcyclohexane-1-carboxaldehyde[3] were synthesized according to literature procedures.

Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 500 (125.78 MHz for ^{13}C) or Bruker Avance 300 (83.85 MHz for ^{13}C) using the residual solvent as internal standard (^{1}H: δ 7.26 ppm, $^{13}C\{^{1}H\}$: δ 77.16 ppm for $CDCl_3$, ^{1}H: δ 2.50 ppm, $^{13}C\{1H\}$: δ 39.52 ppm for DMSO-d_6). Chemical shifts (δ) are given in ppm and coupling constants (J) are quoted in hertz (Hz). Resonances are described as s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sex (sextet), sep (septet), br (broad singlet) and m (multiplet) or combinations thereof. Infrared (IR) spectra were recorded neat using a Shimadzu FTIR-8400s spectrophotometer and wavelengths are reported in cm^{-1}. Electrospray Ionization (ESI) high-resolution mass spectrometry (HRMS) was carried out using a Bruker microTOF-Q instrument in positive ion mode (capillary potential of 4500 V). Flash chromatography was performed on Silicycle Silia-P Flash Silica Gel (particle size 40-63 µm, pore diameter 60Å) using the indicated eluent. Thin Layer Chromatography (TLC) was performed using TLC plates from Merck (SiO_2, Kieselgel 60 F254 neutral, on aluminium with fluorescence indicator) and compounds were visualized by UV detection (254 nm) and $KMnO_4$ stain. X-ray analysis was performed on an Agilent SuperNova diffractometer with Cu K(α) microsource, mirror monochromator and Atlas CCD detector. The data were reduced and corrected for absorption with CrysAlisPro, Agilent Technologies, Version 1.171.38.41r (Rigaku OD, 2015). The structure was solved with SHELXS-2014/7[3] and refined with SHELXL-2014/7[4] and the ShelxLE graphical interface[5].

Synthetic procedures

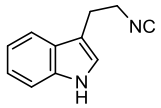
General procedure A: Synthesis of the tryptamine derived isocyanides.



9

Prepared according to a modified procedure by Zhao and coworkers. Tryptamine was mixed with ethyl formate (1.25 M) and heated to reflux for 16 h. The excess ethyl formate was concentrated *in vacuo*. Without further purification, the crude formamide was dissolved in anhydrous dichloromethane (0.5 M). Subsequently triethylamine (5 equiv) and phosphoryl chloride (1.5 equiv) were respectively dropwise added at −78°C. After mixing for 3 h at this temperature, the reaction was quenched by addition of the crude mixture in ice-cold water. The product was extracted three times with dichloromethane, washed with water and brine, dried over sodium sulfate and concentrated *in vacuo*. Pure isocyanide could be obtained by flash column chromatography using dichloromethane as eluent.

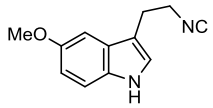
3-(2-isocyanoethyl)-1*H*-indole (9a)



According to procedure A the formamide was prepared using tryptamine (10 g, 62.4 mmol) and ethyl formate (50 ml). Subsequently, the isocyanide could be obtained using crude formamide, triethylamine (43.5 ml, 312 mmol) and phosphoryl chloride (8.7 mL, 96.6 mmol) in dichloromethane (120 ml). The title compound was isolated as a brownish solid (6.1 g, 36 mmol, 57%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.28 (td, *J* = 9.0, 8.1, 2.8 Hz, 1H), 7.21 (td, *J* = 7.5, 2.8 Hz, 1H), 7.10 (t, *J* = 2.1 Hz, 1H), 3.67 (tt, *J* = 7.0, 1.9 Hz, 2H), 3.18 (tt, *J* = 7.0, 2.0 Hz, 2H).

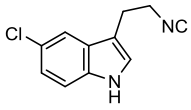
3-(2-isocyanoethyl)-5-methoxy-1*H*-indole (9b)



5-methoxytryptamine hydrochloric acid (4.5 g, 20 mmol) was mixed with ethyl formate (20 ml) and potassium carbonate (2.76 g, 20 mmol) and heated to reflux for 16 h. Water was added to the mixture and the product was extracted with ethyl acetate. After washing with brine, drying over sodium sulfate and concentrating *in vacuo*, the crude formamide was directly used without additional purification in the isocyanide synthesis. Following procedure A the isocyanide could be obtained using crude formamide, triethylamine (13.9 mL, 100 mmol) and phosphoryl chloride (2.79 mL, 30 mmol) in dichloromethane (40 ml). The title compound was isolated as a brownish solid (1.21 g, 6.0 mmol, 30%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.99 (s, 1H), 7.13 (d, *J* = 1.9 Hz, 1H), 6.98 (d, *J* = 2.0 Hz, 1H), 6.89 (dd, *J* = 8.8, 2.3 Hz, 1H), 3.87 (s, 2H), 3.66 (t, *J* = 7.1 Hz, 2H), 3.14 (t, *J* = 7.0 Hz, 2H).

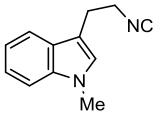
5-chloro-3-(2-isocyanoethyl)-1H-indole (9c)



5-chlorotryptamine hydrochloric acid (2.8 g, 12.1 mmol) was mixed with ethyl formate (15 ml) and potassium carbonate (1.67 g, 12.1 mmol) and heated to reflux for 16 h. Water was added to the mixture and the product was extracted with ethyl acetate. After washing with brine, drying over sodium sulfate and concentrating *in vacuo*, the crude formamide was directly used without additional purification in the isocyanide synthesis. Following procedure A the isocyanide could be obtained using crude formamide, triethylamine (8.43 mL, 60.5 mmol) and phosphoryl chloride (1.69 mL, 18.2 mmol) in dichloromethane (24 ml). The title compound was isolated as a brownish solid (0.52 g, 2.54 mmol, 21%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.51 (d, *J* = 1.8 Hz, 1H), 7.31 (d, *J* = 8.6 Hz, 1H), 7.23 – 7.13 (m, 2H), 3.66 (tt, *J* = 6.9, 1.8 Hz, 2H), 3.12 (td, *J* = 6.9, 3.7 Hz, 2H).

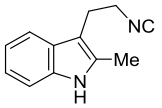
3-(2-isocyanoethyl)-1-methyl-1*H*-indole (9d)



According to procedure A the formamide was prepared using 1-methyltryptamine (1.23 g, 7 mmol) and ethyl formate (10 ml). Subsequently, the isocyanide could be obtained using crude formamide, triethylamine (4.9 ml, 35 mmol) and phosphoryl chloride (0.98 mL, 10.5 mmol) in dichloromethane (14 ml). The title compound was isolated as a colorless oil (0.38 g, 2.06 mmol, 29%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.9 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.01 (s, 1H), 3.78 (s, 3H), 3.65 (tt, *J* = 7.1, 1.8 Hz, 2H), 3.16 (tt, *J* = 7.0, 3.8, 1.5 Hz, 2H).

3-(2-isocyanoethyl)-2-methyl-1*H*-indole (9e)



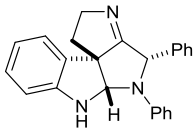
According to procedure A the formamide was prepared using 2-methyltryptamine (1.66 g, 9.5 mmol) and ethyl formate (13 ml). Subsequently, the isocyanide could be obtained using crude formamide, triethylamine (6.63 ml, 47.6 mmol) and phosphoryl chloride (1.33 mL, 14.3 mmol) in dichloromethane (14 ml). The title compound was isolated as a colorless oil (1.26 g, 6.84 mmol, 72%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 7.9 Hz, 1H), 7.17 – 7.13 (m, 1H), 7.12 – 7.08 (m, 1H), 3.59 (tt, *J* = 7.2, 1.8 Hz, 2H), 3.13 (tt, *J* = 7.1, 2.0 Hz, 2H), 2.45 (s, 3H).

General procedure B: interrupted Ugi reaction to form spiroindolines.

To a solution of isocyanide **1** (1.0 equiv) in TFE (0.1M) was added amine (1.1 equiv) and aldehyde (1.1 equiv). The reaction mixture was stirred at room temperature for 16 – 48 h (isocyanide consumption monitored with TLC). Afterwards the reaction was concentrated *in vacuo*. Purification by silica gel chromatography afforded the desired product with an eluent system of either mixtures of MeOH/CH_2Cl_2 or cyclohexane/EtOAc.

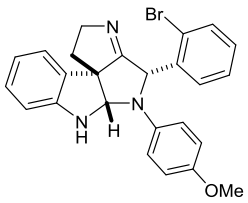
4,5-diphenyl-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8aa)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), aniline (100 µL, 1.1 mmol) and benzaldehyde (112 µL, 1.1 mmol) according to general procedure B within 48 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.31 in cyclohexane/EtOAc 4:1). Isolated as yellow foamy solids (155 mg, 0.44 mmol, 44%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24 (d, *J* = 7.6 Hz, 2H), 7.02 (dd, *J* = 12.5, 5.4 Hz, 6H), 6.81 (t, *J* = 7.3 Hz, 1H), 6.69 – 6.61 (m, 4H), 6.51 (t, *J* = 7.4 Hz, 1H), 5.35 (s, 1H), 5.25 (s, 1H), 5.10 (s, 1H), 4.42 – 4.29 (m, 2H), 2.49 – 2.41 (m, 1H), 2.33 (dd, *J* = 12.3, 5.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.8 (C_q), 148.8 (C_q), 145.2 (C_q), 137.7 (C_q), 130.4 (CH), 129.9 (CH), 129.0 (CH), 128.3 (CH), 127.2 (CH), 125.4 (CH), 123.4 (CH), 120.3 (CH), 118.3 (CH), 112.3 (CH), 110.6 (CH), 79.9 (CH), 67.6 (C_q), 65.8 (CH_2), 64.3 (CH), 38.7 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 2923 (s), 2354 (s), 1678 (s), 1593 (m), 1500 (m), 1483 (m), 1468 (m), 1313 (s), 740 (l), 725 (l); HRMS (ESI): *m/z* calculated for $C_{24}H_{22}N_3$ $[M+H]^+$ 352.1808, found: 352.1798.

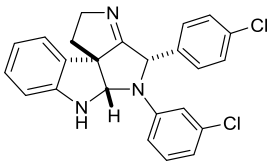
4-(2-bromophenyl)-5-(4-methoxyphenyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ab)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), *p*-anisidine (135 µL, 1.1 mmol) and 2-bromobenzaldehyde (148 µL, 1.1 mmol) according to general procedure B within 48 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.28 in cyclohexane/EtOAc 4:1). Isolated as a brown oil (170 mg, 0.37 mmol, 37%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 6.92 (t, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 9.0 Hz, 2H), 6.76 – 6.72 (m, 2H), 6.67 (d, *J* = 8.8 Hz, 1H), 6.65 – 6.56 (m, 1H), 6.48 (t, *J* = 8.5 Hz, 3H), 5.68 (s, 1H), 5.29 (s, 1H), 5.10 (s, 1H), 4.40 (d, *J* = 7.1 Hz, 2H), 3.72 (s, 3H), 2.51 – 2.39 (m, 1H), 2.31 (dd, *J* = 12.8, 5.4 Hz, 1H); ^{13}C-NMR (126 MHz, $CDCl_3$): δ 181.8 (C_q), 152.6 (C_q), 148.9 (C_q), 139.0 (C_q), 136.6 (C_q), 133.2 (CH), 130.7 (C_q), 129.2 (CH), 128.9 (CH), 127.9 (CH), 127.2 (CH), 123.7 (CH), 122.4 (CH), 120.5 (C_q), 115.4 (CH), 113.0 (CH), 110.7 (CH), 80.0 (CH), 68.3 (C_q), 65.9 (CH_2), 64.3 (CH), 55.8 (CH_3), 38.9 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 2923 (s), 2354 (s), 1678 (s), 1593 (m), 1500 (m), 1483 (m), 1468 (m), 1313 (s), 740 (l), 725 (l); HRMS (ESI): *m/z* calculated for $C_{25}H_{23}BrN_3O$ $[M+H]^+$ 460.1019, found: 460.0937.

5-(3-chlorophenyl)-4-(4-chlorophenyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo [2,3-b]indole (8ac)

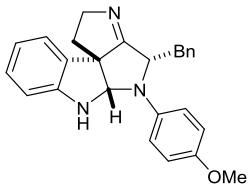


Prepared from isocyanide **1a** (170 mg, 1.0 mmol), 3-chloroaniline (116 µL, 1.1 mmol) and 4-chlorobenzaldehyde (155 mg, 1.1 mmol) according to general procedure B in HFIP instead of TFE (reaction was finished within 1.5 h). Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.61 in cyclohexane/EtOAc 4:1). Isolated as yellow foamy solids (105 mg, 0.25 mmol, 25%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (t, *J* = 8.1 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 6.78 (d, *J* = 7.9 Hz, 1H), 6.70 (d, *J* = 7.8 Hz, 1H), 6.66 (d, *J* = 7.5 Hz, 1H), 6.63 (s, 1H), 6.56 (t,

J = 7.4 Hz, 1H), 6.40 (d, *J* = 8.3 Hz, 1H), 5.31 (s, 1H), 5.18 (s, 1H), 5.14 (s, 1H), 4.42 – 4.29 (m, 2H), 2.49 – 2.40 (m, 1H), 2.33 (dd, *J* = 12.6, 5.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.0 (C_q), 148.4 (C_q), 146.2 (C_q), 135.8 (C_q), 135.6 (C_q), 133.1 (CH), 130.9 (C_q), 130.1 (CH), 129.2 (CH), 128.6 (CH), 126.8 (CH), 123.3 (CH), 120.7 (CH), 118.5 (CH), 112.1 (CH), 110.9 (CH), 110.7 (CH), 80.0 (CH), 67.7 (C_q), 65.7 (CH_2), 63.6 (CH), 38.7 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 2930 (s), 2341 (s), 1593 (l), 1485 (l), 1406 (m), 1265 (s), 1092 (s), 1026 (s), 908 (s), 729 (l); HRMS (ESI): *m/z* calculated for $C_{24}H_{20}Cl_2N_3$ $[M+H]^+$ 420.1029, found: 420.1011.

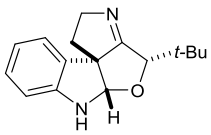
4-benzyl-5-(4-methoxyphenyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ad)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), *p*-anisidine (135 µL, 1.1 mmol) and phenylacetaldehyde (128 µL, 1.1 mmol) according to general procedure B within 24 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.35 in cyclohexane/EtOAc 4:1). Isolated as a brown oil (249 mg, 0.63 mmol, 63%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.2 Hz, 2H), 7.23 – 7.14 (m, 3H), 7.07 (d, *J* = 7.3 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.88 (t, *J* = 7.4 Hz, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 6.67 (d, *J* = 8.9 Hz, 2H), 5.18 (s, 1H), 4.88 (s, 1H), 4.44 – 4.32 (m, 2H), 4.26 (dd, *J* = 14.6, 7.8 Hz, 1H), 3.79 (s, 3H), 3.02 (dd, *J* = 13.7, 3.9 Hz, 1H), 2.43 – 2.33 (m, 2H), 2.10 – 1.98 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 181.9 (C_q), 152.0 (C_q), 148.9 (C_q), 138.8 (C_q), 137.3 (C_q), 131.1 (C_q), 129.3 (CH), 129.2 (CH), 128.6 (CH), 126.8 (CH), 123.1 (CH), 120.3 (CH), 115.7 (CH), 111.9 (CH), 111.1 (CH), 79.9 (CH), 67.8 (C_q), 65.8 (CH_2), 60.7 (CH), 56.0 (CH_3), 39.2 (CH_2), 38.9 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 1678 (m), 1605 (s), 1508 (l), 1483 (m), 1466 (m), 1236 (m), 1036 (m), 812 (m), 740 (l), 698 (m); HRMS (ESI^+) calculated $C_{26}H_{26}N_3O$ $[M+H]^+$ 396.2070, found 396.2068.

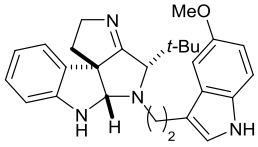
4-(tert-butyl)-2,4,5a,6-tetrahydro-1H-pyrrolo[3',2':3,4]furo[2,3-b]indole (11ae)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol) and pivaldehyde (119 µL, 1.1 mmol) and 6-amino-2-picoline (119 mg, 1.1 mmol) according to general procedure B within 24 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 6:1, R_f = 0.65 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (95 mg, 0.37 mmol, 37%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (t, *J* = 8.3 Hz, 1H), 7.08 (d, *J* = 7.4 Hz, 1H), 6.78 (t, *J* = 7.8 Hz, 1H), 6.70 (d, *J* = 7.8 Hz, 1H), 5.25 (s, 1H), 4.93 (s, 1H), 4.57 – 4.43 (m, 2H), 3.88 (d, *J* = 3.3 Hz, 1H), 2.31 – 2.24 (m, 1H), 2.20 (dd, *J* = 12.6, 5.8 Hz, 1H), 1.03 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.3 (C_q), 148.2 (C_q), 130.1 (C_q), 128.9 (CH), 123.0 (CH), 119.7 (CH), 109.7 (CH), 93.2 (CH), 80.9 (CH), 70.5 (C_q), 67.6 (CH_2), 36.5 (CH_2), 34.0 (C_q), 25.5 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 3340 (m), 2955 (s), 1670 (m), 1608 (m), 1470 (m), 1254 (s), 1178 (s), 970 (m), 945 (m), 864 (m), 742 (l); HRMS (ESI): *m/z* calculated for $C_{16}H_{21}N_2O$ $[M+H]^+$ 257.1648, found 257.1636.

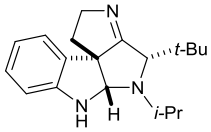
4-(tert-butyl)-5-(2-(5-methoxy-1H-indol-3-yl)ethyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo [2,3-b]indole (8af)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), 5-methoxytryptamine (209 mg, 1.1 mmol) and pivaldehyde (119 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.20 in cyclohexane/EtOAc 4:1). Isolated as a light brown oil (304 mg, 0.71 mmol, 71%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.20 – 7.12 (m, 2H), 7.10 (d, *J* = 7.6 Hz, 2H), 6.96 (dd, *J* = 8.8, 2.3 Hz, 1H), 6.81 (t, *J* = 7.4 Hz, 1H), 6.67 (d, *J* = 7.8 Hz, 1H), 4.59 – 4.45 (m, 1H), 4.43 – 4.34 (m, 2H), 3.93 (s, 3H), 3.30 – 3.24 (m, 2H), 3.16 – 3.07 (m, 2H), 2.46 – 2.35 (m, 1H), 2.31 (dd, *J* = 12.3, 5.4 Hz, 1H), 0.81 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.7 (C_q), 154.0 (C_q), 149.1 (C_q), 131.2 (C_q), 128.5 (CH), 127.6 (C_q), 123.6 (CH), 122.6 (CH), 119.4 (CH), 113.3 (CH), 112.4 (C_q), 112.0 (CH), 110.4 (CH), 100.2 (CH), 86.6 (CH), 74.0 (CH), 69.1 (C_q), 66.0 (CH_2), 58.3 (CH_2), 55.8 (CH_3), 41.5 (CH_2), 35.4 (C_q), 29.8 (C_q), 27.1 (CH_3), 25.2 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 2926 (m), 1655 (m), 1605 (m), 1483 (m), 1464 (m), 1213 (m), 1103 (s), 1094 (s), 1026 (m), 740 (l); HRMS (ESI): *m/z* calculated for $C_{27}H_{33}N_4O$ $[M+H]^+$ 429.2649, found 429.2643.

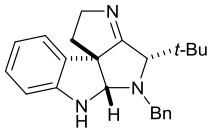
4-(tert-butyl)-5-isopropyl-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ag)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), isopropylamine (95 µL, 1.1 mmol) and pivaldehyde (119 µL, 1.1 mmol) according to general procedure B within 16 h. Evaporation of the solvent and excess starting materials afforded the compound as an orange solid (294 mg, 0.99 mmol, 99%). No further purification was required.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.06 (t, *J* = 7.7 Hz, 1H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.70 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 7.7 Hz, 1H), 4.53 (s, 1H), 4.45 – 4.34 (m, 1H), 4.26 (dd, *J* = 14.5, 7.7 Hz, 1H), 3.24 (s, 1H), 3.17 – 3.04 (m, 1H), 2.29 (d, *J* = 5.2 Hz, 2H), 1.24 (d, *J* = 6.8 Hz, 3H), 1.00 (d, *J* = 6.3 Hz, 3H), 0.63 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 188.2 (C_q), 149.8 (C_q), 131.2 (C_q), 128.8 (CH), 123.5 (CH), 119.4 (CH), 110.2 (CH), 77.6 (CH), 72.2 (CH), 68.8 (C_q), 66.1 (CH_2), 53.2 (CH), 41.8 (CH_2), 35.3 (C_q), 27.0 (CH_3), 23.4 (CH_3), 15.3 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 3275 (s), 2962 (s), 1653 (m), 1605 (m), 1460 (m), 1263 (s), 1164 (m), 1045 (s), 966 (m), 854 (s), 737 (l); HRMS (ESI): *m/z* calculated for $C_{19}H_{28}N_3$ $[M+H]^+$ 298.2278, found: 298.2267

5-benzyl-4-(tert-butyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ah)

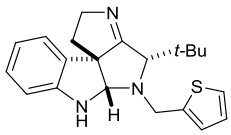


Prepared from isocyanide **1a** (170 mg, 1.0 mmol), benzylamine (120 µL, 1.1 mmol) and pivaldehyde (119 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.43 in cyclohexane/EtOAc 4:1). Isolated as a yellow solid (307 mg, 0.89 mmol, 89%).

m.p.: 140-144 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.2 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.99 (d, *J* = 7.3 Hz, 1H), 6.69 (t, *J* = 7.2 Hz, 1H), 6.44 (d, *J* = 7.8 Hz, 1H), 4.47 – 4.38 (m, 1H), 4.32 (dd, *J* = 14.8, 8.4 Hz, 1H), 4.26 – 4.18 (m, 2H), 3.85 (d, *J* = 13.4 Hz, 1H), 3.27 (s, 1H), 2.37 – 2.29 (m, 1H), 2.16 (dd, *J* = 12.3, 5.3 Hz, 1H), 0.80 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.6

(C_q), 148.8 (C_q), 139.7 (C_q), 130.8 (C_q), 128.8 (CH), 128.8 (CH), 128.4 (CH), 127.7 (CH), 123.7 (CH), 119.0 (CH), 110.0 (CH), 87.7 (CH), 73.5 (CH), 69.1 (C_q), 65.9 (CH_2), 62.1 (CH_2), 41.0 (CH_2), 35.7 (C_q), 27.2 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2951 (s), 2866 (s), 1657 (m), 1605 (m), 1479 (s), 1464 (m), 1086 (s), 1049 (s), 926 (s), 752, (l), 743 (l); HRMS (ESI): *m/z* calculated for $C_{23}H_{28}N_3$ $[M+H]^+$ 346.2278, found: 346.2266.

