Supporting Information

Expanding the "aplysinospin cascade" through DNA-templated [2+2] photocycloaddition

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1. Materials and methods

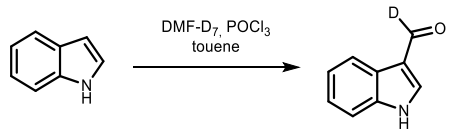
All reactions were carried out under air atmosphere. All reagent-grade chemicals and other solvents were obtained from commercial suppliers and were used as received. Microwave irradiation was performed using an Anton-Paar monowave 200 oven using 30 mL pressurized vials. **Analytical TLC** was performed with Merck silica gel plates, pre-coated with silica gel 60 F254 (0.2 mm). Visualisation was effected by quenching of UV fluorescence (λ_{max} = 254 nm or 360 nm) and by staining with a vanillin TLC stain solution, followed by heating. **Flash column chromatography** employed VWR (230–400 mesh) silica gel. **NMR spectra** were recorded at 298 K using a Bruker AVANCE 400 spectrometer or a 800 MHz Bruker AVANCE III spectrometer. ^{1}H NMR spectra were recorded at 400 MHz or at 800 MHz and residual solvent peaks were used as an internal reference (methanol-d_4 δ 3.31, DMSO-d_6 δ 2.50). Data are reported as follows: chemical shift in ppm, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of non equivalent resonances), coupling constants, and integration. ^{13}C NMR spectra were recorded at 101 MHz or at 200 MHz and residual solvent peaks were used as an internal reference (methanol-d_4 δ 49, DMSO-d_6 δ 39). Data are reported as follows: chemical shift in ppm, multiplicity deduced from DEPT experiments (CH_3, CH_2, CH and C_q). **IR spectra** were recorded on a Vector 22 Bruker spectrometer or with an IRAffinity-1S Shimadzu spectrometer and are reported in frequency of absorption at the peak maximum (cm^{-1}). **Melting points** were determined in open glass capillaries and are uncorrected. **Low resolution mass spectra** were recorded on an Agilent 1100 series LC-MS (with a 6310 ion trap) under electrospray ionisation (ESI). **High resolution mass spectra and LC/MS** were run using a Thermoquest TLM LCQ Deca ion-trap spectrometer with a Sunfire® analytical C_{18} column (150 × 2.1 mm; 3.5 µm, Waters). **High-pressure liquid chromatography** analyses were carried out on a Waters Delta Prep equipped with a binary pump (Waters 2525), a UV-visible diode array detector (190-600 nm, Waters 2996). Xselect® analytical C_{18} column (150 × 2.1 mm; 3.5 µm, Waters) was used for preparative HPLC separations. (*E*)-aplysinopsin hydroiodide (**1a**), 3'-deimino-3'-oxo-aplysinopsin (**3**), 6-bromo (*E*)-aplysinopsin hydroiodide (**1c**), and 6-bromo-desmethyl-*(E)*-aplysinopsin (**1q**) were prepared according to previously reported procedures.[1] **Circular dichroism spectroscopy measurements** were performed on a Jasco 815 model spectrophotometer in 1 cm quartz cell at 5 °C. Prior to all measurements a buffer spectrum was recorded for blank correction.

[1] A. Skiredj, M. A. Beniddir, D. Joseph, K. Leblanc, G. Bernadat, E. Evanno and E. Poupon, *Angew. Chem. Int. Ed.*, 2014, **53**, 6419.

2. Experimental and Spectral data

2.1. Synthesis of aplysinopsine-type monomers

1*H*-Indole-3-carboxaldehyde-^{2}H



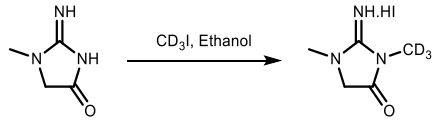
C_8H_7N
MW: 117,15 g/mol

C_9H_6DNO
MW: 146,17 g/mol

To a solution of DMF-d_7 (1.00 g, 12.5 mmol, 1 equiv.) in toluene (30 mL), $POCl_3$ (2.85 mL, 20.0 mMol, 1.6 equiv.) was added over 5 minutes. Then a solution of indole (1.76 g, 15 mmol, 2 equiv.) in toluene (5 mL) was added over 5 minutes. After 3 h of stirring, mixture was cooled to 0°C and quenched by an aqueous solution of NaOH (6M, 50 mL). Then mixture was refluxed for 5 min. After cooling, reaction mixture was partitioned between water (1 L) and diethyl ether (1 L). Organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. Crude product was recrystallized from a 1:1 mixture of petroleum ether and diethyl ether (60 mL) to afford the title product as a brown solid (1.00 g, 55 %).[2]