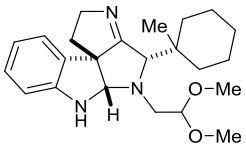
(4S,5aS,10bS)-4-(tert-butyl)-5-(thiophen-2-ylmethyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo [2,3-b]indole (8ai)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), 2-thiophenemethylamine (112 µL, 1.1 mmol) and pivaldehyde (120 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.13 in cyclohexane/EtOAc 5:1). Isolated as a yellow oil (246 mg, 0.70 mmol, 70%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 5.0 Hz, 1H), 7.07 – 7.01 (m, 2H), 7.01 – 6.97 (m, 2H), 6.70 (t, *J* = 7.4 Hz, 1H), 6.51 (d, *J* = 7.8 Hz, 1H), 4.46 – 4.36 (m, 1H), 4.35 – 4.27 (m, 2H), 4.25 – 4.14 (m, 2H), 3.94 (s, 1H), 3.28 (s, 1H), 2.38 – 2.26 (m, 1H), 2.18 (dd, *J* = 12.3, 5.4 Hz, 1H), 0.78 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.35 (C_q), 149.02 (C_q), 143.13 (C_q), 130.90 (C_q), 128.57 (CH), 126.91 (CH), 126.13 (CH), 125.65 (CH), 123.67 (CH), 119.24 (CH), 110.27 (CH), 87.40 (CH), 73.14 (CH), 69.17 (C_q), 66.09 (CH_2), 56.23 (CH_2), 41.18 (CH_2), 35.65 (C_q), 27.17 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2951 (m), 2868 (s), 1736 (m), 1655 (m), 1605 (m), 1481 (m), 1464 (l), 1393 (s), 1310 (s), 1240 (m), 1090 (m), 1047 (m), 1026 (m), 1007 (m), 852 (m), 802 (l), 740 (l); HRMS (ESI): *m/z* calculated for $C_{21}H_{26}N_3S$ $[M+H]^+$ 352.1842, found: 352.1845

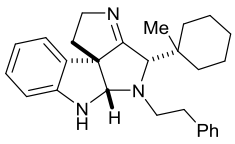
(4S,5aS,10bS)-5-(2,2-dimethoxyethyl)-4-(1-methylcyclohexyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4] pyrrolo[2,3-b]indole (8aj)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), aminoacetaldehyde dimethyl acetal (120 µL, 1.1 mmol) and 1-methylcyclohexane-1-carboxaldehyde (139 mg, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 3:1, R_f = 0.44 in cyclohexane/EtOAc 2:1). Isolated as a yellow oil (281 mg, 0.73 mmol, 73%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.02 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 7.3 Hz, 1H), 6.64 (t, *J* = 7.3 Hz, 1H), 6.58 (d, *J* = 7.7 Hz, 1H), 5.15 (s, 1H), 4.50 (d, *J* = 5.2 Hz, 1H), 4.44 – 4.30 (m, 1H), 4.26 (dd, *J* = 14.7, 8.3 Hz, 1H), 4.14 (s, 1H), 3.44 (s, 3H), 3.34 (s, 3H), 3.18 (s, 1H), 3.12 (dd, *J* = 14.2, 7.3 Hz, 1H), 2.89 (d, *J* = 14.0 Hz, 1H), 2.29 (q, *J* = 10.8, 10.2 Hz, 1H), 2.18 (dd, *J* = 12.1, 5.2 Hz, 1H), 1.51 – 1.36 (m, 2H), 1.36 – 1.29 (m, 1H), 1.28 – 0.97 (m, 7H), 0.45 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.48 (C_q), 149.30 (C_q), 131.00 (C_q), 128.47 (CH), 123.49 (CH), 118.73 (CH), 109.88 (CH), 104.69 (CH), 88.84 (CH), 76.54 (CH), 69.14 (C_q), 66.00 (CH_2), 60.40 (CH_2), 53.59 (CH_3), 53.12 (CH_3), 41.26 (CH_2), 38.12 (C_q), 35.15 (CH_2), 34.79 (CH_2), 26.15 (CH_2), 21.73 (CH_2), 21.66 (CH_2), 19.28 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2926 (m), 2860 (s), 1655 (m), 1604 (m), 1466 (m), 1310 (s), 1267 (s), 1190 (s), 1115 (m), 1057 (m), 964 (m), 947 (m), 858 (s); HRMS (ESI): *m/z* calculated for $C_{23}H_{34}N_3O_2$ $[M+H]^+$ 384.2646, found: 384.2655

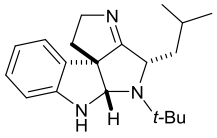
(4S,5aS,10bS)-4-(1-methylcyclohexyl)-5-phenethyl-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ak)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), phenethylamine (119 µL, 1.1 mmol) and 1-methylcyclohexane-1-carboxaldehyde (139 mg, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.40 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (332 mg, 0.83 mmol, 83%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.26 – 7.20 (m, 3H), 7.08 (t, *J* = 7.6 Hz, 1H), 7.00 (d, *J* = 7.3 Hz, 1H), 6.73 (t, *J* = 7.4 Hz, 1H), 6.62 (d, *J* = 7.8 Hz, 1H), 4.48 – 4.36 (m, 1H), 4.35 – 4.26 (m, 2H), 4.16 (s, 1H), 3.29 (s, 1H), 3.16 (dt, *J* = 13.2, 7.8 Hz, 1H), 3.12 – 3.03 (m, 1H), 2.91 (t, *J* = 8.0 Hz, 2H), 2.31 (ddd, *J* = 12.3, 10.7, 8.1 Hz, 1H), 2.24 (dd, *J* = 12.2, 5.5 Hz, 1H), 1.54 – 1.40 (m, 2H), 1.40 – 1.19 (m, 5H), 1.19 – 0.99 (m, 3H), 0.52 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.67 (C_q), 149.15 (C_q), 139.60 (C_q), 131.17 (C_q), 128.80 (CH), 128.63 (CH), 128.58 (CH), 126.39 (CH), 119.46 (CH), 110.36 (CH), 86.62 (CH), 74.65 (CH), 69.18 (C_q), 66.06 (CH_2), 59.92 (CH_2), 41.73 (CH_2), 38.19 (C_q), 35.60 (CH_2), 35.14 (CH_2), 34.69 (CH_2), 26.19 (CH_2), 21.83 (CH_2), 21.73 (CH_2), 19.63 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2926 (l), 2860 (m), 2363 (l), 1654 (m), 1605 (m), 1483 (m), 1466 (m), 1259 (s), 1111 (m), 1094 (s), 964 (m), 698 (m); HRMS (ESI): *m/z* calculated for $C_{27}H_{34}N_3$ $[M+H]^+$ 400.2747, found: 400.2762

N-(tert-butyl)-1-(4,9-dihydro-3H-pyrido[3,4-b]indol-1-yl)-3-methylbutan-1-amine (8al)

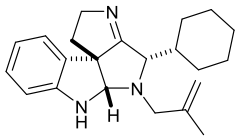


Prepared from isocyanide **1a** (170 mg, 1.0 mmol), *tert*-butylamine (116 µL, 1.1 mmol) and isovaleraldehyde (118 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.29 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (125 mg, 0.40 mmol, 40%).

^{1}H NMR (500 MHz, DMSO-d_6) δ 6.98 (t, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 7.3 Hz, 1H), 6.56 (d, *J* = 7.8 Hz, 1H), 6.53 (t, *J* = 7.3 Hz, 1H), 4.49 (s, 1H), 4.29 – 4.20 (m, 1H), 4.10 (dd, *J* = 14.6, 8.5 Hz, 1H), 3.51 (dd, *J* = 11.4, 4.9 Hz, 1H), 2.25 – 2.15 (m, 1H), 1.99 (dd, *J* = 12.4, 5.9 Hz, 1H), 1.49 – 1.37 (m, 1H), 1.05 (s, 9H), 0.98 (dq, *J* = 14.7, 5.0, 4.6 Hz, 1H), 0.83 (d, *J* = 6.5 Hz, 3H), 0.74 – 0.66 (m, 1H), 0.52 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 185.4 (C_q), 150.0 (C_q), 131.3 (C_q), 128.1 (CH), 122.0 (CH), 117.2 (CH), 109.2 (CH), 79.1 (CH), 67.3 (C_q), 65.3 (CH_2), 55.7 (CH), 54.1 (CH_2), 47.1 (CH_2), 27.6 (CH_3), 24.6 (CH), 23.7 (CH_3), 21.4 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2359 (s), 1682 (s), 1593 (l), 1560 (s), 1485 (s), 1329 (s), 1090 (m), 1015 (m), 908 (s), 731 (m); HRMS (ESI): *m/z* calculated for $C_{20}H_{30}N_3$ $[M+H]^+$ 312.2434, found: 312.2429.

Note: multiple peaks were not present in the ^{1}H and ^{13}C NMR spectrums or showed unclear splitting patterns when taken in $CDCl_3$.

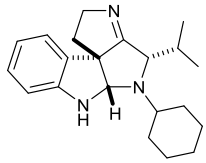
4-cyclohexyl-5-(2-methylallyl)-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8am)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), 2-methylallylamine (100 µL, 1.1 mmol) and cyclohexanecarboxaldehyde (133 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 5:1, R_f =0.53 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (124 mg, 0.37 mmol, 37%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (t, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 7.4 Hz, 1H), 6.72 (t, *J* = 7.3 Hz, 1H), 6.64 (d, *J* = 7.5 Hz, 1H), 4.98 (s, 1H), 4.88 (s, 1H), 4.44 – 4.33 (m, 1H), 4.29 (dd, *J* = 14.7, 8.5 Hz, 1H), 4.17 (s, 1H), 3.33 (d, *J* = 13.6 Hz, 1H), 3.24 (d, *J* = 13.5 Hz, 1H), 3.07 (d, *J* = 4.8 Hz, 1H), 2.29 (q, *J* = 11.8 Hz, 1H), 2.13 (dd, *J* = 12.4, 5.5 Hz, 1H), 1.77 (s, 3H), 1.73 (d, *J* = 10.8 Hz, 1H), 1.65 (s, 1H), 1.58 – 1.41 (m, 2H), 1.32 (d, *J* = 12.7 Hz, 1H), 1.17 – 0.95 (m, 4H), 0.85 (q, *J* = 12.5 Hz, 1H), 0.63 (q, *J* = 11.2 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.9 (C_q), 149.0 (C_q), 144.9 (C_q), 131.4 (C_q), 128.4 (CH), 123.1 (CH), 119.2 (CH), 113.2 (C_q), 110.2 (CH), 86.4 (CH), 69.3 (CH), 69.0 (C_q), 66.0 (CH_2), 62.3 (CH_2), 41.9 (CH), 39.2 (CH_2), 30.2 (CH_2), 28.9 (CH_2), 26.4 (CH_2), 26.3 (CH_2), 26.1 (CH_2), 20.7 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2924 (m), 2851 (s), 1664 (s), 1605 (m), 1481 (m), 1464 (m), 1447 (m), 1097 (s), 964 (m), 897 (m), 738 (l); HRMS (ESI): *m/z* calculated for $C_{22}H_{30}N_3$ $[M+H]^+$ 336.2434, found: 336.2428

(4S,5aS,10bS)-5-cyclohexyl-4-isopropyl-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8an)

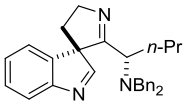


Prepared from isocyanide **1a** (170 mg, 1.0 mmol), cyclohexylamine (126 µL, 1.1 mmol) and isobutyraldehyde (99 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 5:1, R_f = 0.21 in cyclohexane/EtOAc 5:1). Isolated as a yellow solid (155 mg, 0.48 mmol, 48%).

m.p.: 122-126 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 6.97 – 6.90 (m, 2H), 6.49 (t, *J* = 7.5 Hz, 2H), 5.85 (s, 1H), 4.50 (s, 1H), 4.28 (ddd, *J* = 15.4, 10.3, 6.0 Hz, 1H), 4.11 (dd, *J* = 14.6, 8.4 Hz, 1H), 2.91 (d, *J* = 9.3 Hz, 1H), 2.56 (t, *J* = 10.7 Hz, 1H), 2.20 (ddd, *J* = 11.9, 10.2, 8.8 Hz, 1H), 2.02 (dd, *J* = 12.4, 5.7 Hz, 1H), 1.82 (d, *J* = 11.5 Hz, 1H), 1.78 – 1.68 (m, 2H), 1.68 – 1.60 (m, 1H), 1.56 (d, *J* = 11.9 Hz, 1H), 1.32 – 0.98 (m, 6H), 0.92 – 0.81 (m, 1H), 0.79 (d, *J* = 6.4 Hz, 3H), 0.55 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (126 MHz, DMSO-d_6) δ 186.19 (C_q), 150.37 (C_q), 131.35 (C_q), 128.58 (CH), 122.49 (CH), 117.45 (CH), 108.99 (CH), 79.91 (CH), 67.70 (C_q), 67.12 (CH), 65.89 (CH_2), 62.45 (CH), 40.01 (CH_2), 33.47 (CH), 32.19 (CH_2), 29.71 (CH_2), 26.45 (CH_2), 26.03 (CH_2), 25.99 (CH_2), 20.09 (CH_3), 19.06 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2922 (m), 2849 (m), 2336 (m), 1664 (m), 1605 (m), 1483 (m), 1466 (m), 1265 (s), 1053 (m), 1041 (m), 964 (m), 708 (m); HRMS (ESI): *m/z* calculated for $C_{21}H_{30}N_3$ $[M+H]^+$ 324.2434, found: 324.2436

Note: multiple peaks were not present in the ^{1}H and ^{13}C NMR spectrums or showed unclear splitting patterns when taken in $CDCl_3$.

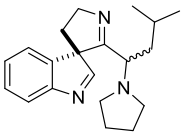
N,N-dibenzyl-1-(4',5'-dihydrospiro[indole-3,3'-pyrrol]-2'-yl)butan-1-amine (12aa)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), dibenzylamine (212 µL, 1.1 mmol) and valeraldehyde (99 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 6:1, R_f = 0.60 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (278 mg, 0.66 mmol, 66%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.55 (d, *J* = 7.7 Hz, 1H), 7.33 – 7.25 (m, 6H), 7.21 (t, *J* = 7.2 Hz, 6H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.04 (d, *J* = 7.4 Hz, 1H), 4.31 – 4.23 (m, 1H), 4.20 – 4.12 (m, 1H), 3.88 (d, *J* = 13.8 Hz, 2H), 3.39 (d, *J* = 13.8 Hz, 2H), 2.55 (d, *J* = 10.6 Hz, 1H), 2.43 – 2.35 (m, 1H), 2.14 – 2.07 (m, 1H), 2.02 – 1.85 (m, 2H), 1.48 – 1.40 (m, 1H), 1.30 – 1.16 (m, 1H), 0.72 – 0.65 (m, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4 (C_q), 173.0 (CH), 155.5 (C_q), 139.2 (C_q), 138.8 (C_q), 129.0 (CH), 128.3 (CH), 128.2 (CH), 126.8 (CH), 126.1 (CH), 121.5 (CH), 121.2 (CH), 74.0 (C_q), 60.5 (CH_2), 57.3 (CH), 54.1 (CH_2), 31.8 (CH_2), 25.0 (CH_2), 19.6 (CH_2), 13.9 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2954 (s), 1634 (s), 1545 (s), 1495 (s), 1452 (m), 1375 (s), 1124 (m), 995 (s), 908 (s), 744 (l), 698 (l); HRMS (ESI): *m/z* calculated for $C_{29}H_{32}N_3$ $[M+H]^+$ 422.2591, found: 422.2587.

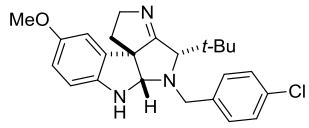
2'-(3-methyl-1-(pyrrolidin-1-yl)butyl)-4',5'-dihydrospiro[indole-3,3'-pyrrole] (12ab)



Prepared from isocyanide **1a** (170 mg, 1.0 mmol), pyrrolidine (92 µL, 1.1 mmol) and aldehyde isovaleraldehyde (118 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (4% MeOH in CH_2Cl_2, R_f = 0.19 in cyclohexane/EtOAc 4:1. Isolated as a brown oil (279 mg, 0.90 mmol, 90%) as a mixture of diastereoisomers (2.6:1 based on ^{1}H NMR).

Major diastereoisomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.23 (d, *J* = 7.3 Hz, 1H), 7.21 – 7.14 (m, 1H), 4.30 – 4.18 (m, 2H), 2.97 (dd, *J* = 10.1, 4.7 Hz, 1H), 2.64 – 2.49 (m, 1H), 2.48 – 2.28 (m, 4H), 2.03 – 1.97 (m, 1H), 1.68 – 1.54 (m, 4H), 1.31 – 1.24 (m, 1H), 1.07 (ddd, *J* = 13.9, 9.9, 5.1 Hz, 1H), 0.79 (d, *J* = 6.5 Hz, 3H), 0.69 (ddd, *J* = 13.9, 10.5, 3.9 Hz, 1H), 0.37 (d, *J* = 6.6 Hz, 3H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 178.5 (C_q), 173.7 (CH), 155.0 (C_q), 139.7 (C_q), 128.3 (CH), 126.0 (CH), 121.9 (CH), 121.1 (CH), 72.6 (C_q), 63.5 (CH), 60.1 (CH_2), 50.8 (CH_2), 39.0 (CH_2), 33.5 (CH_2), 24.8 (CH), 23.3 (CH_2), 23.2 (CH_3), 22.2 (CH_3); ***Minor diastereoisomer***: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.21 – 7.13 (m, 2H), 4.28 – 4.19 (m, 2H), 2.64 – 2.49 (m, 2H), 2.47 – 2.29 (m, 4H), 2.20 – 2.12 (m, 1H), 1.77 (ddd, *J* = 14.4, 9.6, 5.1 Hz, 1H), 1.69 – 1.55 (m, 4H), 1.40 – 1.30 (m, 1H), 1.13 (ddd, *J* = 13.5, 9.0, 4.4 Hz, 1H), 0.75 (d, *J* = 6.6 Hz, 3H), 0.43 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.0 (C_q), 173.3 (CH), 155.9 (C_q), 139.7 (C_q), 128.4 (CH), 126.0 (CH), 121.7 (CH), 120.8 (CH), 73.8 (C_q), 61.0 (CH_2), 54.7 (CH), 47.4 (CH_2), 33.6 (CH_2), 31.1 (CH_2), 24.9 (CH), 23.9 (CH_2), 23.4 (CH_3), 21.6 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2947 (m), 1629 (m), 1541 (m), 1448 (m), 1113 (s), 986 (m), 879 (m), 750 (l). HRMS (ESI): *m/z* calculated for $C_{20}H_{28}N_3$ $[M+H]^+$ 310.2278, found: 310.2265.

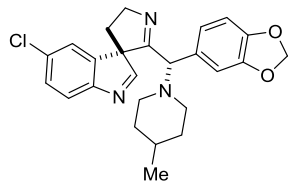
4-(tert-butyl)-5-(4-chlorobenzyl)-9-methoxy-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8ba)



Prepared from isocyanide **1b** (200 mg, 1.0 mmol), 4-chlorobenzylamine (134 µL, 1.1 mmol) and pivaldehyde (119 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.26 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (385 mg, 0.94 mmol, 94%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (q, *J* = 8.3 Hz, 4H), 6.64 (d, *J* = 8.1 Hz, 1H), 6.60 (s, 1H), 6.42 (d, *J* = 8.4 Hz, 1H), 4.44 – 4.35 (m, 1H), 4.32 (dd, *J* = 14.9, 8.4 Hz, 1H), 4.20 (s, 1H), 4.16 (d, *J* = 13.9 Hz, 1H), 3.86 (d, *J* = 14.8 Hz, 1H), 3.72 (s, 3H), 3.25 (s, 1H), 2.32 (q, *J* = 11.2 Hz, 1H), 2.17 (dd, *J* = 12.3, 5.4 Hz, 1H), 0.81 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 187.2 (C_q), 153.9 (C_q), 143.0 (C_q), 138.5 (C_q), 133.5 (C_q), 132.4 (C_q), 130.1 (CH), 129.1 (CH), 113.6 (CH), 110.7 (CH), 110.7 (CH), 88.7 (CH), 73.8 (CH), 69.7 (C_q), 66.2 (CH_2), 61.6 (CH_2), 56.2 (CH_3), 41.0 (CH_2), 35.9 (C_q), 27.4 (CH_3); IR (neat): ν_{max} cm^{-1}) = 2951 (m), 1647 (m), 1488 (l), 1464 (s), 1448 (m), 1435 (s), 1288 (m), 1205 (m), 1090 (m), 1007 (s), 937 (m), 851 (l), 636 (s); HRMS (ESI): *m/z* calculated for $C_{24}H_{29}ClN_3O$ $[M+H]^+$ 410.1994, found: 410.1983.

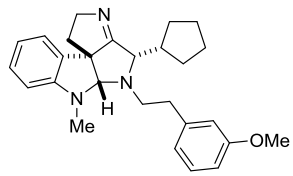
2'-(benzo[d][1,3]dioxol-5-yl(4-methylpiperidin-1-yl)methyl)-5-chloro-4',5'-dihydrospiro[indole-3,3'-pyrrole] (12ca)



Prepared from isocyanide **1c** (205 mg, 1.0 mmol), 4-methylpiperidine (130 µL, 1.1 mmol) and piperonal (165 mg, 1.1 mmol) according to general procedure B within 16 h. Purification: column chromatography on silicagel (4% MeOH in CH_2Cl_2, R_f = 0.30 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (192 mg, 0.44 mmol, 44%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.51 (d, *J* = 10.1 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 6.91 (s, 1H), 6.59 (s, 1H), 6.54 (d, *J* = 7.9 Hz, 1H), 6.45 (d, *J* = 7.8 Hz, 1H), 5.87 (d, *J* = 13.1 Hz, 2H), 4.31 – 4.15 (m, 2H), 3.35 (s, 1H), 2.93 (d, *J* = 10.9 Hz, 1H), 2.58 (d, *J* = 11.2 Hz, 1H), 2.50 – 2.41 (m, 1H), 2.00 – 1.94 (m, 1H), 1.72 (t, *J* = 11.0 Hz, 1H), 1.59 – 1.51 (m, 2H), 1.40 (d, *J* = 12.1 Hz, 1H), 1.34 – 1.21 (m, 2H), 1.08 (q, *J* = 11.6 Hz, 1H), 0.84 (d, *J* = 3.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.7 (C_q), 173.1 (CH), 153.4 (C_q), 147.5 (C_q), 147.3 (C_q), 141.2 (C_q), 132.0 (C_q), 130.0 (CH), 128.3 (CH), 123.1 (CH), 122.1 (CH), 122.0 (CH), 108.9 (CH), 107.7 (CH), 101.0 (C_q), 73.8 (C_q), 71.5 (CH), 60.2 (CH_2), 53.0 (CH_2), 51.6 (CH_2), 33.9 (CH_2), 33.8 (CH_2), 32.3 (CH_2), 30.7 (CH), 21.8 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2962 (m), 1655 (m), 1605 (m), 1462 (s), 1263 (s), 1163 (m), 1045 (s), 906 (m), 733 (l); HRMS (ESI^+) calculated $C_{25}H_{27}ClN_3O_2$ $[M+H]^+$ 436.1786, found 436.1804.

4-cyclopentyl-5-(3-methoxyphenethyl)-6-methyl-1,2,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole (8db)

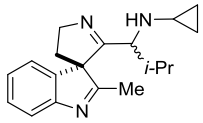


Prepared from isocyanide **1d** (184 mg, 1.0 mmol), 3-methoxyphenethylamine (160 µL, 1.1 mmol) and cyclopentanecarboxaldehyde (117 µL, 1.1 mmol) according to general procedure B within 16 h. Purification: column

chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.57 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (299 mg, 0.72 mmol, 72%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.8 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 7.2 Hz, 1H), 6.79 (d, *J* = 7.4 Hz, 1H), 6.75 (d, *J* = 8.7 Hz, 2H), 6.57 (t, *J* = 7.4 Hz, 1H), 6.34 (d, *J* = 7.8 Hz, 1H), 4.39 – 4.32 (m, 1H), 4.28 (dd, *J* = 14.7, 8.4 Hz, 1H), 3.80 (s, 3H), 3.14 (d, *J* = 8.2 Hz, 1H), 3.07 (t, *J* = 8.2 Hz, 2H), 2.87 (s, 3H), 2.80 – 2.71 (m, 2H), 2.32 (dd, *J* = 12.6, 5.7 Hz, 1H), 2.25 – 2.16 (m, 1H), 1.54 – 1.40 (m, 3H), 1.37 – 1.17 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.5 (C_q), 159.7 (C_q), 150.3 (C_q), 141.4 (C_q), 131.7 (C_q), 129.5 (CH), 128.9 (CH), 122.1 (CH), 121.2 (CH), 117.3 (CH), 114.7 (CH), 111.4 (CH), 106.0 (CH), 90.3 (CH), 67.90 (CH), 66.7 (C_q), 66.0 (CH_2), 58.5 (CH_2), 55.2 (CH_3), 44.5 (CH_3), 40.4 (CH_2), 35.5 (CH_2), 30.9 (CH_2), 30.6 (CH), 28.9 (CH_2), 25.0 (CH_2); IR (neat): ν_{max} (cm^{-1}) = 2954 (m), 2866 (s), 1665 (s), 1601 (m), 1489 (m), 1298 (s), 1258 (m), 1151 (m), 1099 (s), 1043 (s), 953 (s), 738 (m); HRMS (ESI): *m/z* calculated for $C_{27}H_{34}N_3O$ $[M+H]^+$ 416.2696, found: 416.2696.