- **^{1}H NMR (300 MHz, DMSO-d_6)** δ 12.11 (s br, 1H), 8.28 (d, J = 3.1 Hz, 1H), 8.10 (dd, J = 7.9, 1.4 Hz, 1H), 7.57-7.47 (m, 1H), 7.24 (pd_{like}, J = 7.1, 1.4 Hz, 2H).
- **^{13}C NMR (75 MHz, DMSO-d_6)** δ 138.3, 137.0, 124.1, 123.4, 122.1, 120.8, 117.7, 112.4 (CDO not seen).
- **IR (neat)** ν_{max} = 3165, 3111, 1927, 1608, 1575, 1517, 1494, 1458, 1436, 1242, 1138, 1022.
- **HRMS ESI**: *m/z* $[M+H]^+$: 147.0665; calculated for $C_9{}^{1}H_7{}^{2}HNO^+$: 147.0663.

2-Imino-1-methyl-3-(methyl-$^{2}H_3$)imidazolidin-4-one hydroiodide



$C_4H_7N_3O$
MW: 113,12 g/mol

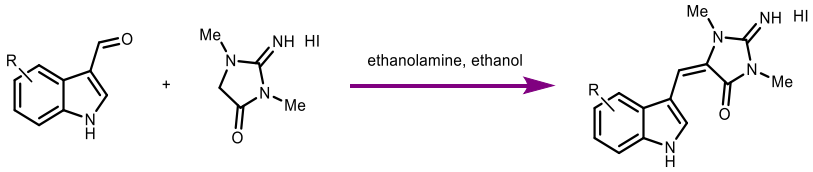
$C_5H_7D_3N_3OI$
MW: 258.08 g/mol

To a solution of MeI-d_3 (5.00 g, 34.5 mmol, 1.18 equiv.) in ethanol (50 mL), creatinine (3.21 g, 29.0 mmol, 1.0 equiv.) was added. Mixture was heated to reflux for 3 h. After cooling, reaction mixture was concentrated under reduced pressure. Crude product was suspended in acetonitrile (70 mL) and heated to reflux for 15 min. Suspension was filtered (at 70 °C) and filtrate was concentrated under reduced pressure to afford the title product as a white solid (5.56 g, 76 %).

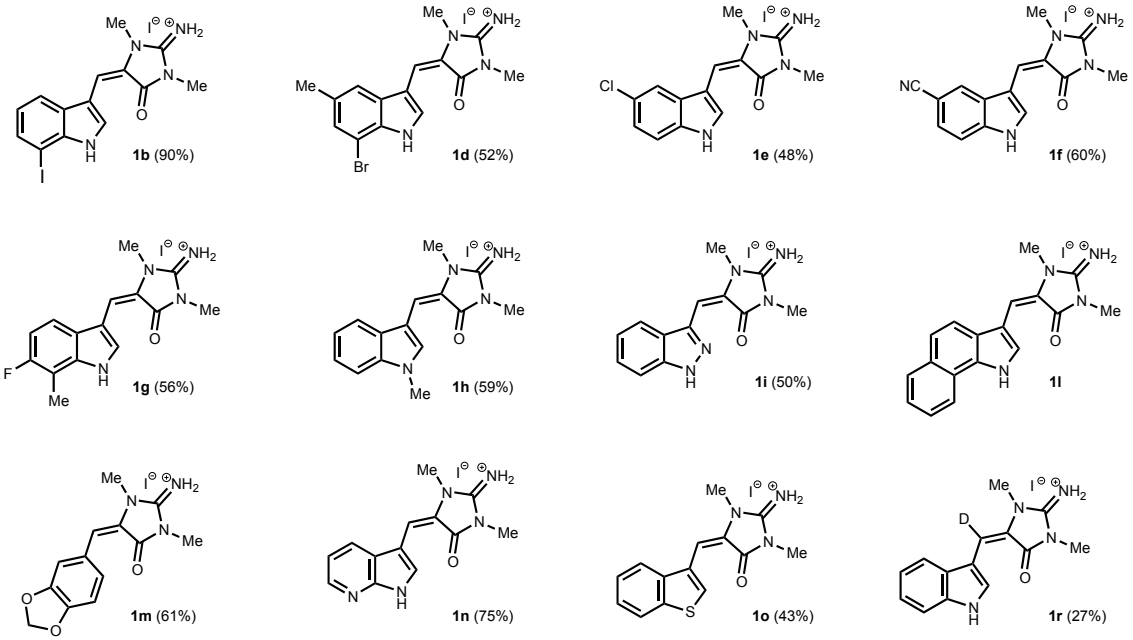
- **^{1}H NMR (300 MHz, DMSO-d_6)** δ 8.95 (s, 2H), 4.25 (s, 2H), 3.09 (s, 3H).
- **^{13}C NMR (75 MHz, DMSO-d_6)** δ 169.6, 157.7, 53.3, 31.8 (CD_3 not seen).
- **IR (neat)** ν_{max} = 3248, 3024, 1670, 1585, 1498, 1417, 1325, 1169, 1142, 1116, 1037, 840.
- **HRMS ESI**: *m/z* $[M+H]^+$: 131.1005; calculated for $C_5{}^{1}H_6{}^{2}H_5N_3^+$: 131.0007.

[2] B. Amir-Heidari, J. Thirlway and J. Micklefield, *Org. Lett.* 2007, **9**, 1513.

2.2. Procedure for the synthesis of aplysinopsine-type monomers in solution

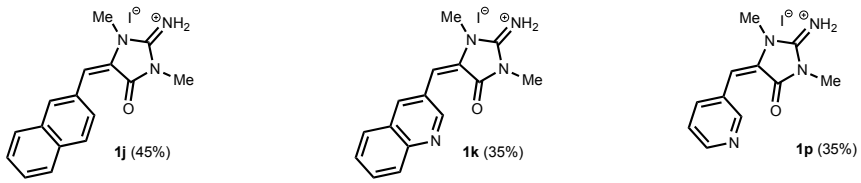


To a solution of the *N*-methylcreatinine hydroiodide (529 mg, 2.1 mmol, 1 equiv.) and the corresponding formylindole-type substrate (2.1 mmol, 1 equiv.) in EtOH (20 mL), ethanolamine (255 µL, 4.25 mmol, 2 equiv.) is added. The solution is heated to reflux for 2 h. After cooling, the product is collected by filtration is washed with Et_2O (3 x 5 mL).



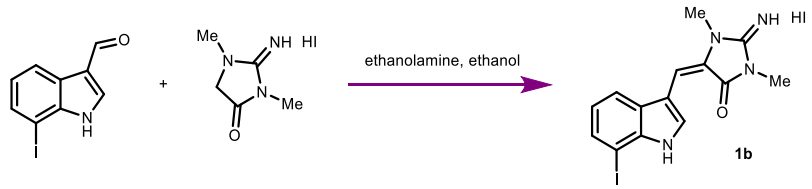
2.3. Neat Procedure for the synthesis of aplysinopsine-type monomers

N-Methylcreatinine hydroiodide (529 mg, 2.1 mmol, 1 equiv.) and the corresponding formyl indole-type substrate (2.1 mmol, 1 equiv.) were placed in a 100 mL round bottomed flask under a nitrogen atmosphere. The flask was then heated cautiously with a Bunsen burner. Considerable frothing of the mixture took place during the reaction. The reaction mixture was cooled to rt. The remaining solid was recrystallized from methanol.



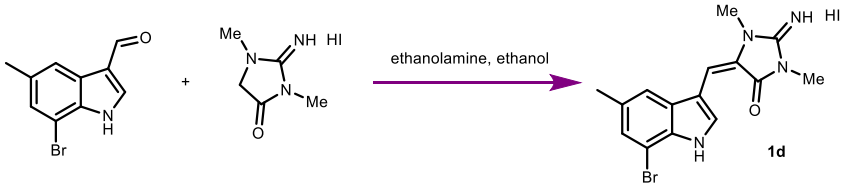
2.4. Description of aplysinopsine-type monomers

7-Iodo-(*E*)-aplysinopsin hydroiodide (1b)