N-(2-methyl-1-(2-methyl-4',5'-dihydrospiro[indole-3,3'-pyrrol]-2'-yl)propyl)cyclopropanamine (12ea)



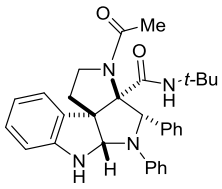
Prepared from isocyanide **1e** (184 mg, 1.0 mmol), cyclopropylamine (76 µL, 1.1 mmol) and isobutyraldehyde (100 µL, 1.1 mmol) according to general procedure B within 16 h. Evaporation of the solvent and excess starting materials afforded the compound as an orange solid. Isolated as an orange oil (292 mg, 0.99 mmol, 99%) as a mixture of diastereoisomers (1:1 based on ^{1}H NMR). Separation of the diastereoisomers was not possible, and since no other side products were formed, no further purification was required.

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.6 Hz, 2H), 7.40 – 7.29 (m, 2H), 7.19 (s, 2H), 7.15 (d, *J* = 4.4 Hz, 2H), 4.39 (dt, *J* = 12.2, 6.0 Hz, 1H), 4.29 (t, *J* = 7.0 Hz, 2H), 4.18 (dt, *J* = 15.9, 7.9 Hz, 1H), 2.53 – 2.50 (m, 1H), 2.49 – 2.42 (m, 2H), 2.41 – 2.36 (m, 2H), 2.29 (s, 3H), 2.30 – 2.25 (m, 1H), 2.23 (s, 3H), 2.10 – 2.01 (m, 3H), 1.96 – 1.89 (m, 1H), 1.31 (s, 1H), 1.01 (dd, *J* = 9.6, 3.8 Hz, 1H), 0.74 – 0.66 (m, 6H), 0.63 (d, *J* = 6.7 Hz, 3H), 0.56 (d, *J* = 6.9 Hz, 3H), 0.34 – 0.28 (m, 2H), 0.28 – 0.22 (m, 3H), 0.11 – 0.02 (m, 2H), -0.16 (s, 1H); ^{13}C-NMR (126 MHz, $CDCl_3$) δ 181.9 (C_q), 181.2 (C_q), 177.9 (C_q), 177.2 (C_q), 155.8 (C_q), 155.2 (C_q), 141.2 (C_q), 140.9 (C_q), 128.5 (CH), 128.5 (CH), 125.7 (CH), 125.5 (CH), 122.0 (CH), 121.9 (CH), 120.1 (CH), 75.3 (C_q), 62.7 (CH), 62.5 (CH), 60.2 (CH_2), 60.1 (CH_2), 34.3 (CH_2), 33.9(CH_2), 30.2 (CH), 29.8 (CH), 29.2 (CH), 29.2 (CH), 20.7 (CH_3), 20.1 (CH_3), 17.1 (CH_3), 16.8 (CH_3), 15.6 (CH_3), 15.6 (CH_3), 6.9 (CH_2), 6.7 (CH_2), 6.2 (CH_2); IR (cm^{-1}): ν_{max} = 2957 (s), 1634 (s) 1573 (s), 1456 (s), 1364 (s), 1014 (s), 906 (m), 727 (l); HRMS (ESI): *m/z* calculated for $C_{19}H_{26}N_3$ $[M+H]^+$ 296.2121, found: 296.2107.

General procedure C: Subsequent Ugi reaction to form compounds of type 8.

To a solution of **3** (1.0 equiv) in dichloromethane (0.1M) were added carboxylic acid (1.2 equiv) and isocyanide (1.2 equiv). After full depletion of the isocyanide on TLC the reaction was concentrated *in vacuo*. Purification by silica gel chromatography afforded the desired product with an eluent system of cyclohexane/EtOAc.

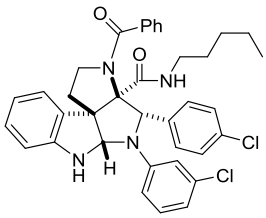
3-acetyl-N-(tert-butyl)-4,5-diphenyl-2,3,4,5,5a,6-hexahydropyrrolo[3',2':3,4]pyrrolo[2,3-b]indole-3a(1H)-carboxamide (13a)



Prepared from interrupted Ugi product **3aa** (86 mg, 0.25 mmol, 1 equiv), acetic acid (15.7 µl, 0.275 mmol, 1.1 equiv) and *tert*-butyl isocyanide (31.1 µl, 0.275 mmol, 1.1 equiv) according to general procedure B within 24 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 1:2, R_f = 0.11 in cyclohexane/EtOAc 1:2). Isolated as a yellow oil (83 mg, 0.17 mmol, 67%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 – 7.45 (m, 2H), 7.21 – 7.13 (m, 7H), 6.74 – 6.68 (m, 3H), 6.53 (d, *J* = 8.2 Hz, 2H), 5.68 (s, 1H), 5.55 (s, 1H), 4.39 (s, 1H), 3.90 (t, *J* = 9.5 Hz, 1H), 3.75 – 3.57 (m, 1H), 2.73 (q, *J* = 12.5, 10.5 Hz, 1H), 2.30 (dd, *J* = 12.7, 6.4 Hz, 1H), 2.12 (s, 3H), 0.87 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.2 (C_q), 165.7 (C_q), 149.8 (C_q), 144.0 (C_q), 139.5 (C_q), 129.7 (CH), 129.6 (CH), 129.5 (CH), 129.3 (CH), 128.5 (CH), 126.9 (CH), 125.9 (C_q), 119.0 (CH), 117.6 (CH), 112.4 (CH), 109.6 (CH), 82.0 (CH), 81.3 (C_q), 71.9 (CH), 66.8 (C_q), 51.3 (C_q), 46.8 (CH_2), 36.6 (CH_2), 28.0 (CH_3), 23.7 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 2361 (s), 1636 (s), 1504 (s), 1213 (s), 1041 (s), 906 (l), 727 (l); HRMS (ESI): *m/z* calculated for $C_{31}H_{35}N_4O_2$ $[M+H]^+$ 495.2755, found: 495.2742.

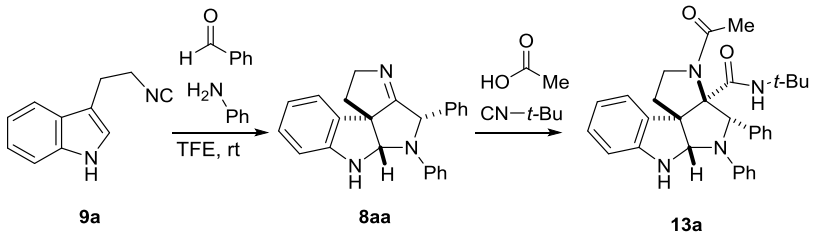
3-benzoyl-N-butyl-5-(3-chlorophenyl)-4-(4-chlorophenyl)-2,3,4,5,5a,6-hexahydropyrrolo[3',2':3,4] pyrrolo[2,3-b]indole-3a(1H)-carboxamide (13b)



Prepared from interrupted Ugi product **3ac** (59 mg, 0.14 mmol, 1 equiv), benzoic acid (21 mg, 0.17 mmol, 1.2 equiv) and 1-pentyl isocyanide (21.4 µl, 0.17 mmol, 1.2 equiv) according to general procedure B within 24 h. Purification: column chromatography on silicagel (cyclohexane/EtOAc 4:1, R_f = 0.27 in cyclohexane/EtOAc 4:1). Isolated as a yellow oil (40 mg, 0.44 mmol, 44%).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers observed in 5:1 ratio) δ 8.10 (d, *J* = 8.0 Hz, 1.2H), 7.63 – 7.51 (m, 4.4H), 7.46 (t, *J* = 7.7 Hz, 1.4H), 7.44 – 7.35 (m, 2.6H), 7.25 – 7.14 (m, 3.4H), 7.11 (t, *J* = 7.8 Hz, 0.4H), 7.05 (t, *J* = 8.2 Hz, 1H), 6.95 (d, *J* = 7.6 Hz, 1H), 6.79 – 6.68 (m, 2.6H), 6.62 (s, 1H), 6.52 (s, 0.2H), 6.42 – 6.34 (m, 1.2H), 5.73 (s, 1.2H), 5.67 (s, 1.2H), 4.66 (s, 0.2H), 4.14 (s, 1H), 3.86 – 3.74 (m, 2H), 3.72 – 3.62 (m, 0.2H), 3.58 – 3.50 (m, 0.2H), 3.49 – 3.42 (m, 0.2H), 3.34 – 3.27 (m, 0.2H), 3.13 – 3.04 (m, 1H), 2.97 (dt, *J* = 12.8, 5.9 Hz, 0.2H), 2.89 – 2.76 (m, 1.2H), 2.70 (q, *J* = 10.7, 10.3 Hz, 1H), 2.22 (dd, *J* = 12.8, 5.4 Hz, 1H), 1.11 (h, *J* = 7.1 Hz, 2.2H), 0.98 – 0.85 (m, 1H), 0.82 – 0.64 (m, 5.6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9 (C_q), 170.8 (C_q), 170.1 (C_q), 166.3 (C_q), 149.4 (C_q), 147.5 (C_q), 144.9 (C_q), 137.0 (C_q), 136.4 (C_q), 135.4 (C_q), 134.9 (C_q), 133.6 (CH), 130.8 (CH), 130.7 (CH), 130.5 (CH), 130.4 (CH), 130.2 (CH), 130.1 (CH), 129.6 (CH), 129.5 (CH), 128.7 (CH), 128.5 (CH), 128.4 (CH), 127.2 (CH), 125.8 (C_q), 125.7 (CH), 122.4 (CH), 122.3 (CH), 119.8 (CH), 119.6 (CH), 118.9 (CH), 118.6 (CH), 118.1 (CH), 113.7 (CH), 112.4 (CH), 112.0 (CH), 111.5 (CH), 111.2 (CH), 110.1 (CH), 82.0 (CH), 81.5 (C_q), 71.6 (CH), 67.3 (C_q), 62.5 (CH), 48.7 (CH_2), 40.0 (CH_2), 39.7 (CH_2), 37.1 (CH_2), 29.0 (CH_2), 27.8 (CH_2), 27.0 (CH_2), 25.1 (CH_2), 22.3 (CH_2), 14.0 (CH_3); IR (neat): ν_{max} (cm^{-1}) = 3427 (s), 2930 (s), 2357 (s), 2357 (s), 1624 (s), 1593 (l), 1485 (l), 1447 (s), 1406 (m), 1265 (m), 1173 (s), 1092 (m), 1026 (s), 908 (m), 729 (l); HRMS (ESI): *m/z* calculated for $C_{37}H_{36}Cl_2N_4O_2$ $[M+Na]^+$ 661.2108, found: 661.2088.

One pot interrupted Ugi / Joullié-Ugi towards 8a.



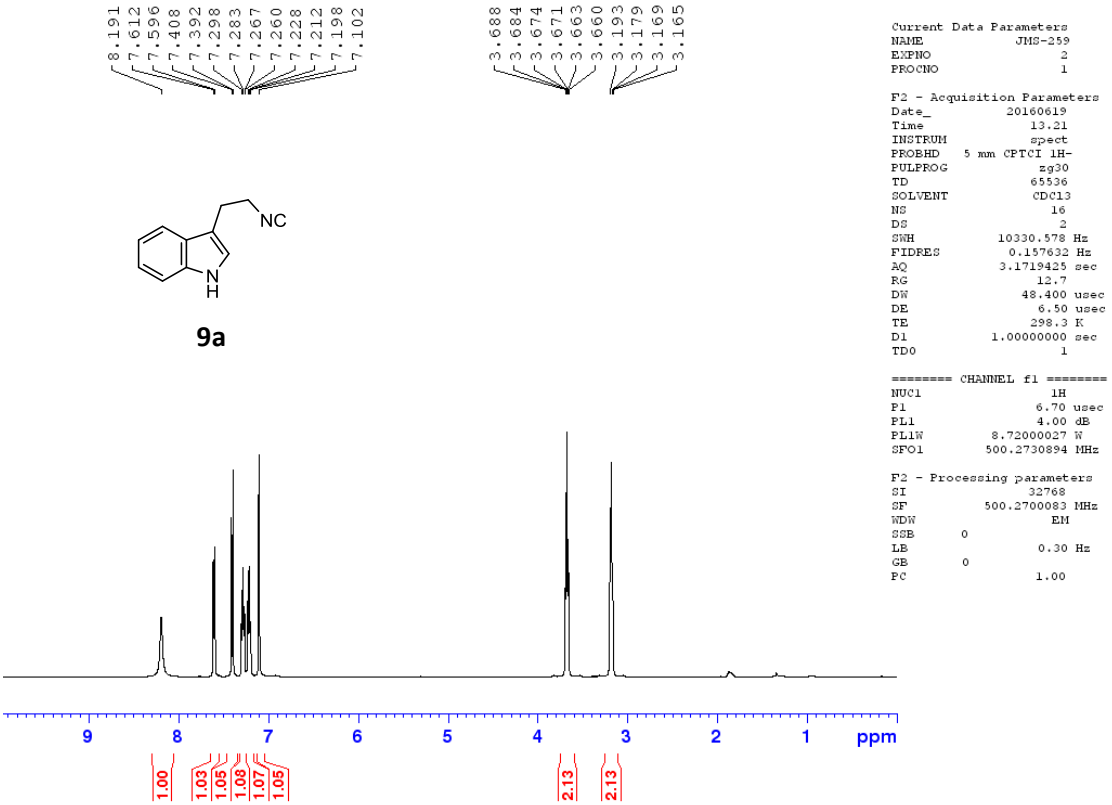
To a solution of isocyanide **1a** (170 mg, 1.0 mmol, 1.0 equiv) in TFE (10.0 mL) was added aniline (100 µl, 1.1 mmol, 1.1 equiv) and benzaldehyde (112 µl, 1.1 mmol, 1.1 equiv). The reaction mixture was stirred at room temperature for 48 h. After full depletion of **1a** (monitored by TLC), acetic acid (69 µl , 1.2 mmol, 1.2 equiv) and *tert*-butyl isocyanide (136 µl, 1.2 mmol, 1.2 equiv) were respectively added. The intermediate interrupted Ugi product was fully converted after 24h. The reaction was concentrated and purified by column chromatography (cyclohexane/EtOAc, 2:1), to afford compound **8a** as a pale yellow oil (207 mg, 0.42 mmol, 42 %).

X-ray analysis

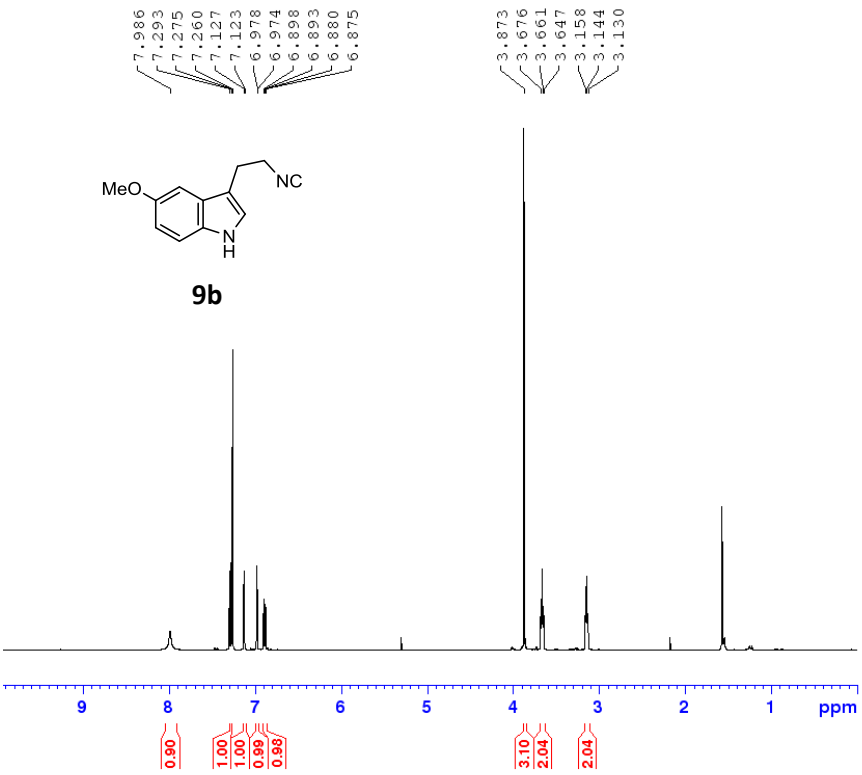
A single crystal of **8ah** was mounted on a Mitegen kapton loop and placed in the cold stream (104K) of the diffractometer setup described in the experimental section. Data collection and refinement led to the CIF file in the supporting information, in which all further pertinent details are described.

The main finding from the diffraction experiment is that **8ah** crystallizes in the centrosymmetric space group $P2_1/c$, as a racemic mixture of two enantiomers, (*S,S,S*) and (*R,R,R*), while NMR allows to conclude that the bulk product is diastereomerically pure. In the packing, the benzyl substituent of one molecule points into the cleft formed by the spiroindole, benzyl and *tert*-butyl substituents of its enantiomer, and *vice versa*. The resulting bimolecular building block is much more regularly shaped and thus easier to pack into a crystal. Intermolecular contacts below the van der Waals radius just barely occur between N12 of the hexahydropyrrole and H3 in the indoline benzene ring of the molecule in the a-direction, and the same N12 and H4 in the same ring of the molecule in the b-direction (both distances 2.72 Å, van der Waals distance 2.75 Å). This is consistent with the packing being largely determined by dispersion forces.

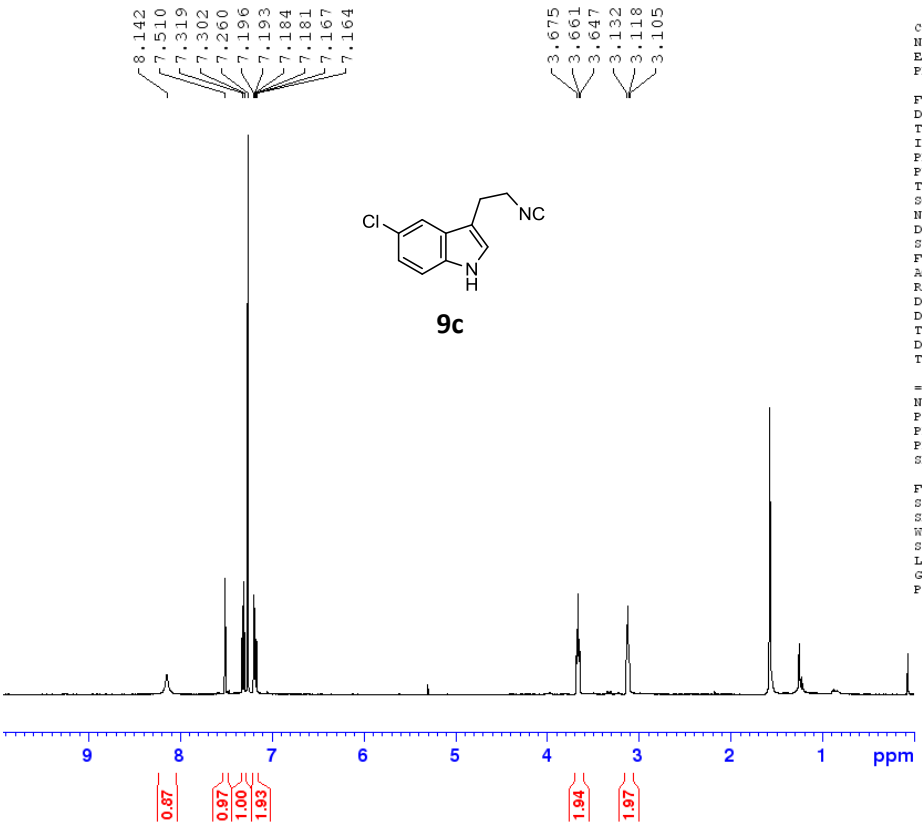
Copies of Spectra



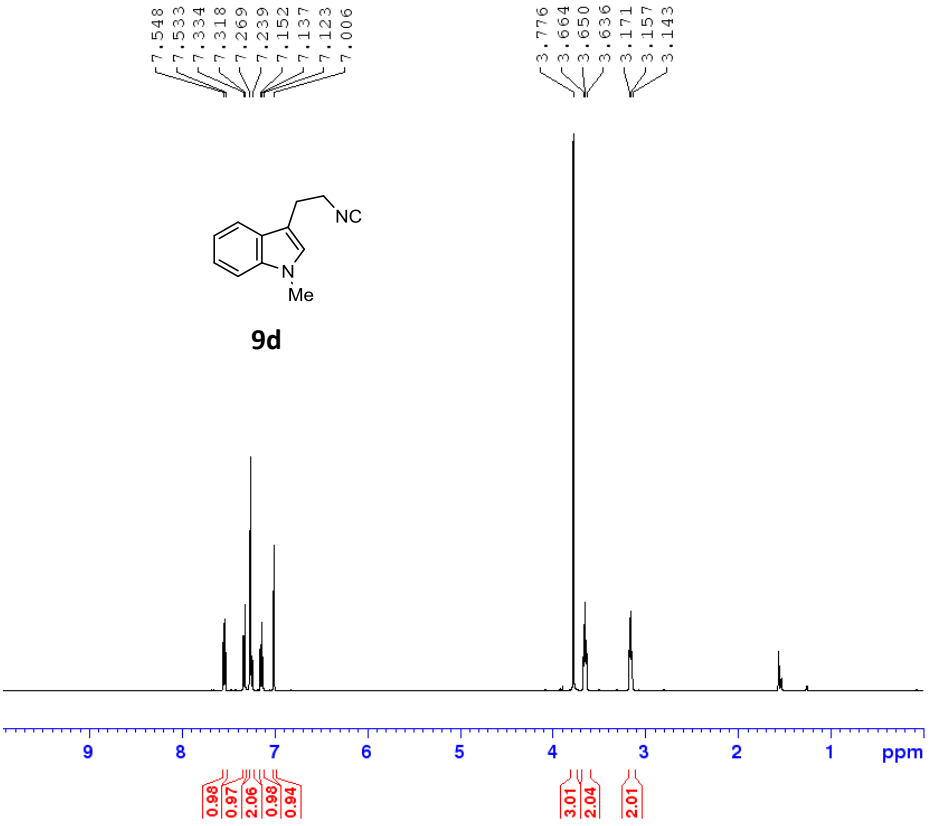

```
Current Data Parameters
NAME                  JMS-259
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160619
Time                   13.21
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                      12.7
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      6.70 usec
PL1                     4.00 dB
PL1W              8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                      32768
SF                500.2700083 MHz
WDW                        EM
SSB          0
LB                     0.30 Hz
GB           0
PC                      1.00
```