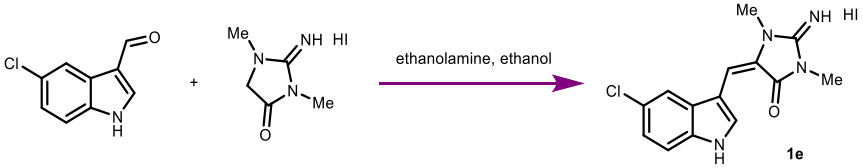
- **Yield:** 90%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 8.73 (d, 1H), 7.92 (dd, *J* = 8.0, 0.9 Hz, 1H), 7.55 (dd, *J* = 7.4, 0.9 Hz, 1H), 6.92 (dd, *J* = 7.9, 7.4 Hz, 1H), 6.38 (s, 1H), 3.26 (s, 3H), 3.07 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.1, 150.5, 137.3, 130.6, 128.2, 128.0, 127.1, 121.2, 118.3, 110.0, 102.3, 77.2, 27.0, 24.9.
- **IR (neat)** ν_{max} = 3329, 3261, 1728, 1653, 1631, 1498, 1425, 1396, 1197, 1070, 1055.
- **HRMS ESI**: *m/z* [M+H]$^+$: 381.0210; calculated for $C_{14}H_{13}IN_4O^+$: 381.0207.

7-Bromo-5-methyl-(*E*)-aplysinopsin hydroiodide (1d)



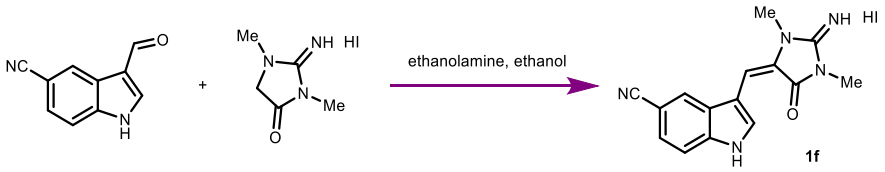
- **Yield:** 52%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 11.5 (brs, 1H), 8.70 (d, *J* = 2.6 Hz, 1H), 7.69 (s, 1H), 7.20 (s, 1H), 6.65 (brs, 1H), 6.33 (s, 1H), 3.25 (s, 3H), 3.06 (s, 3H), 2.42 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.2, 150.5, 132.3, 130.0, 129.4, 128.3, 127.0, 125.4, 117.4, 109.6, 103.9, 101.9, 26.9, 24.8, 20.8.
- **IR (neat)** ν_{max} = 3352, 2854, 1710, 1654, 1627, 1427, 1392, 1246, 1093, 974.
- **HRMS ESI**: *m/z* [M+H]$^+$: 347.0506; calculated for $C_{15}H_{15}Br^{79}Br^{81}N_4O^+$: 347.0502.

5-Chloro-(*E*)-aplysinopsin hydroiodide (1e)



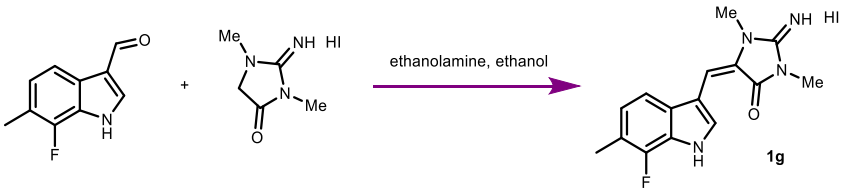
- **Yield:** 48%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 11.6 (brs, 1H), 8.74 (d, *J* = 2.5 Hz, 1H), 8.02 (d, *J* = 2.0 Hz, 1H), 7.43 (dd, *J* = 8.5, 0.5 Hz, 1H), 7.14 (dd, *J* = 8.6, 1.0 Hz, 1H), 6.44 (s, 1H), 3.26 (s, 3H), 3.06 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.2, 150.5, 134.0, 128.9, 128.7, 126.8, 124.3, 121.6, 117,7, 113.2, 108.8, 102.2, 27.0, 24,8.
- **IR (neat)** ν_{max} = 3331, 2987, 2900, 1735, 1653, 1456, 1425, 1388, 1328, 1190, 1074.
- **HRMS ESI**: *m/z* [M+H]$^+$: 289.0853; calculated for $C_{14}H_{13}ClN_4O^+$: 289.081.