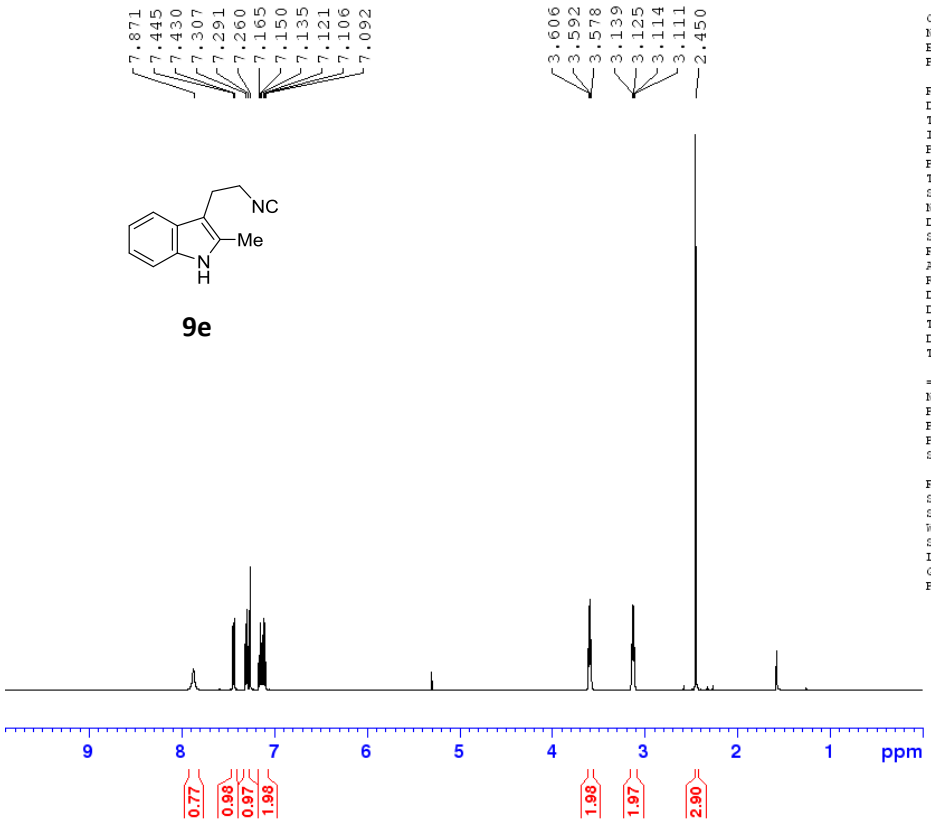

```
Current Data Parameters
NAME                  JMS-268
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160629
Time                   15.53
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                        16
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      6.70 usec
PL1                     4.00 dB
PL1W              8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                      32768
SF                500.2700085 MHz
WDW                        EM
SSB          0
LB                     0.30 Hz
GB           0
PC                      1.00
```




```
Current Data Parameters
NAME            BOR-037-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160621
Time                11.04
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719425 sec
RG                     18
DW                48.400 usec
DE                 6.50 usec
TE                298.3 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1        500.2730894 MHz

F2 - Processing parameters
SI                 32768
SF          500.2700084 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

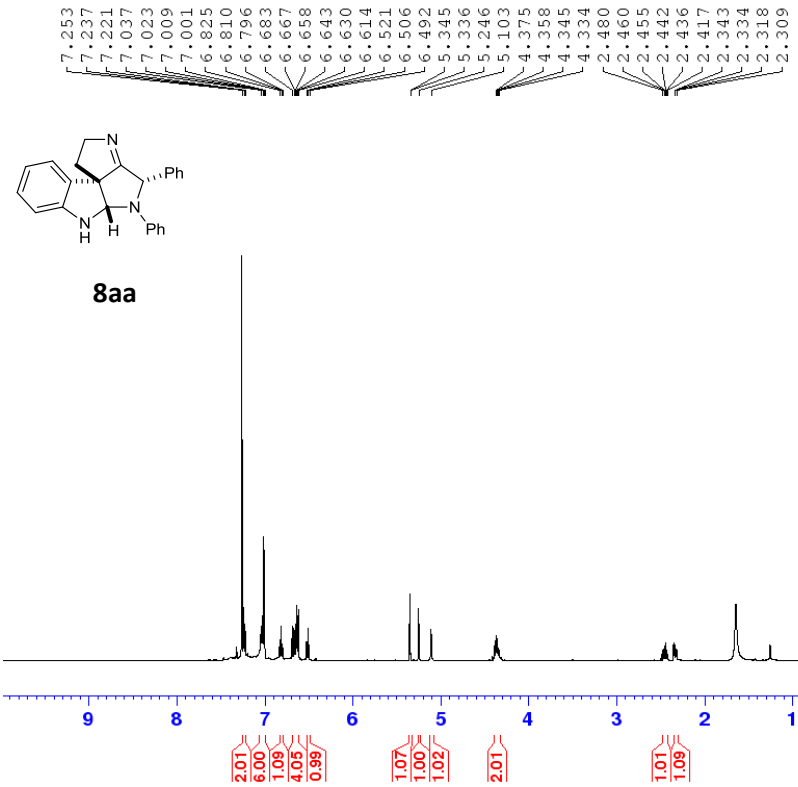



```
Current Data Parameters
NAME             JMS-257
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160620
Time                17.34
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719425 sec
RG                     18
DW                48.400 usec
DE                 6.50 usec
TE                298.3 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1        500.2730894 MHz

F2 - Processing parameters
SI                 32768
SF          500.2700085 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

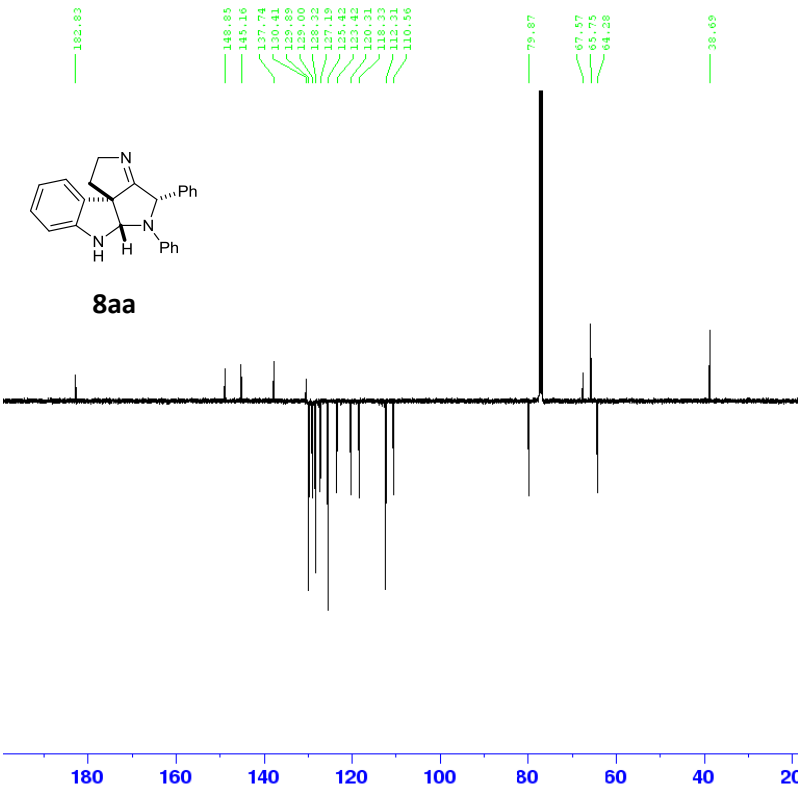


9e

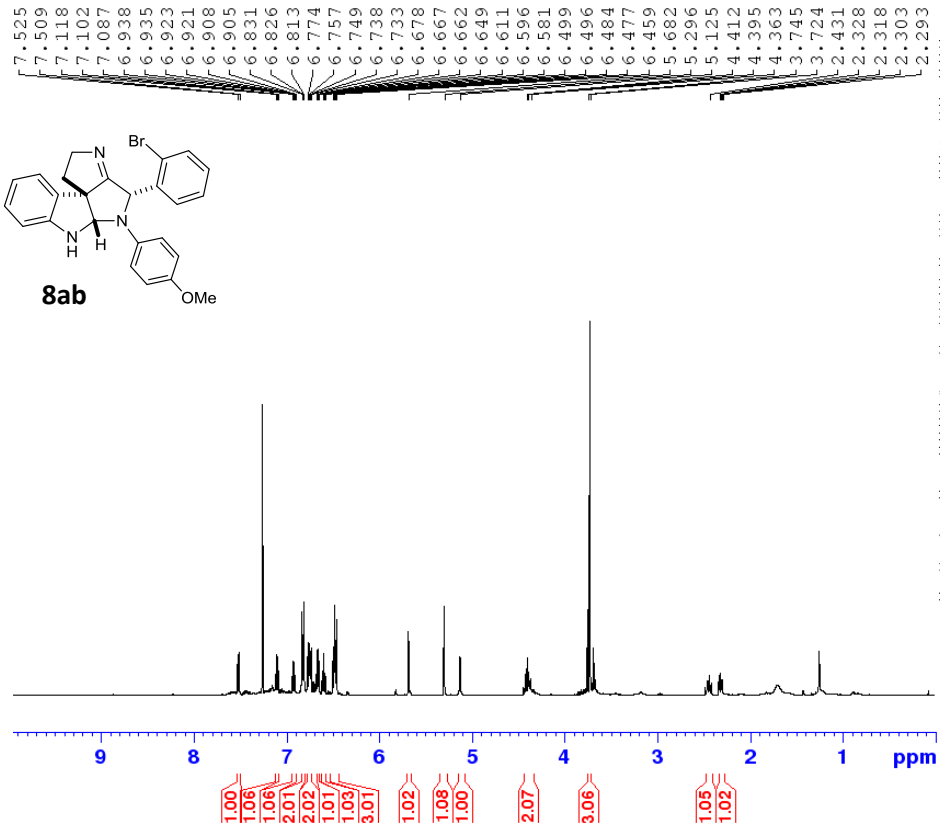
```
Current Data Parameters
NAME                  JMS-258
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160620
Time                  17.40
INSTRUM               spect
PROBHD    5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                       16
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      6.70 usec
PL1                     4.00 dB
PL1W              8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                      32768
SF               500.2700085 MHz
WDW                        EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



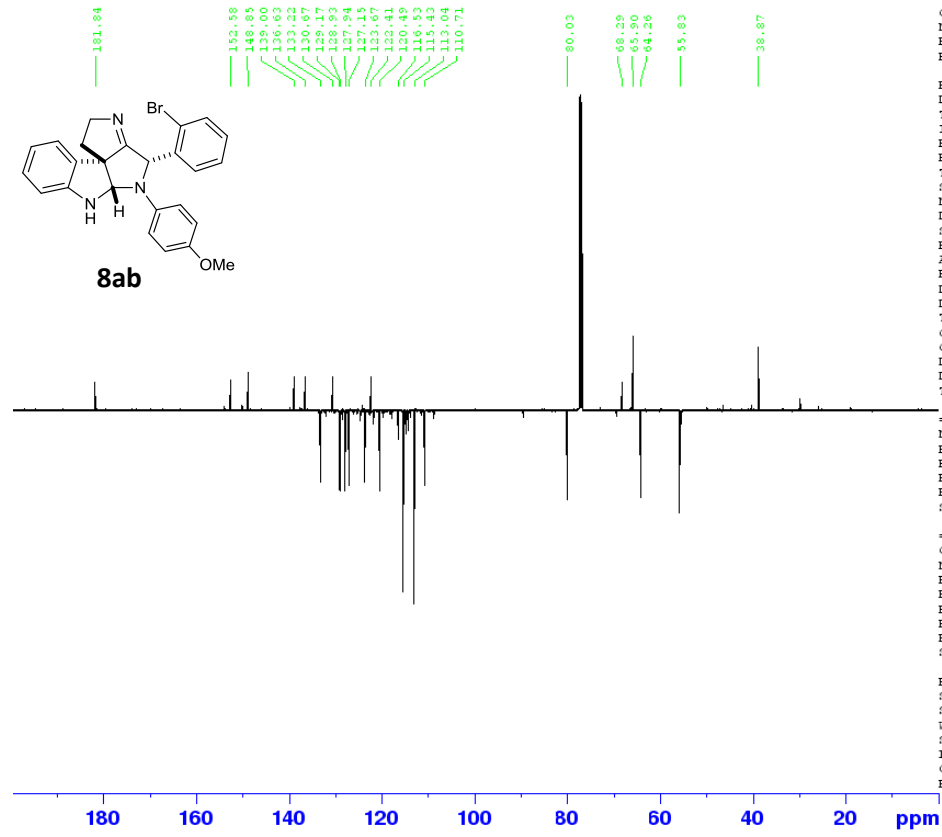

```
Current Data Parameters
NAME            BOR-024-2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_             20160716
Time                17.25
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                     128
DS                       0
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719425 sec
RG                      18
DW                 48.400 usec
DE                  6.50 usec
TE                 286.9 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W           8.72000027 W
SFO1          500.2730894 MHz

F2 - Processing parameters
SI                   32768
SF            500.2700088 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



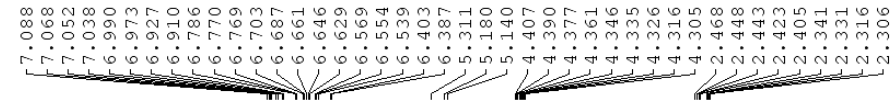

```
Current Data Parameters
NAME            BOR-024-2
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_             20160716
Time                19.11
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              jmod
TD                   65536
SOLVENT              CDCl3
NS                    2000
DS                       4
SWH             29761.904 Hz
FIDRES           0.454131 Hz
AQ               1.1010048 sec
RG                    2050
DW                 16.800 usec
DE                  6.50 usec
TE                 287.0 K
CNST2         145.0000000
CNST11          1.0000000
D1              2.00000000 sec
D20             0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
P2                 22.40 usec
PL1                -2.00 dB
PL1W           88.77790070 W
SFO1          125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               25.28 dB
PL2W           8.72000027 W
PL12W          0.06494062 W
SFO2          500.2720011 MHz

F2 - Processing parameters
SI                   32768
SF            125.7929809 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

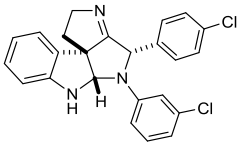



```
Current Data Parameters
NAME            BOR-024-3
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160715
Time                17.56
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      0
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719425 sec
RG                     16
DW                48.400 usec
DE                 6.50 usec
TE                287.2 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   6.70 usec
PL1                  4.00 dB
PL1W          8.72000027 W
SFO1         500.2730894 MHz

F2 - Processing parameters
SI                  32768
SF           500.2700085 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



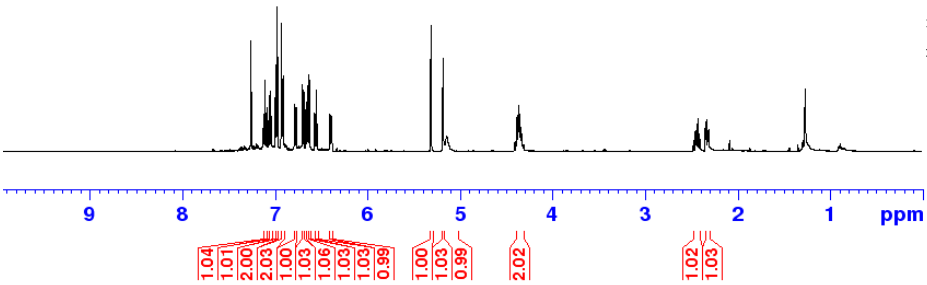

```
Current Data Parameters
NAME            BOR-024-3
EXPNO                   9
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160716
Time                21.00
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                  65536
SOLVENT             CDCl3
NS                   2000
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                   2050
DW                16.800 usec
DE                 6.50 usec
TE                287.0 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.20 usec
P2                  22.40 usec
PL1                 -2.00 dB
PL1W         88.77790070 W
SFO1        125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  4.00 dB
PL12                25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2         500.2720011 MHz

F2 - Processing parameters
SI                  32768
SF           125.7929828 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

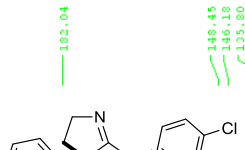




8ac

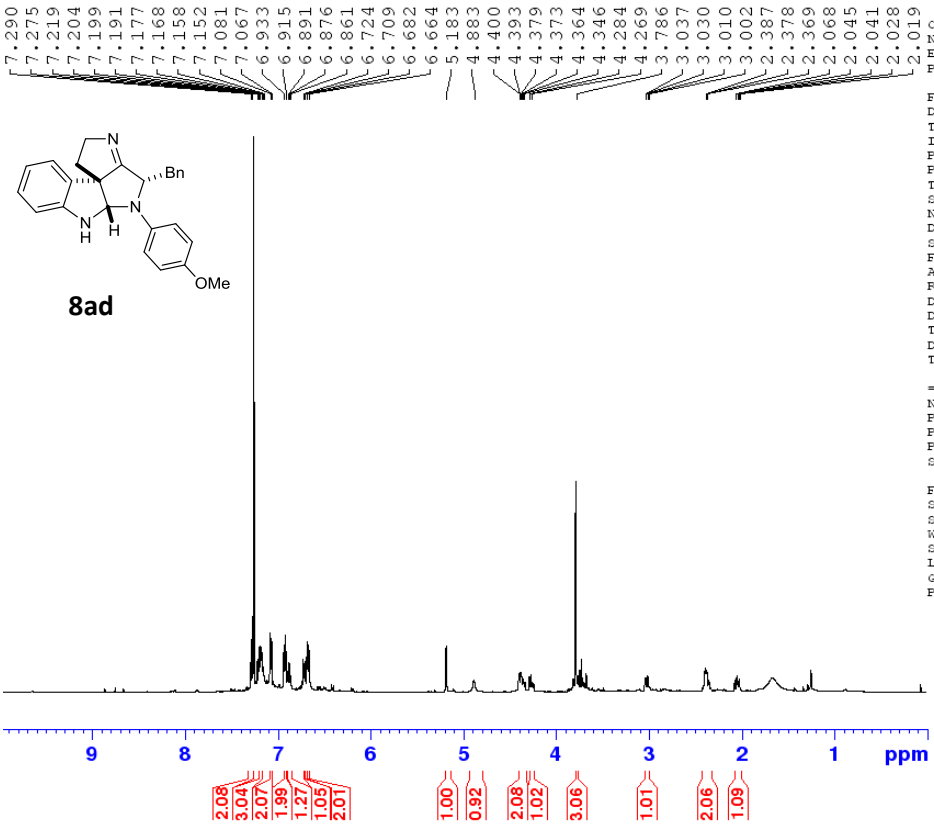
```
Current Data Parameters
NAME            BOR-048-1
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160722
Time                7.58
INSTRUM            spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                   14.2
DW                 48.400 usec
DE                  6.50 usec
TE                 298.3 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   6.70 usec
PL1                  4.00 dB
PL1W          8.72000027 W
SFO1         500.2730894 MHz

F2 - Processing parameters
SI                  32768
SF           500.2700085 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



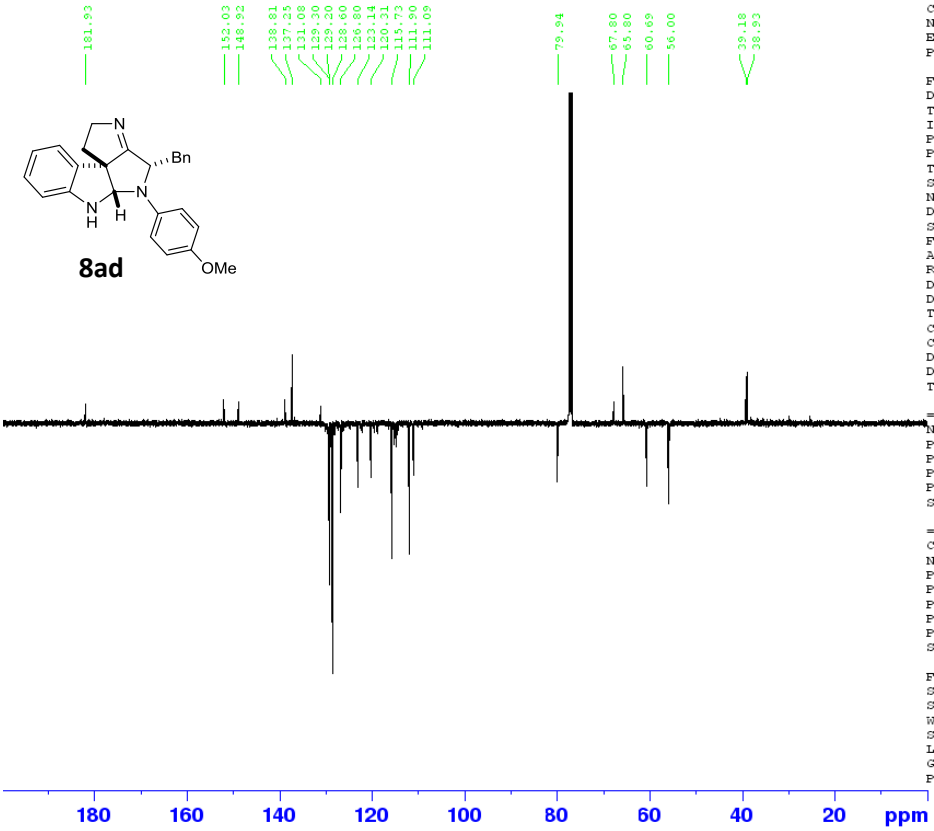


8ac

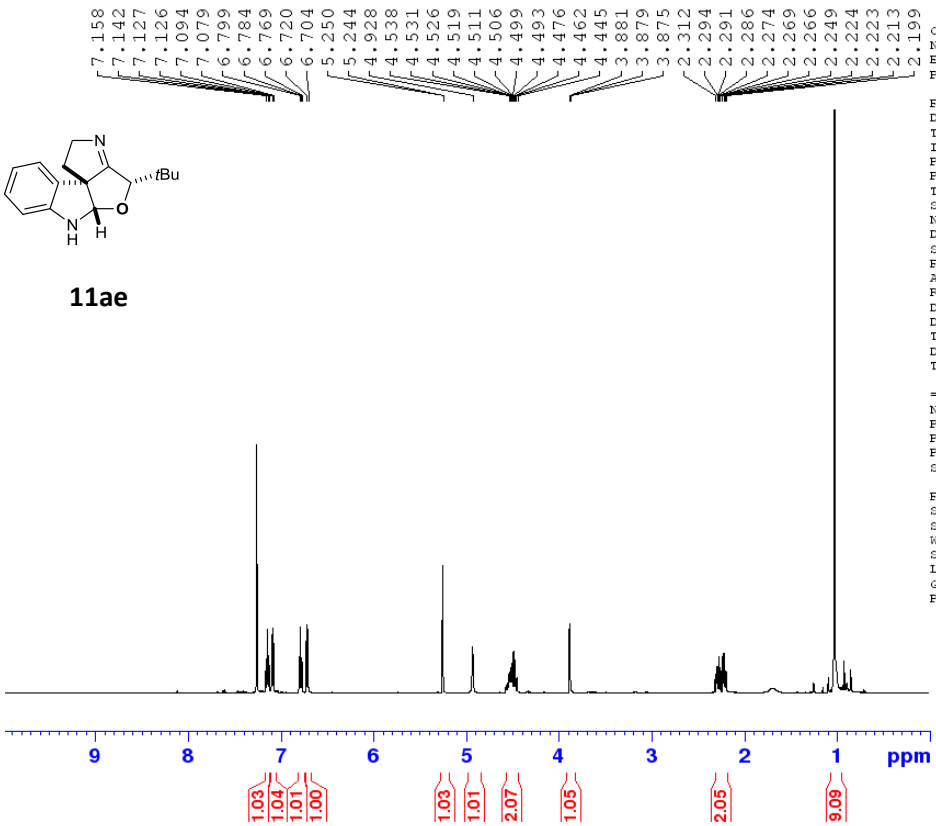

```
Current Data Parameters
NAME            BOR-048-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160720
Time                8.55
INSTRUM            spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                   2050
DW                 16.800 usec
DE                  6.50 usec
TE                 298.2 K
CNST2        145.0000000
CNST11         1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.20 usec
P2                  22.40 usec
PL1                 -2.00 dB
PL1W         88.77790070 W
SFO1        125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  4.00 dB
PL12                25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2         500.2720011 MHz

F2 - Processing parameters
SI                  32768
SF           125.7929836 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



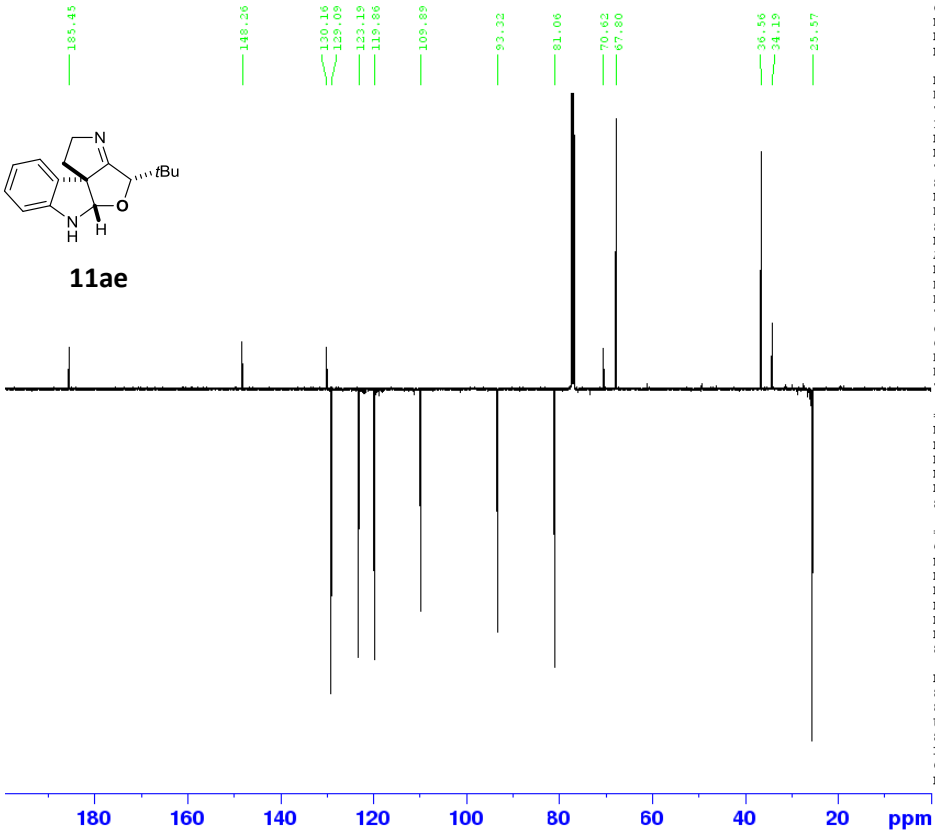

```
Current Data Parameters
NAME                  JMS-265-C
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20160716
Time                      15.24
INSTRUM                   spect
PROBHD     5 mm CPTCI 1H-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                         128
DS                           0
SWH                  10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1719425 sec
RG                          18
DW                       48.400 usec
DE                        6.50 usec
TE                       287.0 K
D1                    2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        6.70 usec
PL1                       4.00 dB
PL1W                 8.72000027 W
SFO1               500.2730894 MHz

F2 - Processing parameters
SI                           32768
SF                 500.2700085 MHz
WDW                             EM
SSB                 0
LB                            0.30 Hz
GB                  0
PC                            1.00
```



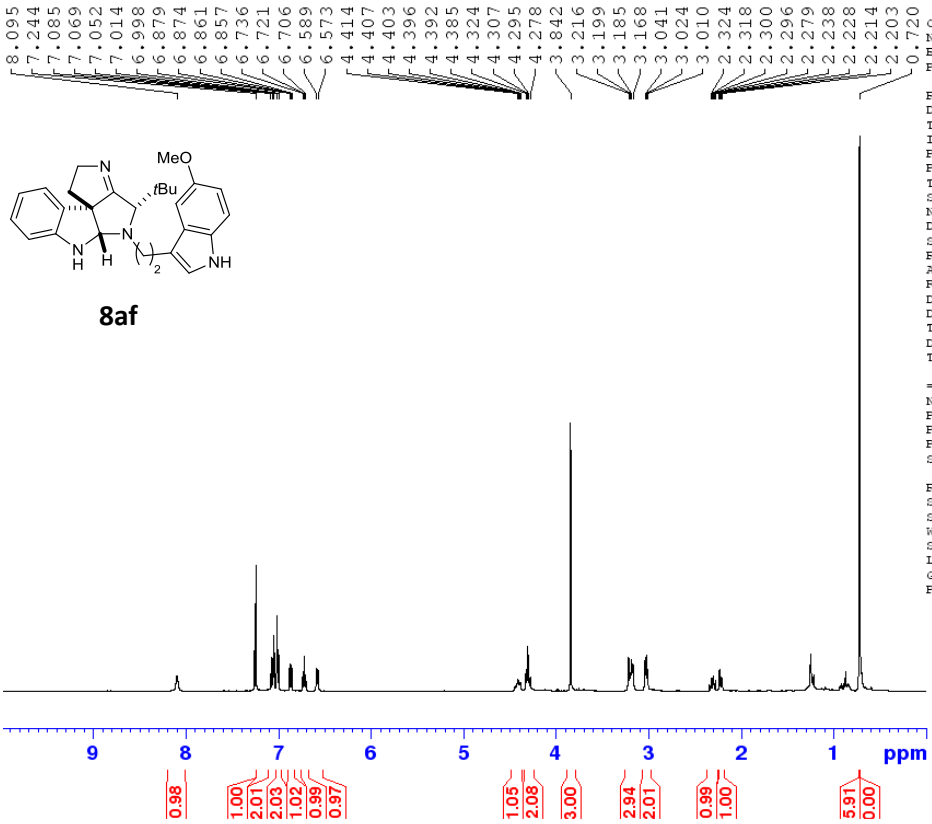

```
Current Data Parameters
NAME                  JMS-265-C
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20160716
Time                      17.10
INSTRUM                   spect
PROBHD     5 mm CPTCI 1H-
PULPROG                   jmod
TD                       65536
SOLVENT                  CDCl3
NS                        2000
DS                           4
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        2050
DW                       16.800 usec
DE                        6.50 usec
TE                       286.9 K
CNST2                145.0000000
CNST11                 1.0000000
D1                    2.00000000 sec
D20                   0.00689655 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         13C
P1                       11.20 usec
P2                       22.40 usec
PL1                      -2.00 dB
PL1W                88.77790070 W
SFO1               125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       4.00 dB
PL12                     25.28 dB
PL2W                 8.72000027 W
PL12W                0.06494062 W
SFO2               500.2720011 MHz

F2 - Processing parameters
SI                           32768
SF                 125.7929813 MHz
WDW                             EM
SSB                 0
LB                            1.00 Hz
GB                  0
PC                            1.40
```




```
Current Data Parameters
NAME                  JMS-243-D
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160716
Time                       9.20
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                         128
DS                           0
SWH                  10330.578 Hz
FIDRES                0.157632 Hz
AQ                   3.1719425 sec
RG                          18
DW                      48.400 usec
DE                       6.50 usec
TE                      287.0 K
D1                   2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        6.70 usec
PL1                       4.00 dB
PL1W                8.72000027 W
SFO1              500.2730894 MHz

F2 - Processing parameters
SI                        32768
SF                  500.2700085 MHz
WDW                          EM
SSB             0
LB                        0.30 Hz
GB              0
PC                        1.00
```

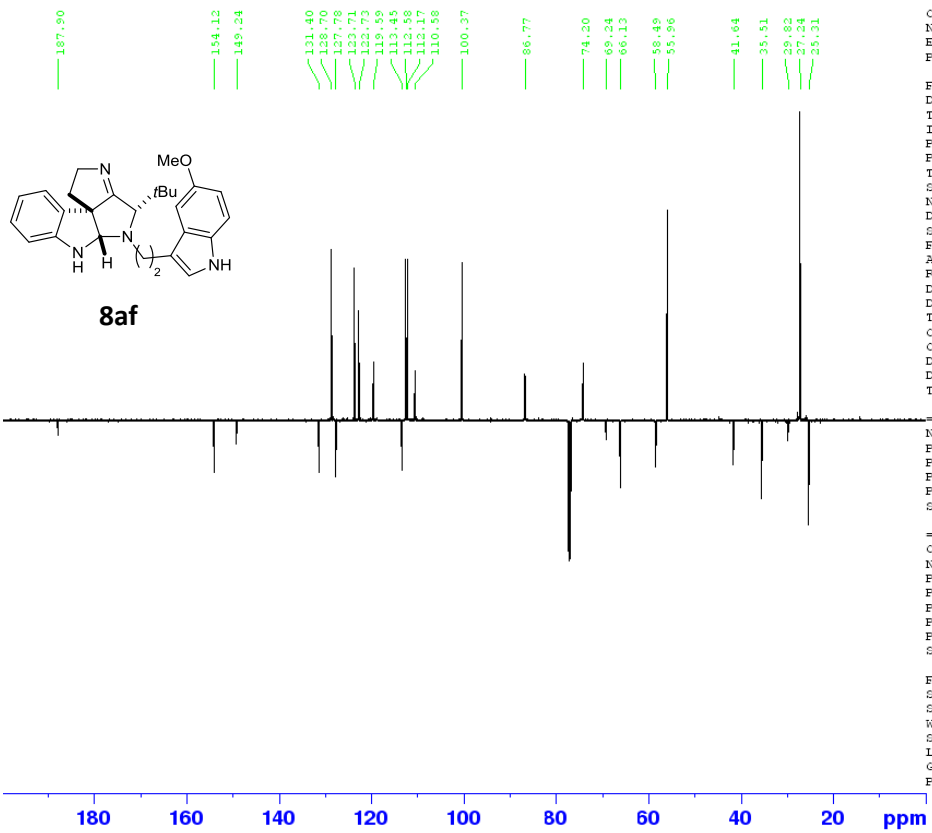



```
Current Data Parameters
NAME                  JMS-243-D
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160716
Time                      11.05
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                   jmod
TD                       65536
SOLVENT                  CDCl3
NS                        2000
DS                           4
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                   1.1010048 sec
RG                        2050
DW                      16.800 usec
DE                       6.50 usec
TE                      286.9 K
CNST2               145.0000000
CNST11                1.0000000
D1                   2.00000000 sec
D20                  0.00689655 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         13C
P1                       11.20 usec
P2                       22.40 usec
PL1                      -2.00 dB
PL1W               88.77790070 W
SFO1              125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                       4.00 dB
PL12                     25.28 dB
PL2W                8.72000027 W
PL12W               0.06494062 W
SFO2              500.2720011 MHz

F2 - Processing parameters
SI                        32768
SF                  125.7929818 MHz
WDW                          EM
SSB             0
LB                        1.00 Hz
GB              0
PC                        1.40
```



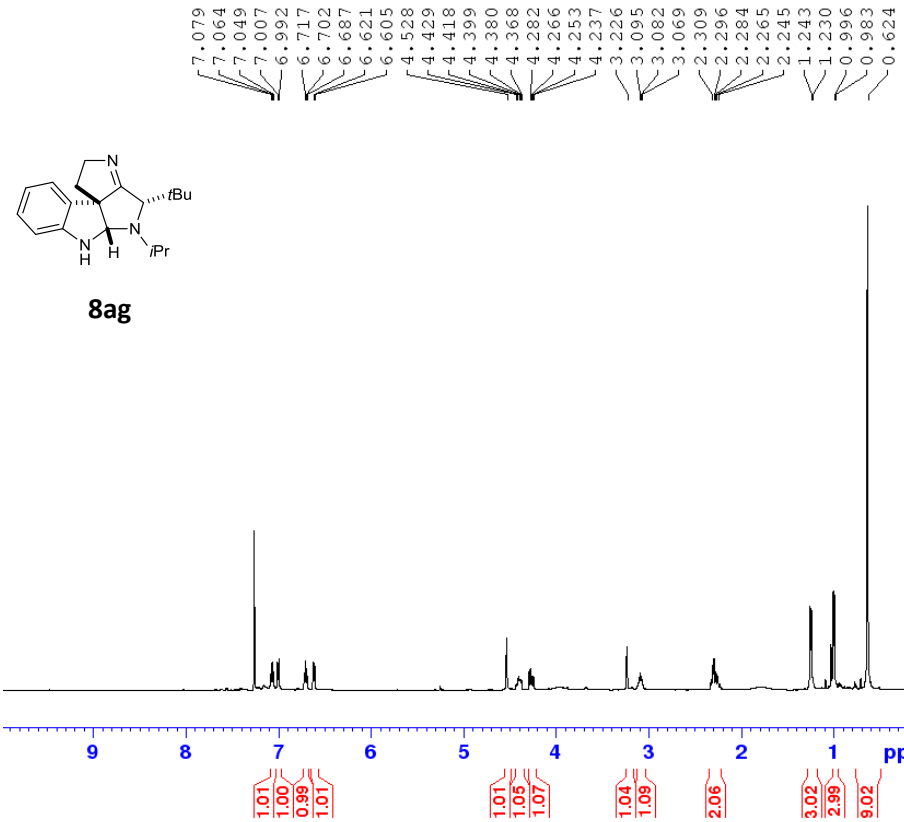
8af

```
Current Data Parameters
NAME                  BOR-035-1
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                20160626
Time                    17.29
INSTRUM                 spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                      14.2
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.70 usec
PL1                    4.00 dB
PL1W              8.72000027 W
SFO1            500.2730894 MHz

F2 - Processing parameters
SI                     32768
SF              500.2700161 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



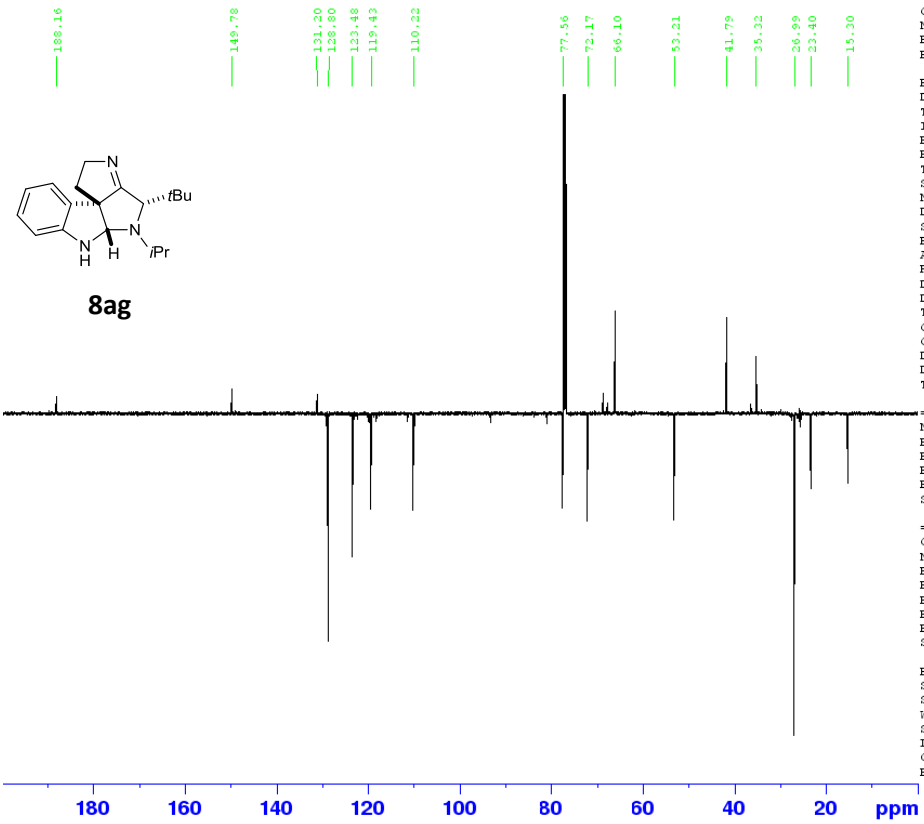
8af

```
Current Data Parameters
NAME                  BOR-035-1
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                20160626
Time                    17.56
INSTRUM                 spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 jmod
TD                     65536
SOLVENT                CDCl3
NS                       512
DS                         4
SWH                29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                      2050
DW                    16.800 usec
DE                     6.50 usec
TE                    298.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                 2.00000000 sec
D20                0.00689655 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
P2                    22.40 usec
PL1                   -2.00 dB
PL1W             88.77790070 W
SFO1            125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  25.28 dB
PL2W              8.72000027 W
PL12W             0.06494062 W
SFO2            500.2720011 MHz

F2 - Processing parameters
SI                     32768
SF              125.7929809 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



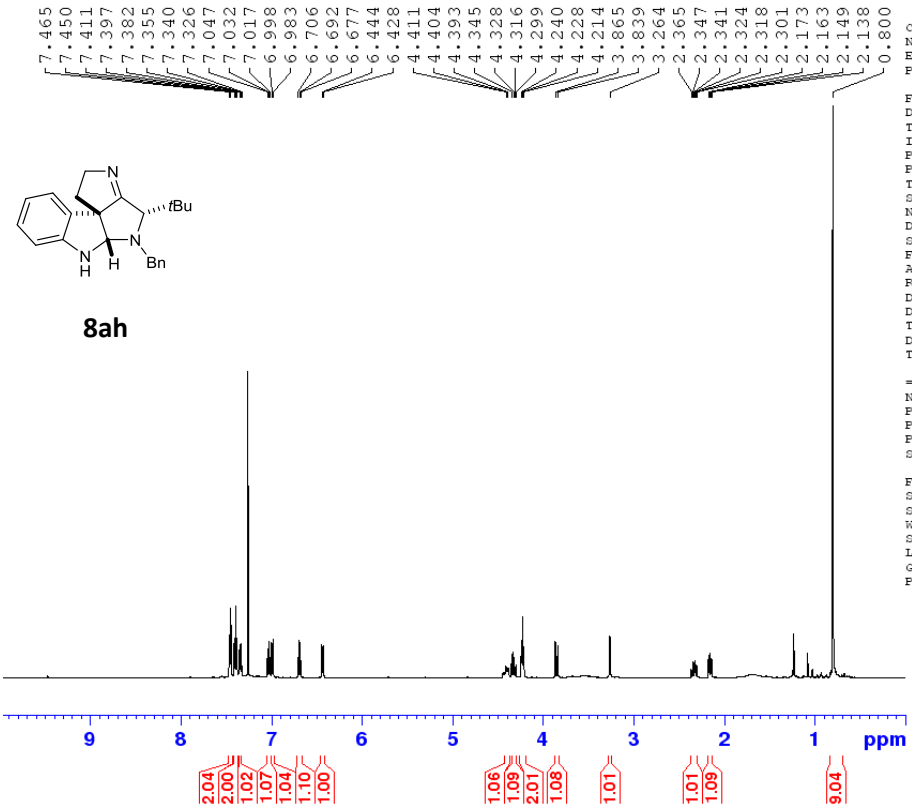
8ag

```
Current Data Parameters
NAME            BOR-025-1
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160716
Time                11.22
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      0
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719425 sec
RG                     18
DW                48.400 usec
DE                 6.50 usec
TE                286.9 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1         500.2730894 MHz

F2 - Processing parameters
SI                  32768
SF           500.2700085 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



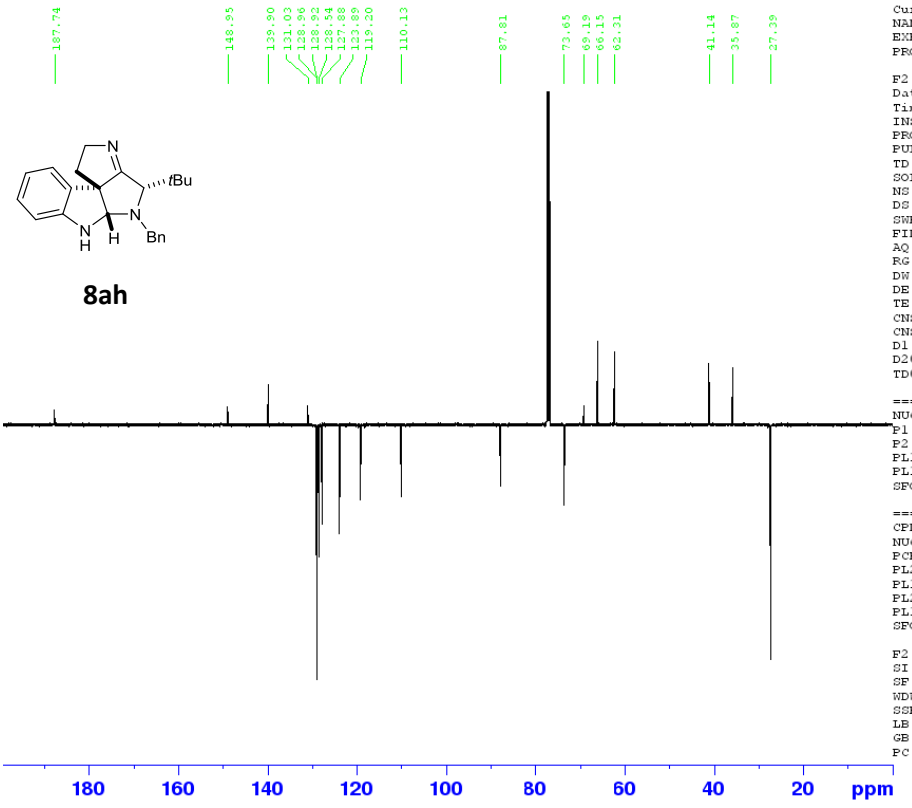
8ag

```
Current Data Parameters
NAME            BOR-025-1
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160716
Time                13.08
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                  65536
SOLVENT             CDCl3
NS                   2000
DS                      4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                   2050
DW                16.800 usec
DE                 6.50 usec
TE                286.9 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
P2                 22.40 usec
PL1                -2.00 dB
PL1W         88.77790070 W
SFO1        125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2         500.2720011 MHz

F2 - Processing parameters
SI                  32768
SF           125.7929818 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



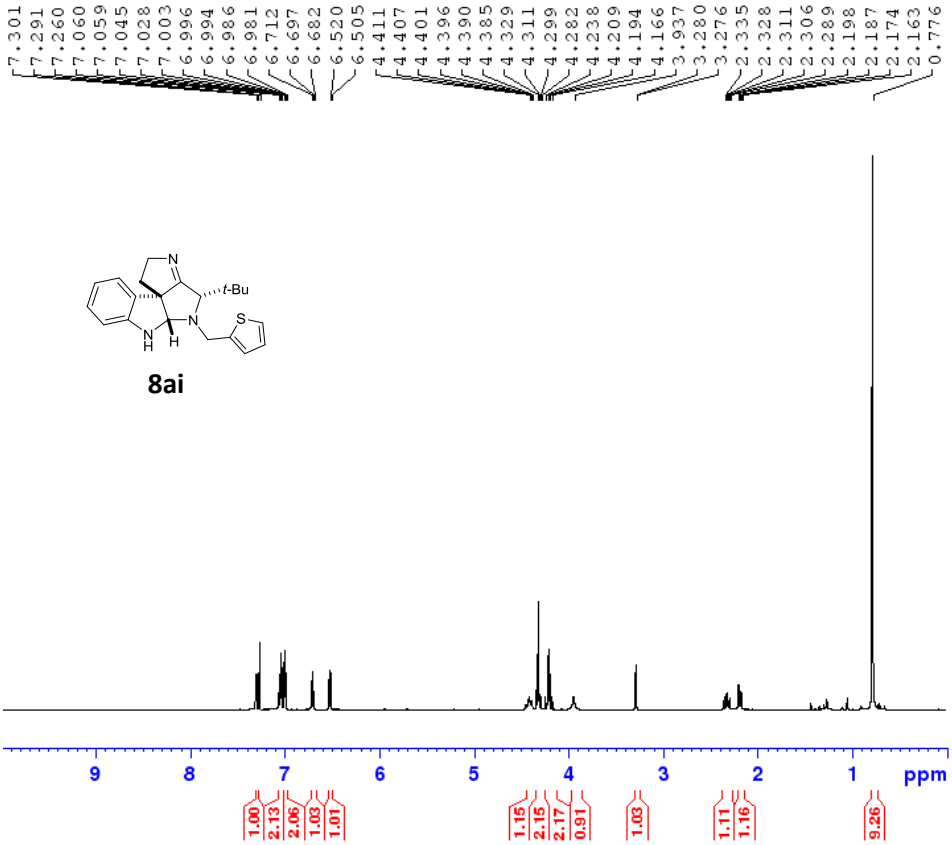
8ah

```
Current Data Parameters
NAME            BOR-022-1
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160716
Time               13.23
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     0
SWH           10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719425 sec
RG                    18
DW                48.400 usec
DE                 6.50 usec
TE                286.9 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 6.70 usec
PL1                4.00 dB
PL1W          8.72000027 W
SFO1        500.2730894 MHz

F2 - Processing parameters
SI                 32768
SF          500.2700086 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