5-Cyano-5-methyl-(*E*)-aplysinopsin hydroiodide (1f)



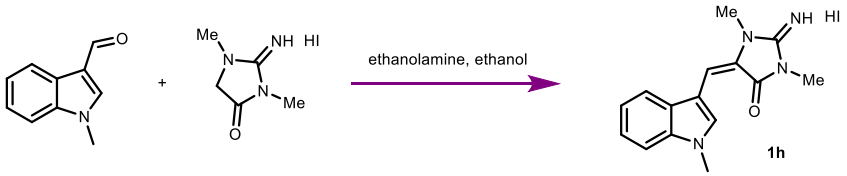
- **Yield:** 60%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 11.64 (brs, 1H), 8.89 (s, 1H), 8.08 (d, *J* = 8.3 Hz, 1H), 7.93 (dd, *J* = 1.5, 0.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 1.5 Hz, 1H), 6.70 (brs, 1H), 6.42 (s, 1H), 3.26 (s, 3H), 3.06 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.2, 150.4, 134.3, 130.9, 130.5, 127.7, 121.9, 120.4, 119.5, 116.6, 109.7, 103.0, 100.9, 26.9, 24.9.
- **IR (neat)** ν_{max} = 330, 2987, 2900, 2224, 1720, 1653, 1635, 1456, 1392, 1222, 1093.
- **HRMS ESI**: *m/z* [M+H]$^+$: 280.1195; calculated for $C_{15}H_{13}N_5O^+$: 280.1193.

7-Fluoro-6-methyl-(*E*)-aplysinopsin hydroiodide (1g)



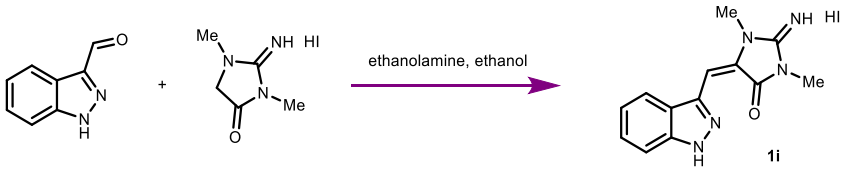
- **Yield:** 56%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 11.9 (s, 1H), 8.84 (d, *J* = 2.7 Hz, 1H), 8.70 (brs, 2H), 7.82 (dd, *J* = 8.7, 4.9 Hz, 1H), 7.01 (dd, *J* = 10.2, 8.7 Hz, 1H), 6.94 (s, 1H), 3.40 (s, 3H), 3.15 (s, 3H), 2.41 (d, *J* = 1.7 Hz, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 174.1, 161.0, 157.2 (d, *J* = 234.4 Hz), 151.4, 135.6 (d, *J* = 9.5 Hz), 129.8, 124.0 (d, *J* = 10.8 Hz), 116.5 (d, *J* = 10.3 Hz), 110.4, 108.8 (dd, *J* = 28.5, 4.0 Hz), 107.2 (d, *J* = 21.5 Hz), 28.0, 25.6, 8.9 (d, *J* = 4.0 Hz).
- **^{19}F NMR (377 MHz, DMSO-d$_6$)** δ −127.4 (s, 1F).
- **IR (neat)** ν_{max} = 3327, 2958, 1724, 1637, 1618, 1410, 1394, 1205, 1166, 1089, 975.
- **HRMS ESI**: *m/z* [M+H]$^+$: 287.1305; calculated for $C1_5H_{15}FN_4O^+$: 287.1303.

1-Methyl-(*E*)-aplysinopsin hydroiodide (1h)



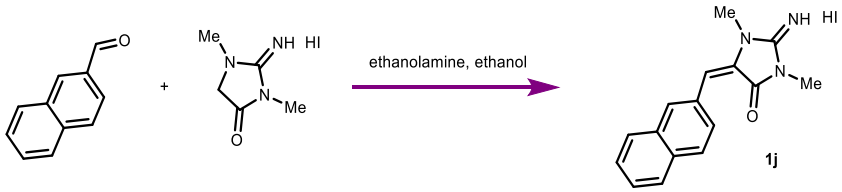
- **Yield:** 59%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 8.83 (brs, 1H), 8.73 (brs, 1H), 8.02 (d, *J* = 7.7 Hz, 1H), 7.53 (d, *d* = 7.2 Hz, 1H), 7.28 (t_{app}, *J* = 7.4 Hz, 1H), 7.22 (t_{app}, *J* = 7.6 Hz, 1H), 6.99 (brs, 1H), 3.90 (s, 3H), 3.41 (s, 3H), 3.15 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.6, 151.4, 136.3, 133.5, 128.1, 123.1, 122.5, 120.5, 118.4, 111.1, 110.5, 107.8, 33.3, 28.2, 25.7.
- **IR (neat)** ν_{max} = 3323, 3045, 1718, 1654, 1618, 1544, 1436, 1355, 1234, 1198, 1097, 1043.
- **HRMS ESI**: *m/z* $[M+H]^+$: 269.1398; calculated for $C_{15}H_{16}N_4O^+$: 269.1397.

2-*Aza*-(*E*)-aplysinopsin hydroiodide (1i)



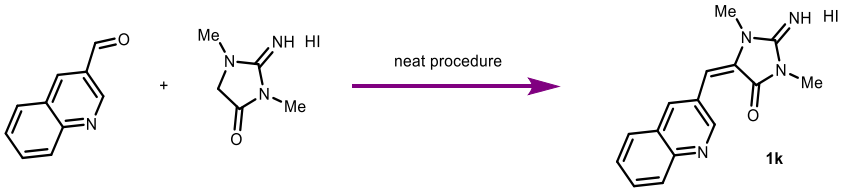
- **Yield:** 50% (obtained as 75/25 mixture of isomers).
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 7.84 (d, *J* = 8.2 Hz, 0.75H, major isomer), 7.34 (brs, 0.25H, minor isomer), 7.55 (d, *J* = 8.4 Hz, 1H, major + minor isomer), 7.40 (ddd, *J* = 8.3, 6.9, 1.1 Hz, 0.75H, major isomer), 7.32 (ddd, *J* = 8.4, 6.7, 1.1 Hz, 0.25H, minor isomer), 7.18 (ddd, *J* = 7.9, 6.8, 1.1 Hz, 0.75H, major isomer), 7.09 (brt, *J* = 7.7 Hz, 0.25H, minor isomer), 6.56 (s, 0.75H, major isomer), 6.4 (brs, 1H, minor isomer), 3.47 (s, 2H, major isomer), 3.27 (s, 1H, minor isomer), 3.09 (s, 2H, major isomer), 3.05 (s, 1H, minor isomer).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ (major isomer) 152.1, 150.2, 140.3, 137.9, 130.9, 126.5, 125.7, 123.2, 120.9, 119.6, 110.3, 30.6, 25.4.
- **IR (neat)** ν_{max} = 3227, 1732, 1664, 1625, 1438, 1307, 1122, 1104.
- **HRMS ESI**: *m/z* $[M+H]^+$: 256.1193; calculated for $C_{13}H_{13}N_5O^+$: 256.1193.

Compound 1j



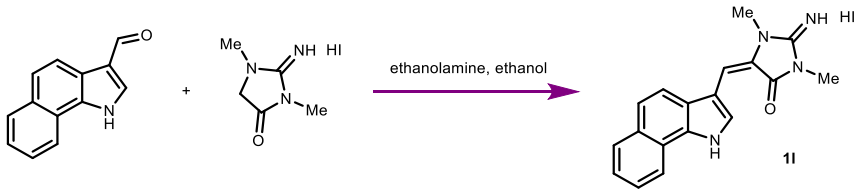
- **Yield:** 45% (neat procedure).
- **^{1}H NMR (300 MHz, DMSO-d$_6$)** δ 8.69 (brs, 1H), 8.44 (s, 1H), 8.12 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.96-7.79 (m, 3H), 7.59-7.43 (m, 2H), 6.74 (s, 1H), 3.32 (s, 3H), 3.11 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.7, 157.2, 151.9, 132.6, 132.5, 130.4, 129.9, 129.2, 128.1, 127.6, 127.4, 127.2, 126.7, 126.3, 116.8, 28.0, 25.6.
- **IR (neat)** ν_{max} = 3294, 2980, 2900, 1749, 1726, 1695, 1625, 1610, 1544, 1490, 1400, 1309, 1082, 1045.
- **HRMS ESI**: *m/z* [M+H]$^+$: 266.1290; calculated for $C_{16}H_{15}N_3O^+$: 266.1288.