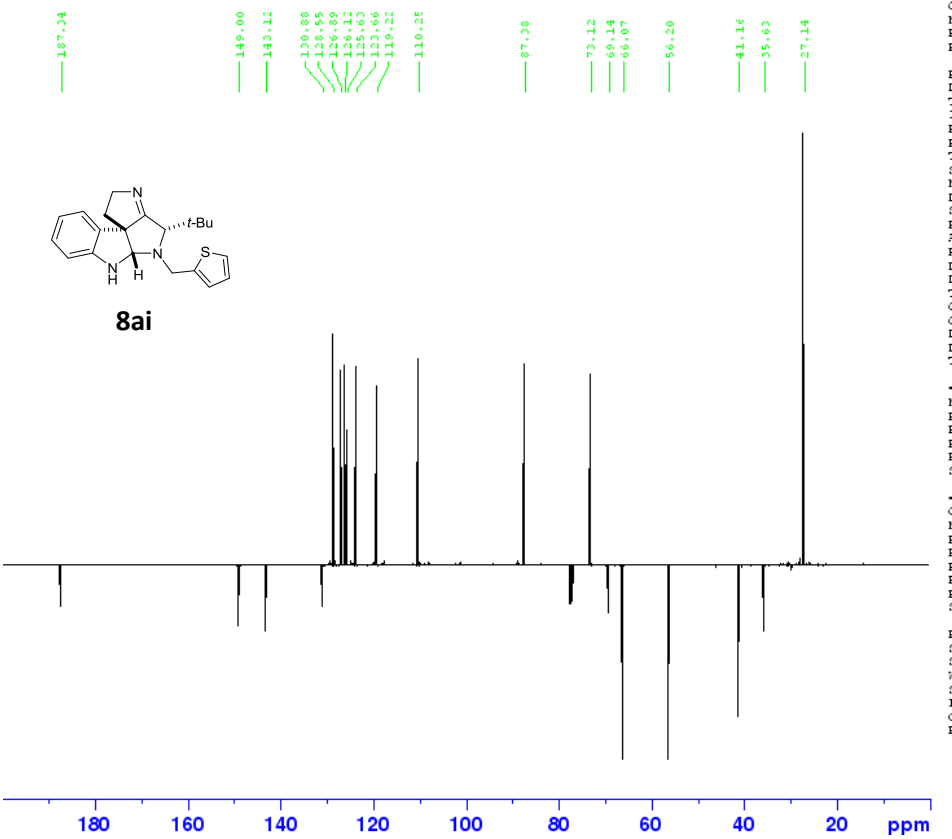
8ah

```
Current Data Parameters
NAME            BOR-022-1
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160716
Time               15.09
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG             jmod
TD                 65536
SOLVENT            CDCl3
NS                  2000
DS                     4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010048 sec
RG                  2050
DW                16.800 usec
DE                 6.50 usec
TE                286.9 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                11.20 usec
P2                22.40 usec
PL1               -2.00 dB
PL1W         88.77790070 W
SFO1        125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                4.00 dB
PL12              25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2        500.2720011 MHz

F2 - Processing parameters
SI                 32768
SF          125.7929809 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



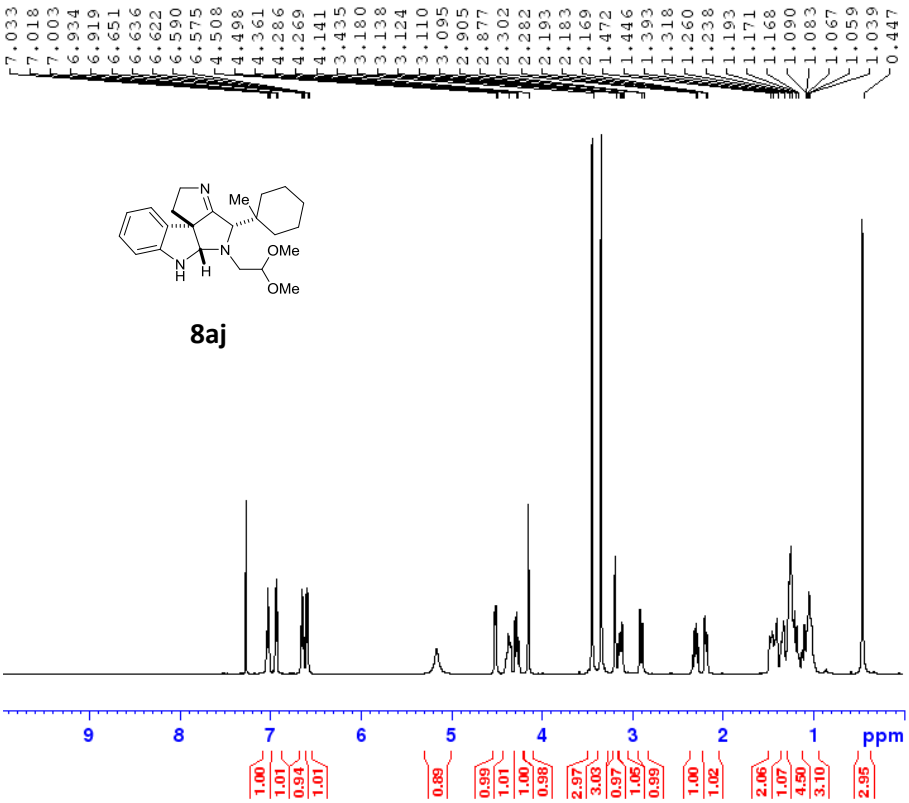
8ai

```
Current Data Parameters
NAME                  JMS-291B
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20160909
Time                   18.07
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                       10
DW                   48.400 use
DE                    6.50 use
TE                   298.3 K
D1                 1.00000000 sec
TD0                          1

-------- CHANNEL f1 -------
NUC1                        1H
P1                        6.70 use
PL1                       4.00 dB
PL1W                8.72000027 W
SFO1               500.2730894 MHz

F2 - Processing parameters
SI                        32768
SF                 500.2700081 MHz
WDW                          EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
```



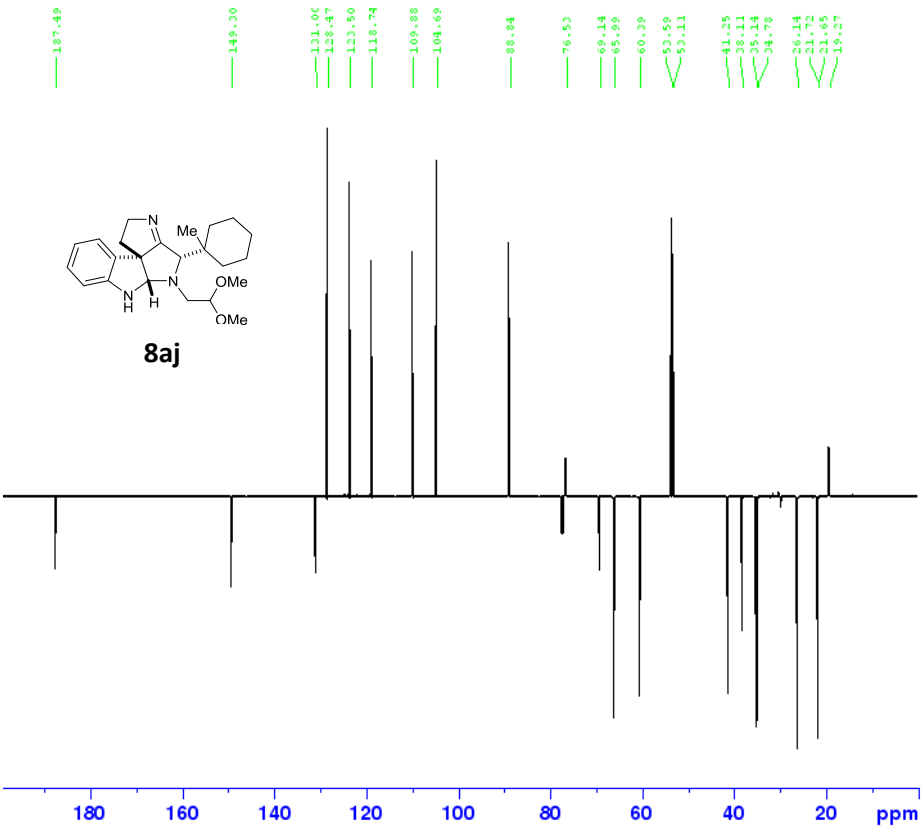
8ai

```
Current Data Parameters
NAME                  JMS-291B
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20160909
Time                   18.21
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                jmod
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH                29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                     2050
DW                  16.800 usec
DE                   6.50 usec
TE                   298.2 K
CNST2              145.0000000
CNST11               1.0000000
D1                 2.00000000 sec
D20                0.00689655 sec
TD0                          1

-------- CHANNEL f1 --------
NUC1                       13C
P1                      11.20 usec
P2                      22.40 usec
PL1                     -2.00 dB
PL1W               88.77790070 W
SFO1               125.8055709 MHz

-------- CHANNEL f2 --------
CPDPRG[2              waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     4.00 dB
PL12                   25.28 dB
PL2W                8.72000027 W
PL12W               0.06494062 W
SFO2               500.2720011 MHz

F2 - Processing parameters
SI                        32768
SF                 125.7929920 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                        1.40
```



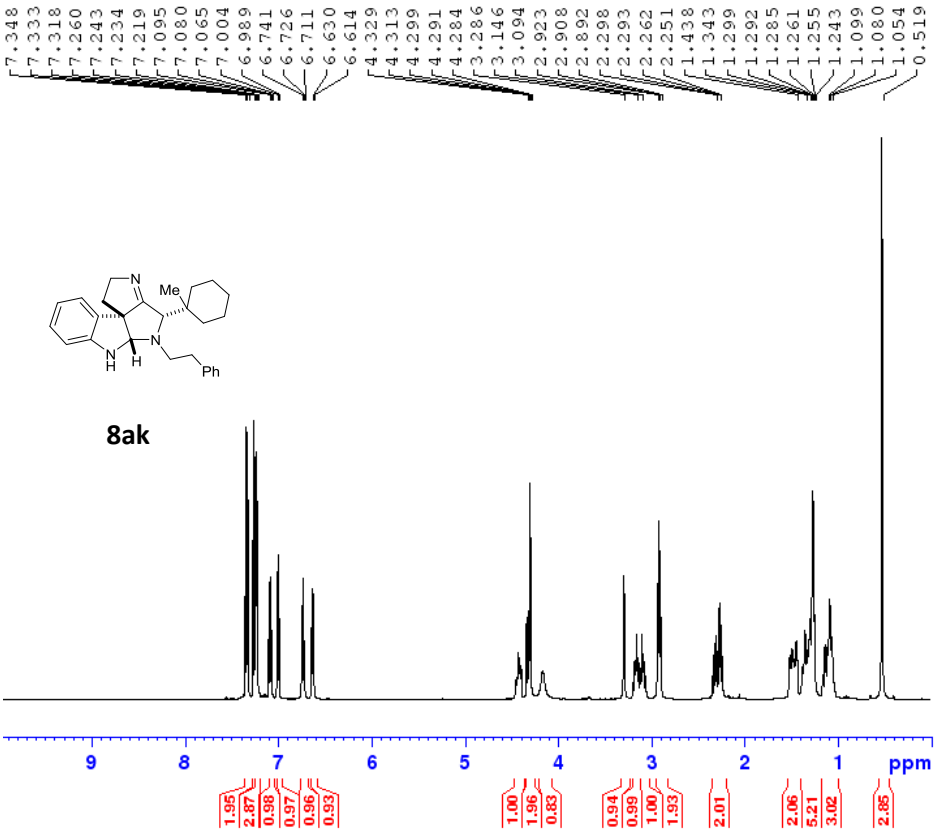
8aj

```
Current Data Parameters
NAME                  JMS-289B
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20160909
Time                   10.56
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                         8
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 =======
NUC1                      1H
P1                     6.70 usec
PL1                    4.00 dB
PL1W               8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                        32768
SF               500.2700082 MHz
WDW                          EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

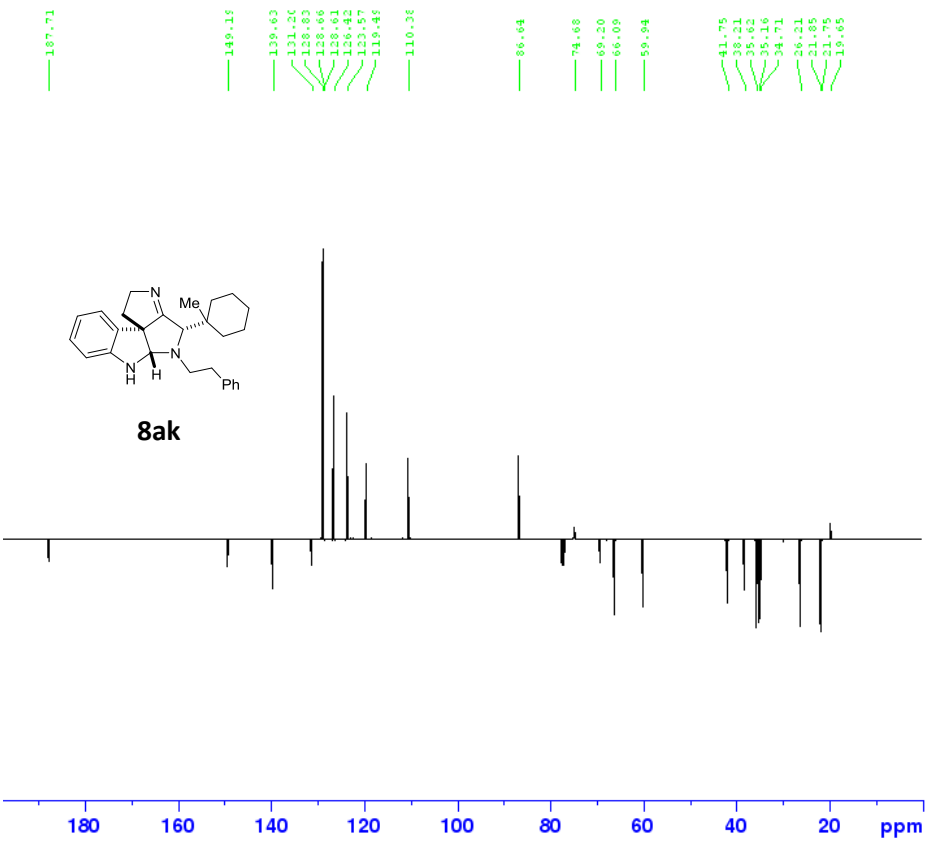


8aj

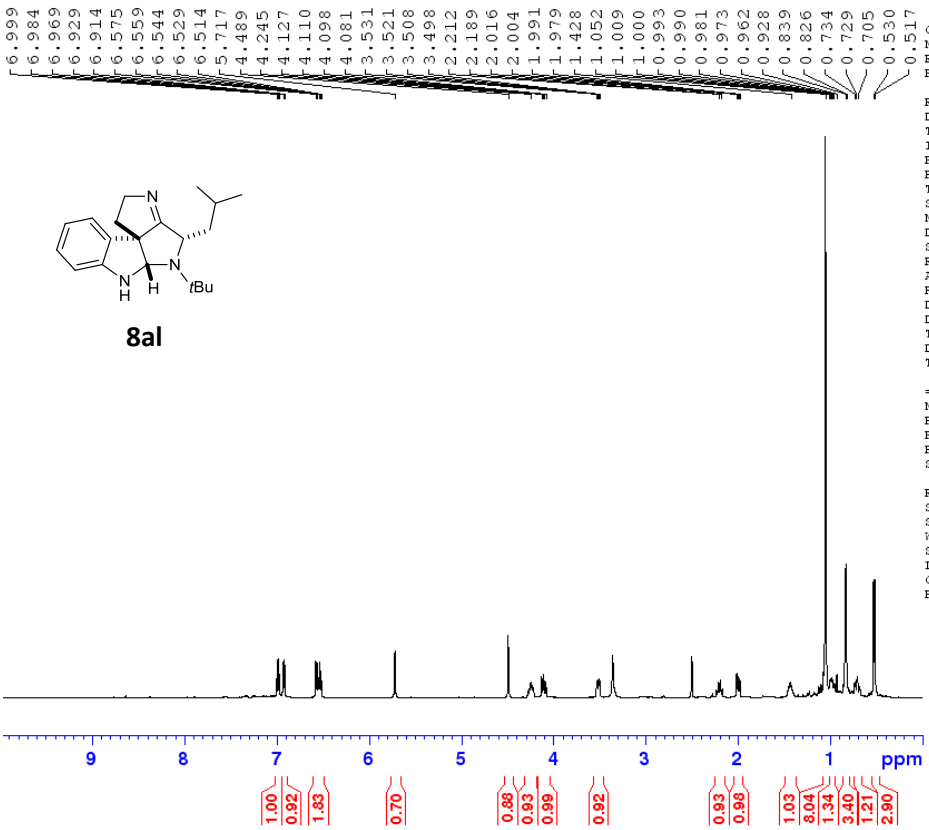
```
Current Data Parameters
NAME                  JMS-289B
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20160909
Time                   11.10
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                jmod
TD                     65536
SOLVENT                CDCl3
NS                       256
DS                         4
SWH                29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                      2050
DW                   16.800 usec
DE                     6.50 usec
TE                    298.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                 2.00000000 sec
D20                0.00689655 sec
TD0                        1

======== CHANNEL f1 =======
NUC1                     13C
P1                    11.20 usec
P2                    22.40 usec
PL1                   -2.00 dB
PL1W              88.77790070 W
SFO1             125.8055709 MHz

======== CHANNEL f2 =======
CPDPRG[2              waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  25.28 dB
PL2W               8.72000027 W
PL12W              0.06494062 W
SFO2             500.2720011 MHz

F2 - Processing parameters
SI                        32768
SF               125.7929933 MHz
WDW                          EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```




```
Current Data Parameters
NAME                  JMS-289A2
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160909
Time                      17.42
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1719425 sec
RG                          10
DW                       48.400 use
DE                        6.50 use
TE                       298.3 K
D1                  1.00000000 sec
TD0                          1

--------- CHANNEL f1 -------
NUC1                        1H
P1                        6.70 use
PL1                       4.00 dB
PL1W                8.72000027 W
SFO1               500.2730894 MHz

F2 - Processing parameters
SI                       32768
SF                 500.2700082 MHz
WDW                         EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
```

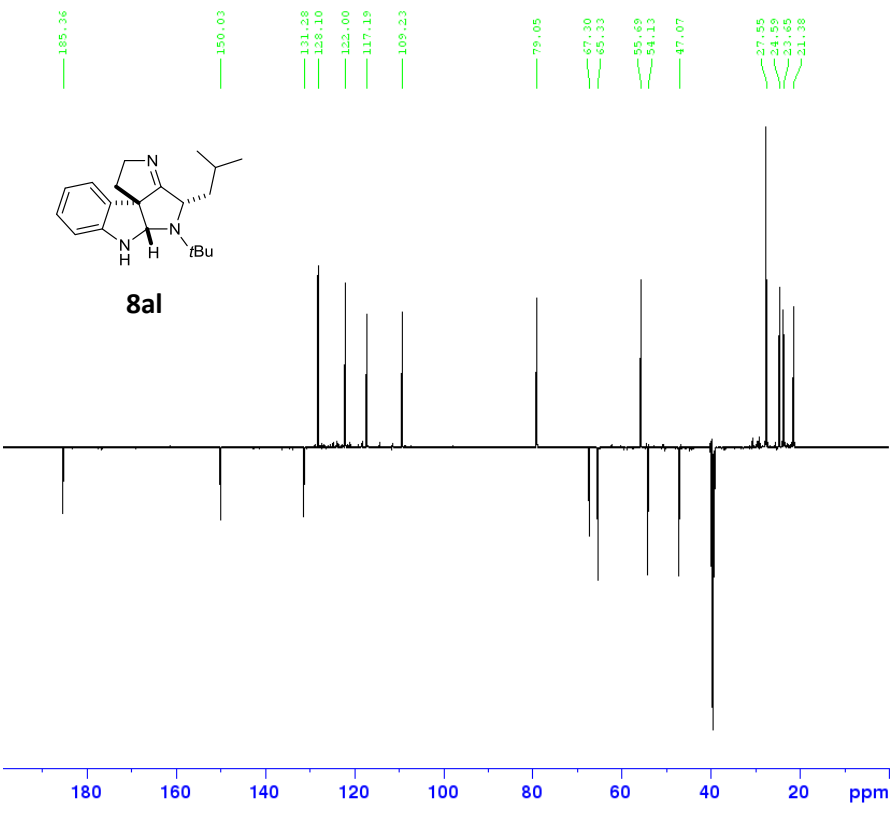



```
Current Data Parameters
NAME                  JMS-289A2
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160909
Time                      17.56
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                   jmod
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        2050
DW                      16.800 usec
DE                        6.50 usec
TE                       298.2 K
CNST2                145.0000000
CNST11                 1.0000000
D1                  2.00000000 sec
D20                 0.00689655 sec
TD0                          1

--------- CHANNEL f1 --------
NUC1                        13C
P1                       11.20 usec
P2                       22.40 usec
PL1                      -2.00 dB
PL1W               88.77790070 W
SFO1              125.8055709 MHz

--------- CHANNEL f2 --------
CPDPRG[2              waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       4.00 dB
PL12                     25.28 dB
PL2W                8.72000027 W
PL12W               0.06494062 W
SFO2               500.2720011 MHz

F2 - Processing parameters
SI                       32768
SF                 125.7929894 MHz
WDW                         EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```



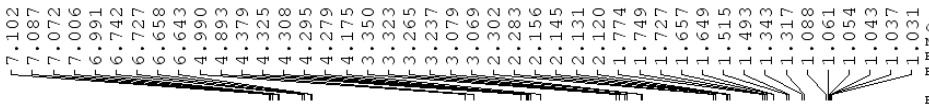
8al

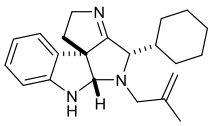
```
Current Data Parameters
NAME      BOR-047-3_DMSO
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160726
Time               9.58
INSTRUM           spect
PROBHD   5 mm CPTCI 1H-
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  16
DS                   2
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719425 sec
RG                12.7
DW              48.400 usec
DE               6.50 usec
TE              298.1 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               6.70 usec
PL1              4.00 dB
PL1W         8.72000027 W
SFO1        500.2730894 MHz

F2 - Processing parameters
SI                 32768
SF          500.2700000 MHz
WDW                   EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



8al

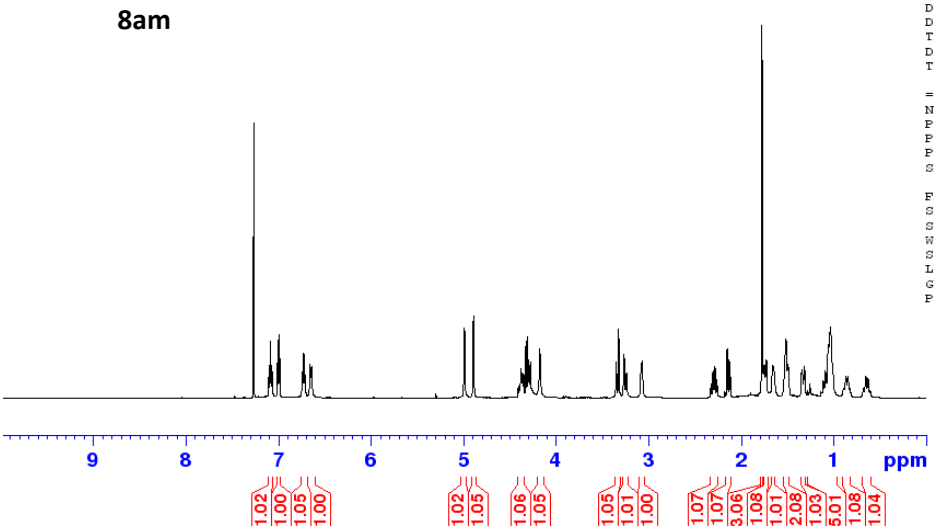
```
Current Data Parameters
NAME      BOR-047-3_DMSO
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160726
Time               5.29
INSTRUM           spect
PROBHD   5 mm CPTCI 1H-
PULPROG           jmod
TD               65536
SOLVENT           DMSO
NS                1024
DS                   4
SWH          29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG                2050
DW              16.800 usec
DE               6.50 usec
TE              298.2 K
CNST2       145.0000000
CNST11        1.0000000
D1            2.00000000 sec
D20           0.00689655 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              11.20 usec
P2              22.40 usec
PL1             -2.00 dB
PL1W        88.77790070 W
SFO1       125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            80.00 usec
PL2               4.00 dB
PL12             25.28 dB
PL2W         8.72000027 W
PL12W        0.06494062 W
SFO2        500.2720011 MHz

F2 - Processing parameters
SI                 32768
SF          125.7930467 MHz
WDW                   EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