Compound 1k



- **Yield:** 20% (neat procedure).
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 9.83 (brs, 1H), 9.27 (d, *J* = 2.2 Hz, 1H), 8.99 (d, *J* = 2.2 Hz, 1H), 8.03 (t_{app}, *J* = 9.5 Hz, 2H), 7.83 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.67 (ddd, 8.1, 6.9, 1.2 Hz, 1H), 7.27 (s, 1H), 3.46 (s, 3H), 3.19 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.2, 154.1, 151.7, 147.1, 136.9, 130.8, 128.9, 128.7, 128.6, 127.3, 126.7, 125.3, 118.8, 29.1, 26.4.
- **IR (neat)** ν_{max} = 3450, 2920, 1745, 1691, 1618, 1541, 1494, 1435, 1408, 1375, 1325, 1286, 1255, 1201, 1128, 1101, 1045, 1016, 1001, 954, 939, 867, 786, 759, 690, 644, 621.
- **HRMS ESI**: *m/z* [M+H]$^+$: 267.1239; calculated for $C_{15}H_{15}N_4O^+$: 267.1240.

Compound 1l

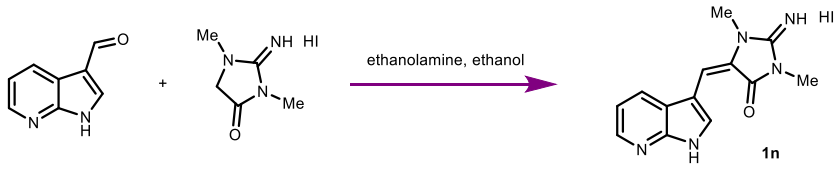


- **Yield:** 73%
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 12.6 (s, 1H), 8.86 (s, 1H), 8.37 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.63 (d, *J* = 8.7 Hz, 1H), 7.59 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.46 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 6.95 (s, 1H), 3.41 (s, 3H), 3.15 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 161.1, 151.3,130.3, 130.1, 128.4, 126.9, 125.7, 124.7, 124.1, 123.7, 121.8, 120.7, 120.5, 118.4, 110.4, 109.2, 27.9, 25.5.
- **IR (neat)** ν_{max} = 3332, 3282, 3265, 3155, 1689, 1624, 1541, 1494, 1463, 1456, 1427, 1388, 1247, 1209, 1149, 1130, 1111, 1091, 1060, 1049, 970, 958, 937, 883, 856, 796, 756, 719, 700, 686, 617.
- **HRMS ESI**: *m/z* [M+H]$^+$: 305.1385; calculated for $C_{18}H_{17}N_4O^+$: 305.1397.

Compound 1m

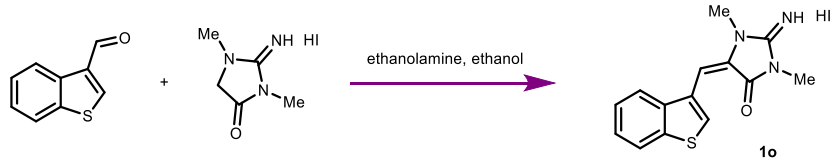
- **Yield:** 61%
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 8.86 (s, 1H), 7.94 (d, *J* = 1.7 Hz, 1H), 7.42 (dd, *J* = 8.5, 1.2 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 1H), 6.63 (s, 1H), 6.08 (s, 1H), 3.29 (s, 3H), 3.11 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.7, 152.0, 148.2, 147.1, 127.1, 126.8, 126.7, 118.9, 109.5, 108.1, 101.5, 28.1, 25.7.
- **IR (neat)** ν_{max} = 3566, 3338, 3072, 1741, 1716, 1662, 1624, 1595, 1558, 1489, 1448, 1431, 1363, 1263, 1217, 1141, 1093, 1035, 983, 935, 920, 891, 817, 796, 767, 756, 734, 615.
- **HRMS ESI**: *m/z* [M+H]$^+$: 260.1024; calculated for $C_{13}H_{14}N_3O_3^+$: 260.1030.