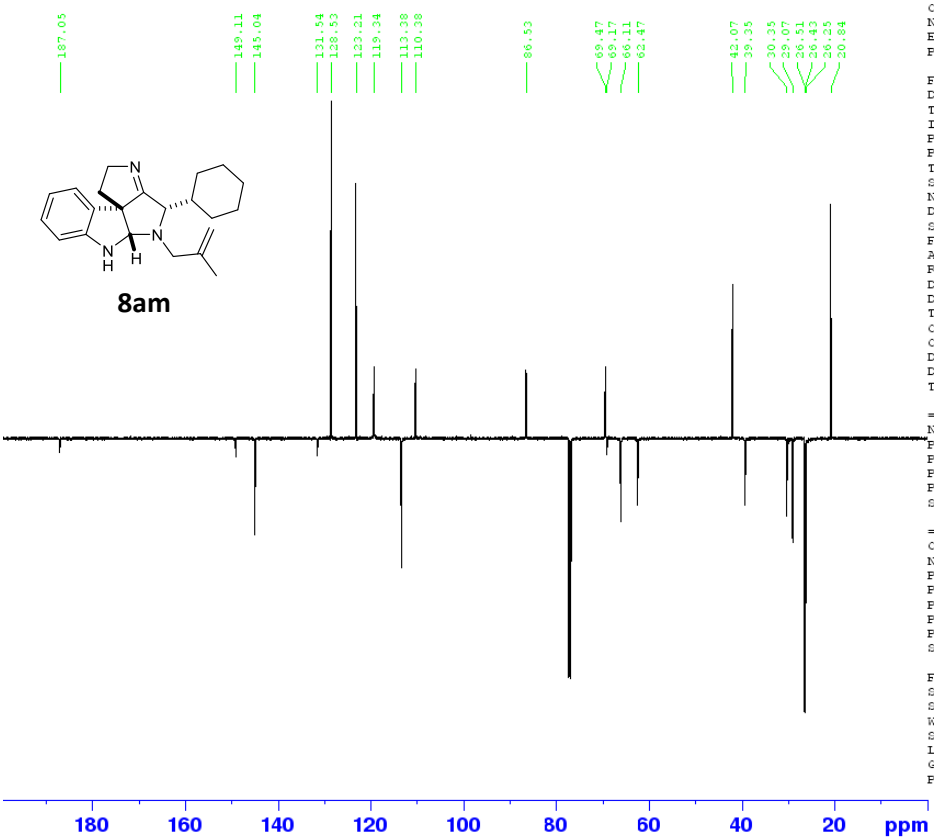




8am

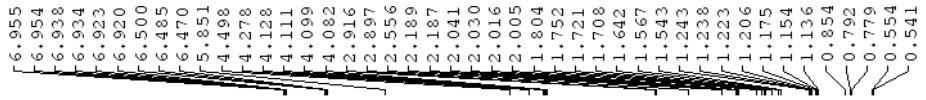
```
Current Data Parameters
NAME                 BOR-034
EXPNO                      5
PROCNO                     1

F2 - Acquisition Parameters
Date_               20160701
Time                   23.39
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES            0.315264 Hz
AQ                1.5859712 sec
RG                     22.6
DW                   48.400 usec
DE                    6.50 usec
TE                   298.2 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.70 usec
PL1                    4.00 dB
PL1W            8.72000027 W
SFO1           500.2730894 MHz

F2 - Processing parameters
SI                    16384
SF              500.2700041 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

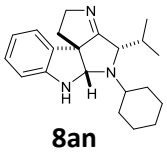




8am

```
Current Data Parameters
NAME                 BOR-034
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_               20160701
Time                   23.38
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                jmod
TD                    65536
SOLVENT               CDCl3
NS                       512
DS                         4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010048 sec
RG                     2050
DW                   16.800 usec
DE                    6.50 usec
TE                   298.2 K
CNST2          145.0000000
CNST11           1.0000000
D1                2.00000000 sec
D20               0.00689655 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
P2                    22.40 usec
PL1                   -2.00 dB
PL1W           88.77790070 W
SFO1          125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  25.28 dB
PL2W            8.72000027 W
PL12W           0.06494062 W
SFO2           500.2720011 MHz

F2 - Processing parameters
SI                    32768
SF              125.7929800 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```





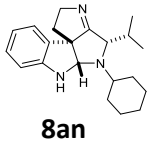
8an

Current Data Parameters
NAME JMS-287B1
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160909
Time 10.33
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 14.2
DW 48.400 use
DE 6.50 use
TE 298.3 K
D1 1.00000000 sec
TD0 1

-------- CHANNEL f1 -------
NUC1 1H
P1 6.70 use
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2730894 MHz

F2 - Processing parameters
SI 32768
SF 500.2699995 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



8an

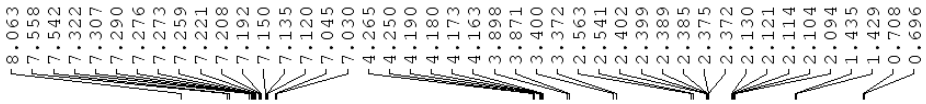
Current Data Parameters
NAME JMS-287B1
EXPNO 4
PROCNO 1

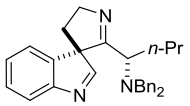
F2 - Acquisition Parameters
Date_ 20160908
Time 2.28
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1

-------- CHANNEL f1 --------
NUC1 13C
P1 11.20 usec
P2 22.40 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.8055709 MHz

-------- CHANNEL f2 --------
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
SFO2 500.2720011 MHz

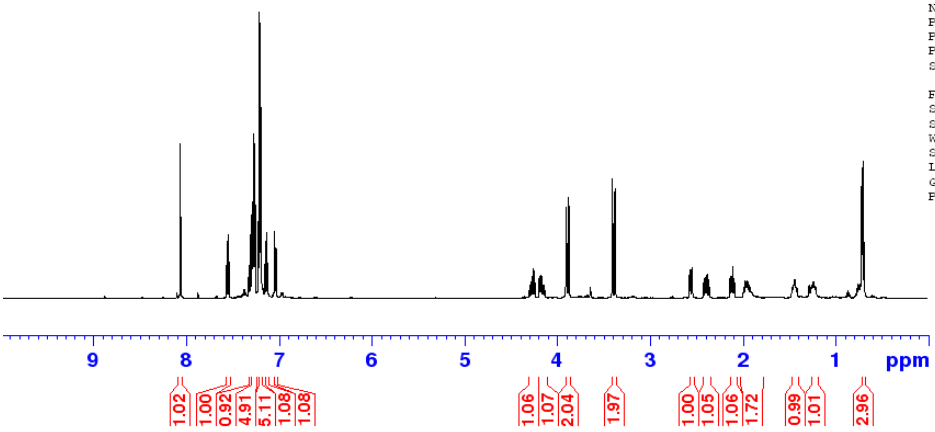
F2 - Processing parameters
SI 32768
SF 125.7930473 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

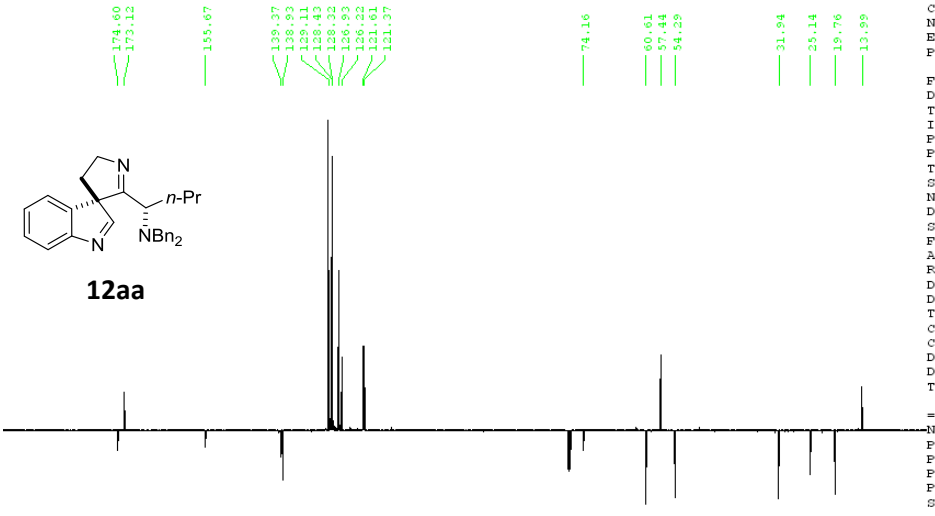




12aa

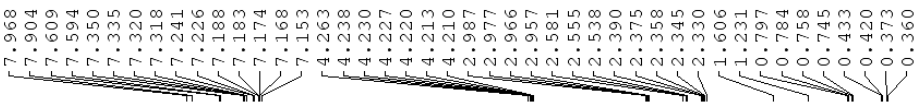
```
Current Data Parameters
NAME            BOR-030-2
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160701
Time                22.37
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719425 sec
RG                  14.2
DW                48.400 usec
DE                 6.50 usec
TE                298.3 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1        500.2730894 MHz

F2 - Processing parameters
SI                  32768
SF          500.2700000 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

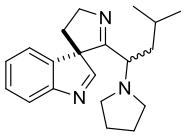




12aa

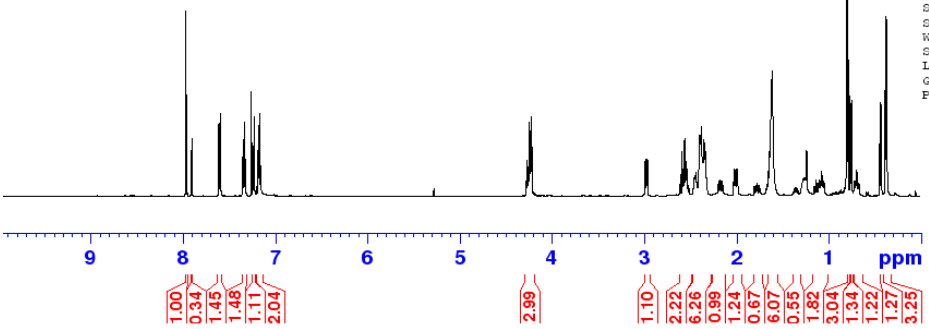
```
Current Data Parameters
NAME            BOR-030-2
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160701
Time                23.05
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                  2050
DW                16.800 usec
DE                 6.50 usec
TE                298.2 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                 11.20 usec
P2                 22.40 usec
PL1                -2.00 dB
PL1W         88.77790070 W
SFO1        125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2        500.2720011 MHz

F2 - Processing parameters
SI                  32768
SF          125.7929818 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

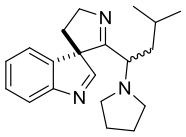




12ab

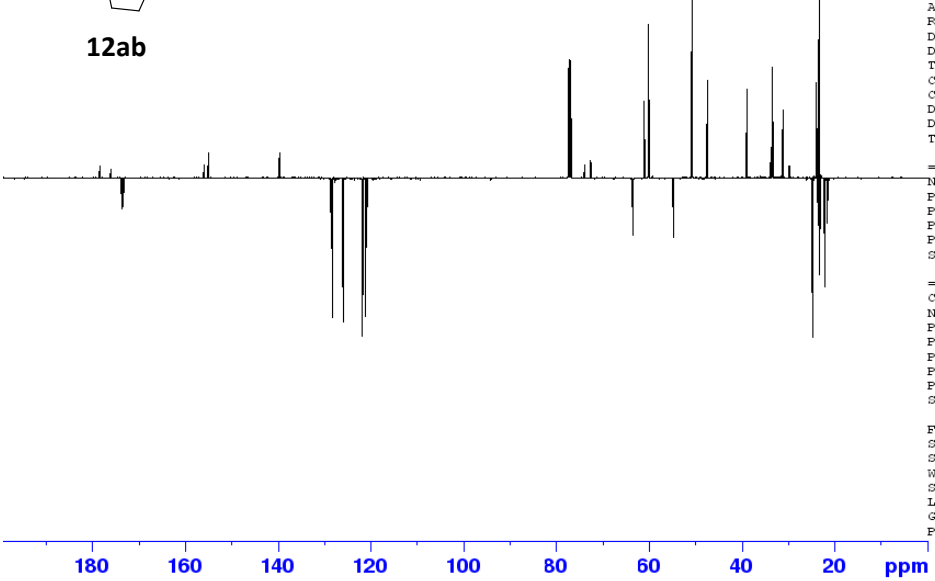
```
Current Data Parameters
NAME                  JMS-243A
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20160615
Time                   20.33
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                      12.7
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        6.70 usec
PL1                       4.00 dB
PL1W               8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                       32768
SF                500.2700085 MHz
WDW                         EM
SSB             0
LB                        0.30 Hz
GB              0
PC                        1.00
```

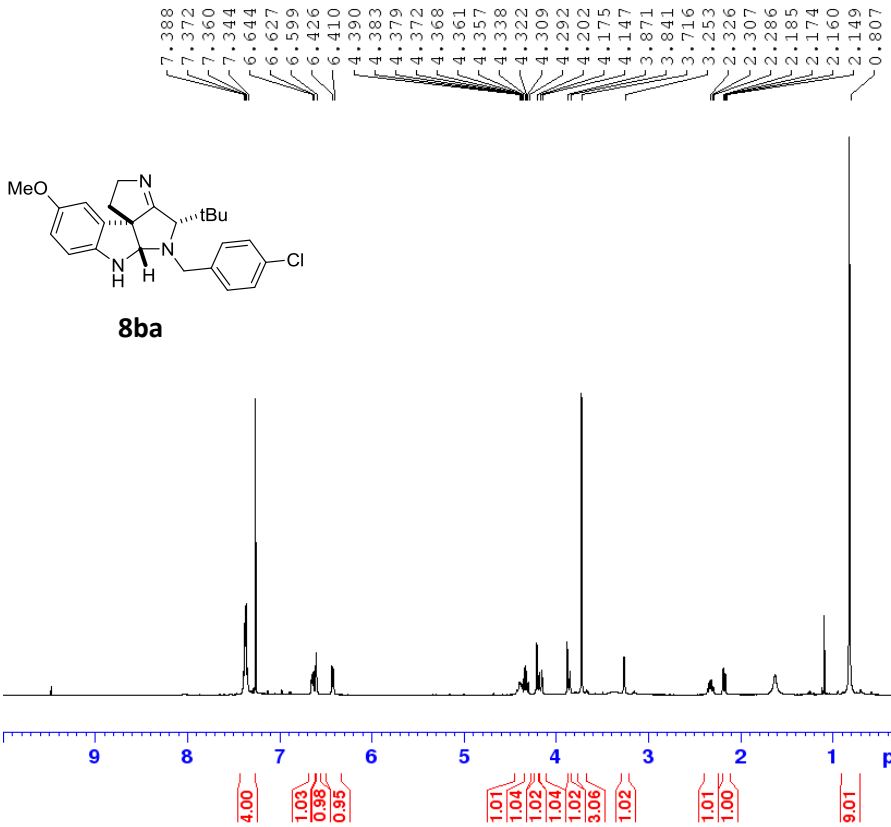




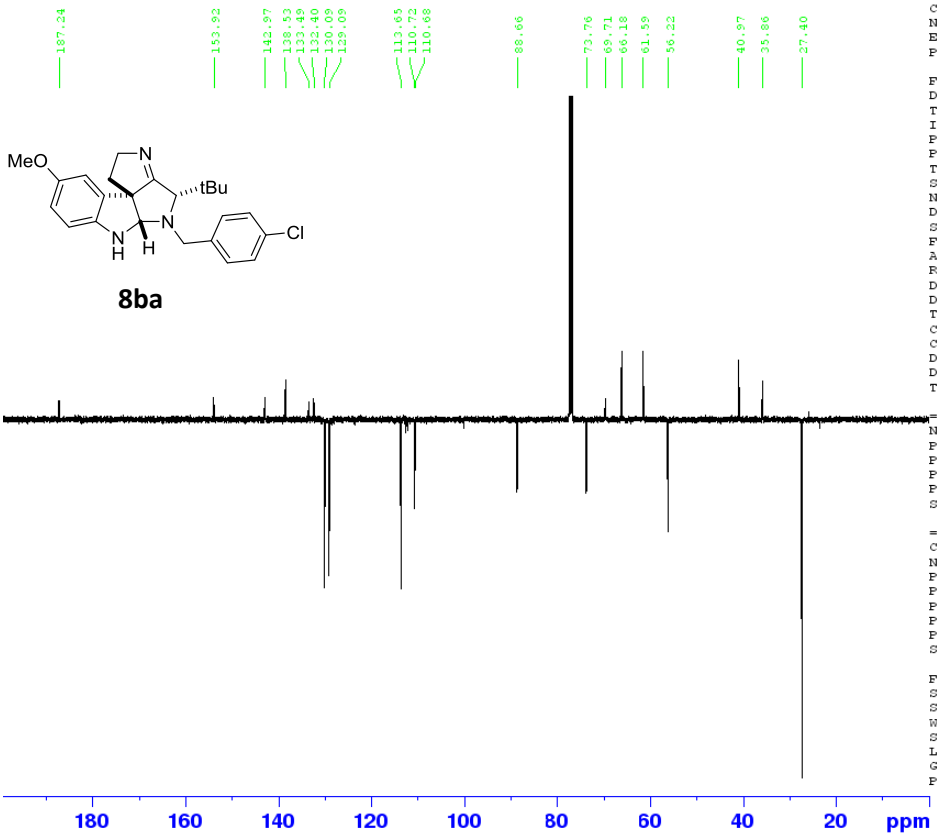
12ab

```
Current Data Parameters
NAME                  JMS-243A
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20160615
Time                   21.27
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                jmod
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                      2050
DW                    16.800 usec
DE                     6.50 usec
TE                    298.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                2.00000000 sec
D20               0.00689655 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       11.20 usec
P2                       22.40 usec
PL1                      -2.00 dB
PL1W              88.77790070 W
SFO1             125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       4.00 dB
PL12                     25.28 dB
PL2W               8.72000027 W
PL12W              0.06494062 W
SFO2             500.2720011 MHz

F2 - Processing parameters
SI                       32768
SF                125.7929809 MHz
WDW                         EM
SSB             0
LB                        1.00 Hz
GB              0
PC                        1.40
```

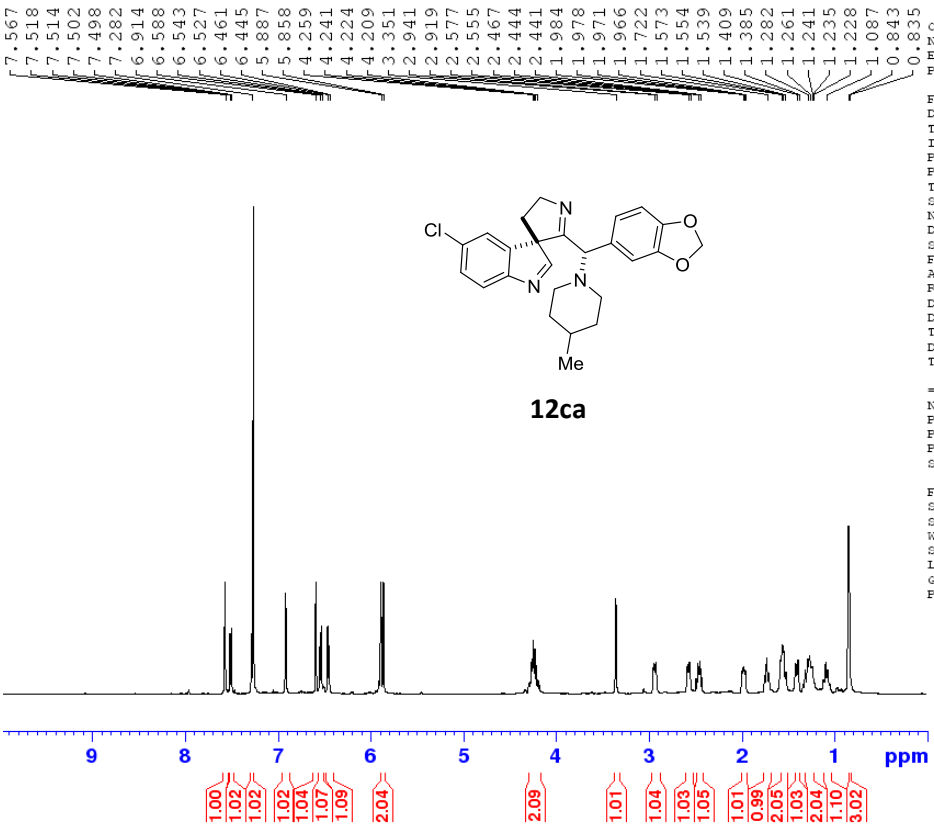




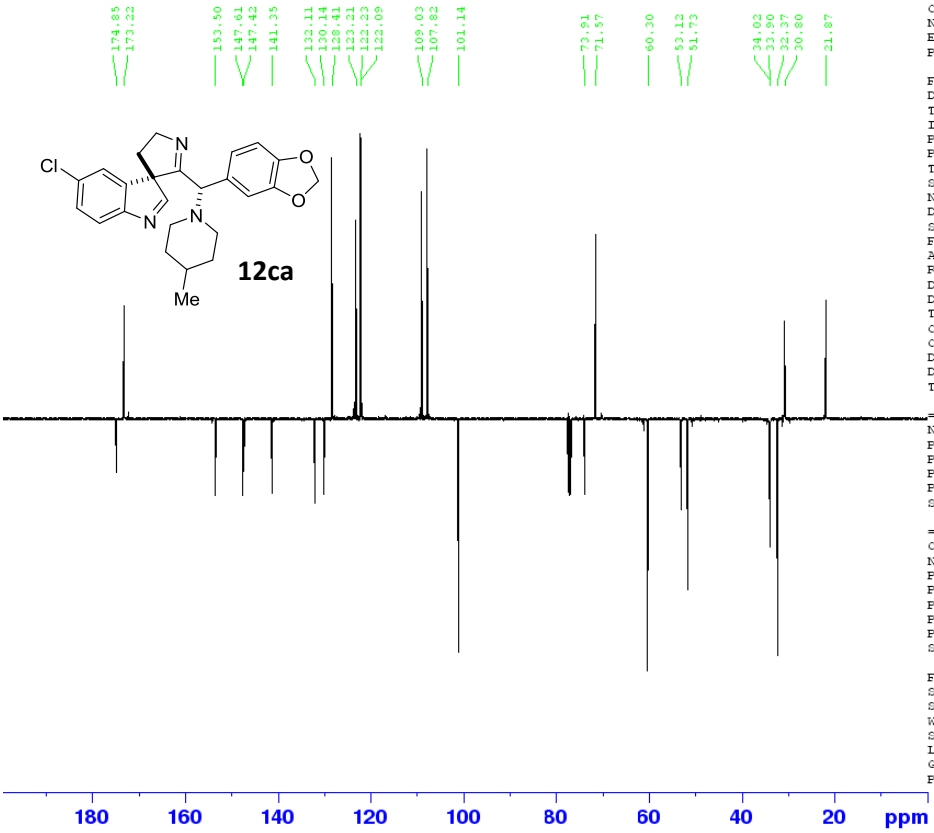

```
Current Data Parameters
NAME                  JMS-270
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160709
Time                    5.24
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719425 sec
RG                         18
DW                   48.400 usec
DE                    6.50 usec
TE                   298.3 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    6.70 usec
PL1                   4.00 dB
PL1W           8.72000027 W
SFO1          500.2730894 MHz

F2 - Processing parameters
SI                      32768
SF             500.2700085 MHz
WDW                        EM
SSB         0
LB                    0.30 Hz
GB          0
PC                    1.00
```



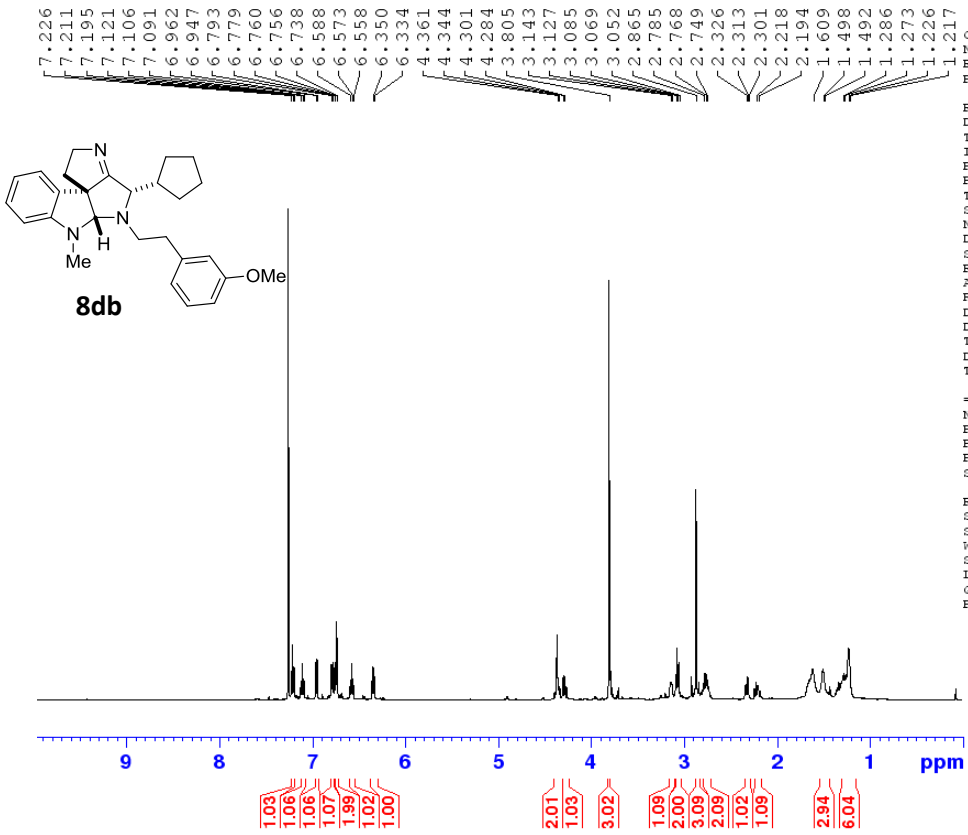

```
Current Data Parameters
NAME                  JMS-270
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160709
Time                    6.17
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 jmod
TD                     65536
SOLVENT                CDCl3
NS                       1000
DS                          4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010048 sec
RG                       2050
DW                   16.800 usec
DE                    6.50 usec
TE                   298.2 K
CNST2          145.0000000
CNST11           1.0000000
D1                2.00000000 sec
D20               0.00689655 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                   11.20 usec
P2                   22.40 usec
PL1                  -2.00 dB
PL1W          88.77790070 W
SFO1         125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                       1H
PCPD2                80.00 usec
PL2                   4.00 dB
PL12                 25.28 dB
PL2W           8.72000027 W
PL12W          0.06494062 W
SFO2          500.2720011 MHz

F2 - Processing parameters
SI                      32768
SF             125.7929764 MHz
WDW                        EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```



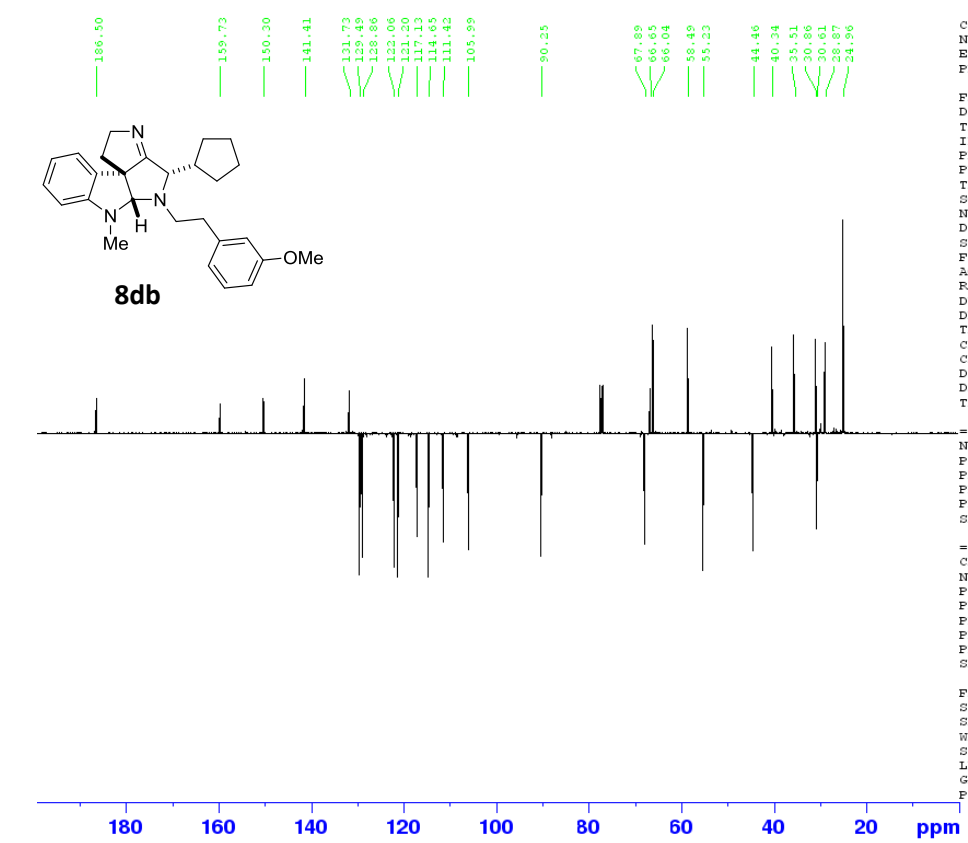

```
Current Data Parameters
NAME            BOR-039-4
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160626
Time                20.49
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                    14.2
DW                48.400 usec
DE                 6.50 usec
TE                298.3 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1         500.2730894 MHz

F2 - Processing parameters
SI                   32768
SF           500.2700085 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



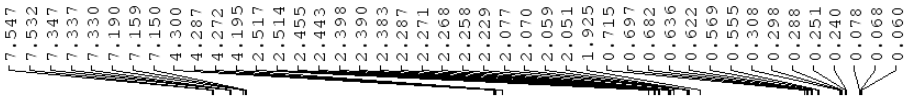

```
Current Data Parameters
NAME            BOR-039-4
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160626
Time                21.17
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              jmod
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                    2050
DW                16.800 usec
DE                 6.50 usec
TE                298.2 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
P2                 22.40 usec
PL1                -2.00 dB
PL1W          88.77790070 W
SFO1         125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2         500.2720011 MHz

F2 - Processing parameters
SI                   32768
SF           125.7929808 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

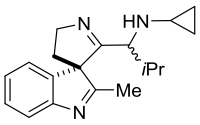



```
Current Data Parameters
NAME            BOR-039-6
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160711
Time                19.04
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      2
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                     18
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  6.70 usec
PL1                 4.00 dB
PL1W          8.72000027 W
SFO1         500.2730894 MHz

F2 - Processing parameters
SI                  32768
SF            500.2700082 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



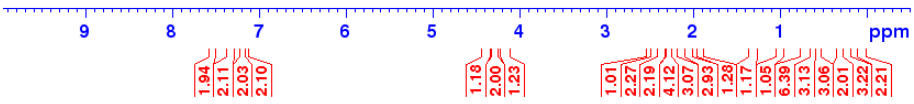

```
Current Data Parameters
NAME            BOR-039-6
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160727
Time                14.08
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                  65536
SOLVENT             CDCl3
NS                    256
DS                      4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                   2050
DW                16.800 usec
DE                 6.50 usec
TE                298.3 K
CNST2          145.0000000
CNST11           1.0000000
D1               2.00000000 sec
D20              0.00689655 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                 11.20 usec
P2                 22.40 usec
PL1                -2.00 dB
PL1W          88.77790070 W
SFO1         125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2         500.2720011 MHz

F2 - Processing parameters
SI                  32768
SF            125.7929879 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

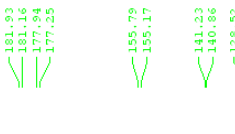


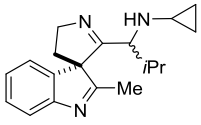


12ea

```
Current Data Parameters
NAME            BOR-040-1
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160626
Time               19.09
INSTRUM            spect
PROBHD    5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                  12.7
DW                48.400 usec
DE                 6.50 usec
TE                298.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 6.70 usec
PL1                4.00 dB
PL1W          8.72000027 W
SFO1          500.2730894 MHz

F2 - Processing parameters
SI                 32768
SF            500.2700085 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

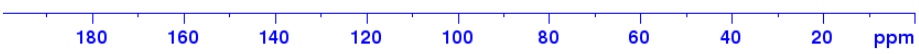


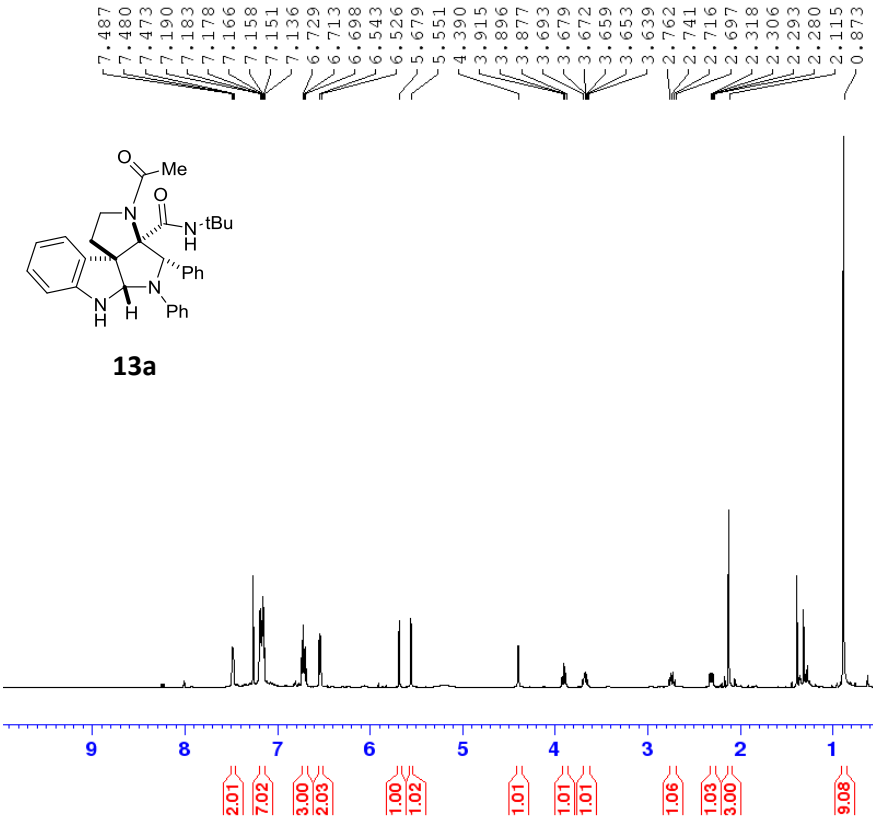




12ea

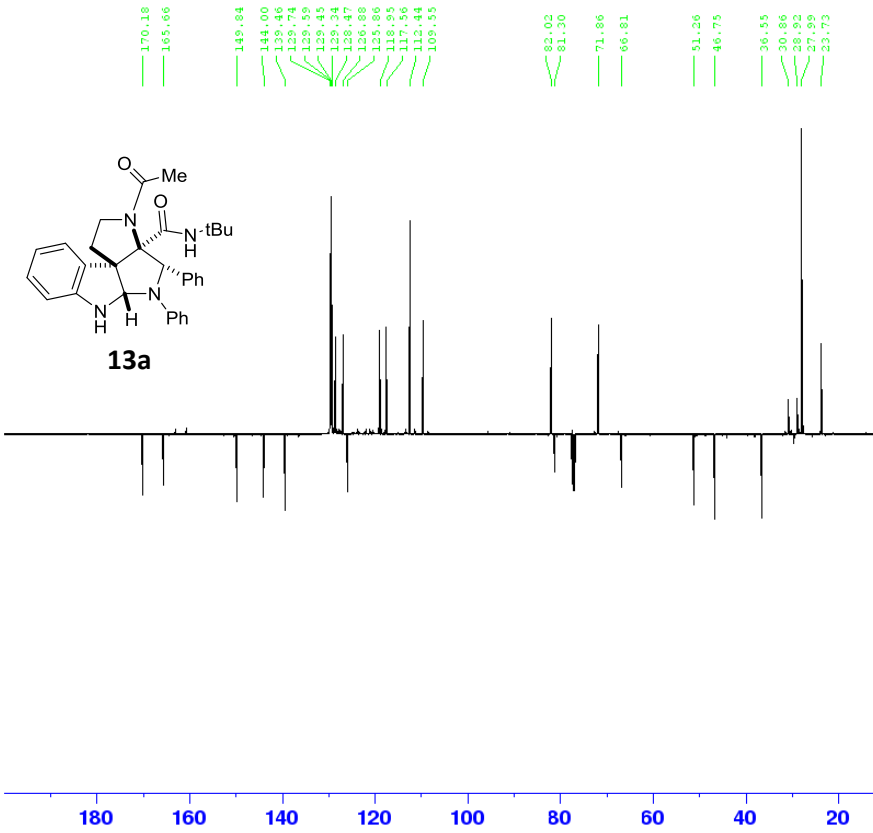
```
Current Data Parameters
NAME            BOR-040-1
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160626
Time               19.37
INSTRUM            spect
PROBHD    5 mm CPTCI 1H-
PULPROG             jmod
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                  2050
DW                16.800 usec
DE                 6.50 usec
TE                298.2 K
CNST2         145.0000000
CNST11          1.0000000
D1             2.00000000 sec
D20            0.00689655 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                11.20 usec
P2                22.40 usec
PL1               -2.00 dB
PL1W          88.77790070 W
SFO1          125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                4.00 dB
PL12              25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2          500.2720011 MHz

F2 - Processing parameters
SI                 32768
SF            125.7929920 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





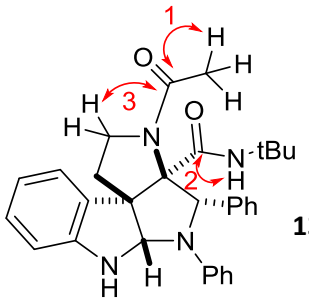
13a

```
Current Data Parameters
NAME                  JMS-237
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160722
Time                    1.42
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                         9
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     6.70 usec
PL1                    4.00 dB
PL1W            8.72000027 W
SFO1           500.2730894 MHz

F2 - Processing parameters
SI                     32768
SF              500.2700081 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

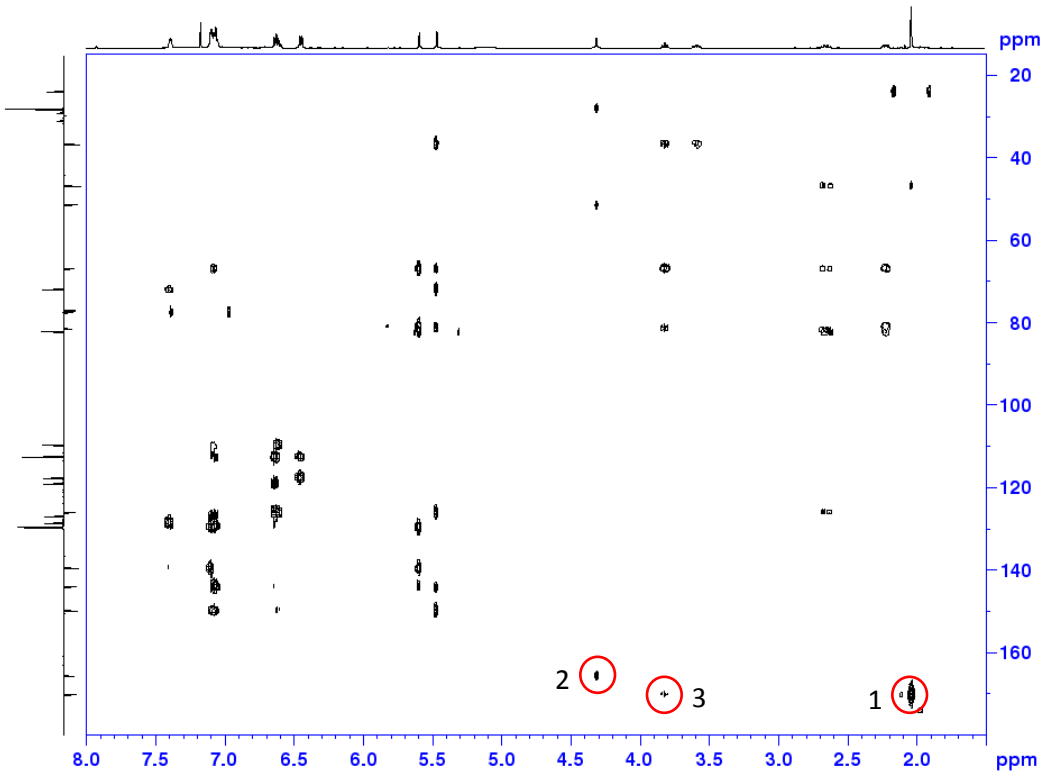


13a

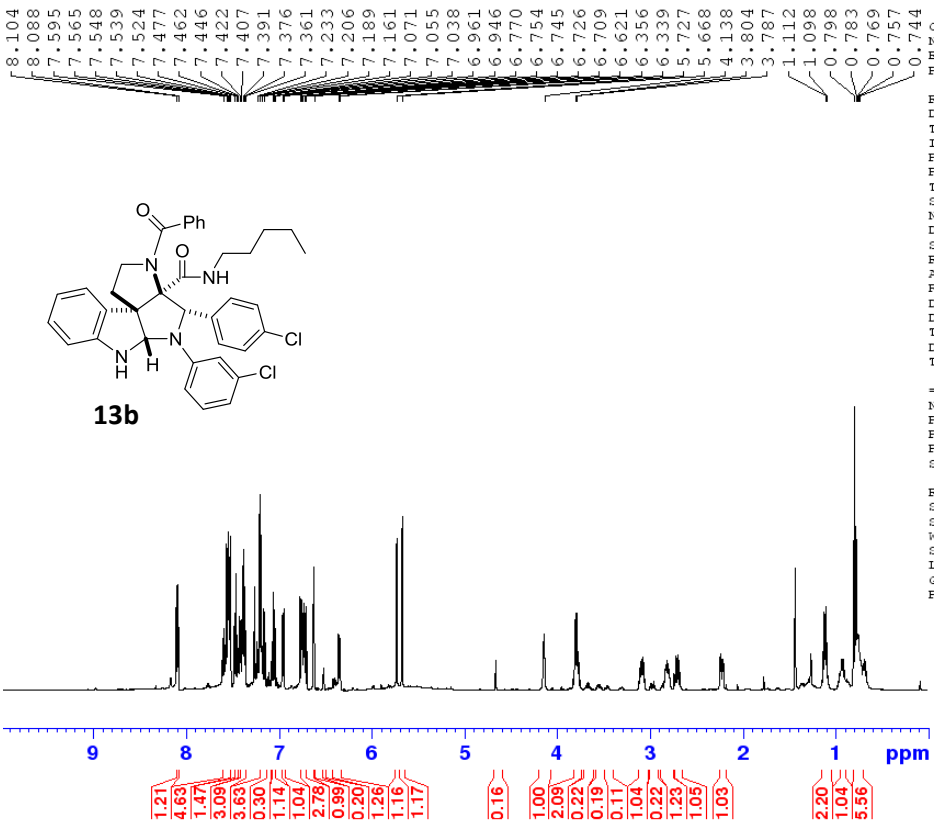
```
Current Data Parameters
NAME                  JMS-237
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160626
Time                   16.35
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                jmod
TD                     65536
SOLVENT                CDCl3
NS                       512
DS                         4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                      2050
DW                    16.800 usec
DE                     6.50 usec
TE                    298.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
D20               0.00689655 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
P2                    22.40 usec
PL1                   -2.00 dB
PL1W           88.77790070 W
SFO1          125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  25.28 dB
PL2W            8.72000027 W
PL12W           0.06494062 W
SFO2           500.2720011 MHz

F2 - Processing parameters
SI                     32768
SF              125.7929946 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



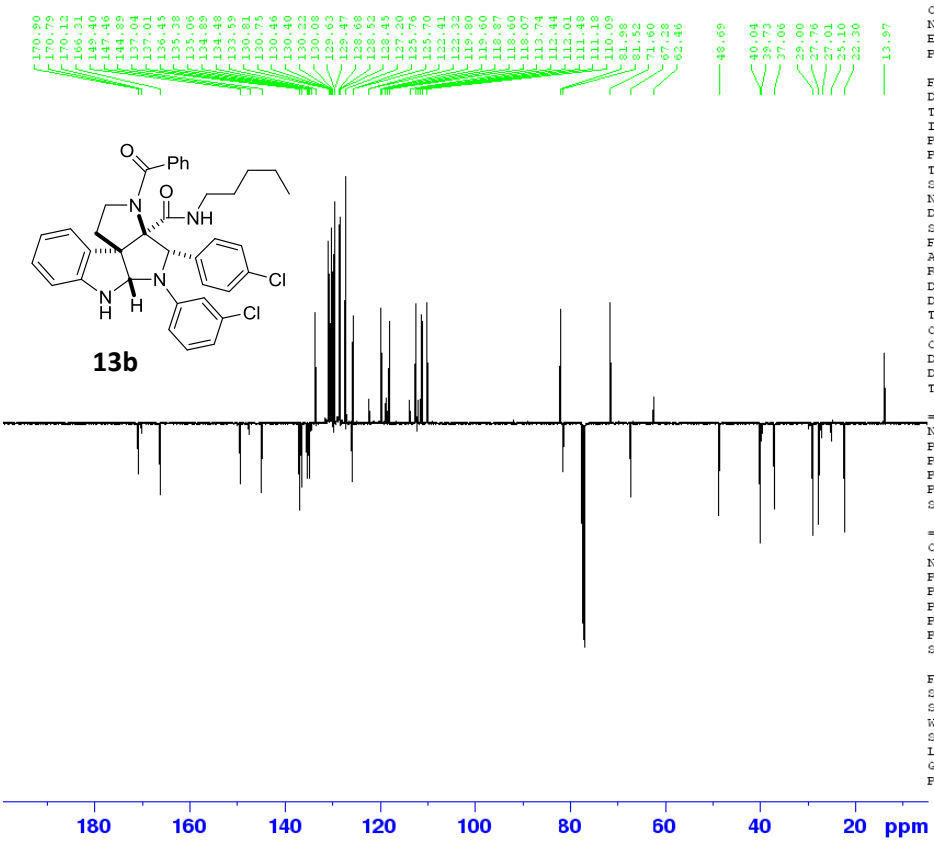
13a




```
Current Data Parameters
NAME                  JH2-237
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160722
Time                   1.59
INSTRUM                spect
PROBHD     5 mm CPTCI 1H-
PULPROG    hmbcgplpndqf
TD                     4096
SOLVENT                CDCl3
NS                        8
DS                       16
SWH                5882.353 Hz
FIDRES             1.436181 Hz
AQ                 0.3481600 sec
RG                      2050
DW                   85.000 usec
DE                    6.50 usec
TE                  298.3 K
CNST2              145.0000000
CNST13              10.0000000
D0                 0.00000200 sec
D1                 1.46441362 sec
D2                 0.00344828 sec
D6                 0.05000000 sec
D16                0.00020000 sec
ID0                0.00001790 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       6.70 usec
P2                      13.40 usec
PL1                      4.00 dB
PL1W               8.72000027 W
SFO1               500.2719276 MHz

======== CHANNEL f2 ========
NUC2                      13C
P3                      11.20 usec
PL2                     -2.00 dB
PL2W              88.77750670 W
SFO2               125.8055568 MHz

====== GRADIENT CHANNEL =====
GPNAM[1]             SINE.100
GPNAM[2]             SINE.100
GPNAM[3]             SINE.100
GPZ1                    50.00 %
GPZ2                    30.00 %
GPZ3                    40.10 %
P16                   1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1               125.8056 MHz
FIDRES             436.574860 Hz
SW                 222.095 ppm
FnMODE                     QF

F2 - Processing parameters
SI                       2048
SF                 500.2700493 MHz
WDW                      SINE
SSB                         0
LB                      0 Hz
GB                          0
PC                        1.40

F1 - Processing parameters
SI                       1024
MC2                        QF
SF                 125.7929945 MHz
WDW                      SINE
SSB                         0
LB                      0 Hz
GB                          0
```




```
Current Data Parameters
NAME                  JMS-273
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160723
Time                   19.44
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719425 sec
RG                      14.2
DW                    48.400 usec
DE                     6.50 usec
TE                    298.3 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                       6.70 usec
PL1                      4.00 dB
PL1W               8.72000027 W
SFO1             500.2730894 MHz

F2 - Processing parameters
SI                       32768
SF               500.2700082 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```




```
Current Data Parameters
NAME                  JMS-273
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20160722
Time                    9.26
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 jmod
TD                     65536
SOLVENT                CDCl3
NS                        512
DS                          4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010048 sec
RG                      2050
DW                    16.800 usec
DE                     6.50 usec
TE                    298.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 2.00000000 sec
D20                0.00689655 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      11.20 usec
P2                      22.40 usec
PL1                     -2.00 dB
PL1W              88.77790070 W
SFO1             125.8055709 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     4.00 dB
PL12                   25.28 dB
PL2W               8.72000027 W
PL12W              0.06494062 W
SFO2             500.2720011 MHz

F2 - Processing parameters
SI                       32768
SF               125.7929810 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

References

(1) A. V Lygin and A. de Meijere, *Eur. J. Org. Chem.* 2009, **30**, 5138.
(2) T. D. Montgomery, A. E. Nibbs, Y. Zhu and V. H.Rawal, *Org. Lett.* 2014, **16**, 3480.
(3) J. M. Allen and T. H. Lambert, *J. Am. Chem. Soc*, 2011, **133**, 1260–1262.
(4) G.M. Sheldrick, *Acta Cryst.* 2008, **A64**, 112.
(5) C. B. Hübschle, G. M. Sheldrick and B. Dittrich, *J. Appl. Cryst.* 2011, **44**, 1281.