Compound 1n



- **Yield:** 75%
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 12.33 (s, 1H), 8.92 (d, *J* = 3.9 Hz, 1H), 8.87 (s, 1H), 8.44 (d, *J* = 8.2 Hz, 1H), 8.33-8.31 (m, 1H), 7.26-7.24 (m, 1H), 7.05-6.99 (m, 1H), 3.42 (d, *J* = 2.0 Hz, 3H), 3.17 (d, *J* = 1.7 Hz, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.8, 151.7, 148.2, 143.8, 129.8, 127.1, 124.4, 119.9, 116.5, 110.7, 107.3, 28.3, 25.8.
- **IR (neat)** ν_{max} = 3251, 3097, 3018, 1735, 1683, 1654, 1625, 1583, 1544, 1506, 1490, 1425, 1409, 1392, 1332, 1315, 1294, 1251, 1224, 1215, 1136, 1095, 1055, 1033, 979, 950, 898, 889, 813, 800, 758, 715, 692, 657, 648, 609.
- **HRMS ESI**: *m/z* [M+H]$^+$: 256.1200; calculated for $C_{13}H_{14}N_5O^+$: 256.1193.

Compound 1o

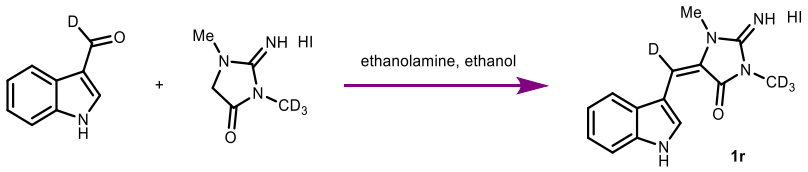


- **Yield:** 43%.
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 8.75 (s, 1H), 8.09 (d, *J* = 7.4 Hz, 1H), 8.02 (d, *J* = 7.3 Hz, 1H), 7.47 (ddd, *J* = 8.1, 7.1, 1.4 Hz, 1H), 7.44-7.39 (m, 1H), 7.03 (brs, 1H), 3.29 (s, 3H), 3.06 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.3, 150.6, 139.2, 139.0, 132.1, 128.2, 126.6, 125.0, 124.8, 123.4, 122.3, 100.9, 27.5, 25.5.
- **IR (neat)** ν_{max} = 3323, 3118, 2933, 1710, 1658, 1618, 1498, 1456, 1433, 1421, 1392, 1319, 1307, 1271, 1249, 1192, 1155, 1103, 1056, 1037, 972, 931, 871, 813, 786, 769, 752, 742, 727, 705, 696, 682, 609.
- **HRMS ESI**: *m/z* [M+H]$^+$: 272.0851; calculated for $C_{14}H_{14}N_3OS^+$: 272.0852.

Compound 1p

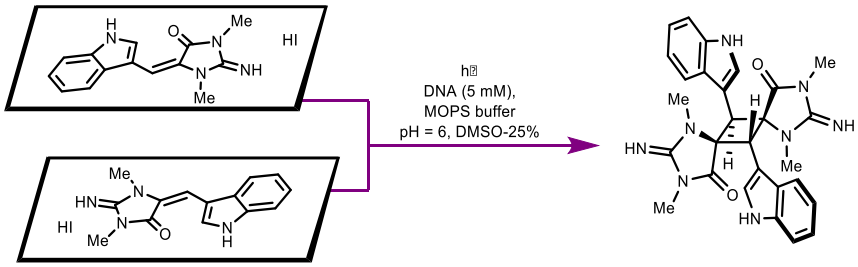
- **Yield:** 35% (neat procedure).
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 9.76 (brs, 1H), 8.98 (d, *J* =2.3 Hz, 1H), 8.57 (dd, *J* = 4.8, 1.7 Hz, 1H), 8.43 (dt, *J* = 8.2, 2.1 Hz, 1H), 7.49 (ddd, *J* = 8.0, 4.8, 0.9 Hz, 1H), 7.09 (s, 1H), 3.42 (s, 3H), 3.16 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 160.1, 154.1, 151.0, 149.9, 137.0, 128.9, 127.9, 123.3, 118.7, 29.0, 26.3.
- **IR (neat)** ν_{max} = 3084, 2875, 1772, 1745, 1691, 1587, 1541, 1494, 1433, 1371, 1359, 1319, 1292, 1251, 1192, 1151, 1101, 1043, 1031, 956, 893, 844, 813, 754, 702, 669.
- **HRMS ESI**: *m/z* [M+H]$^+$: 217.1086; calculated for $C_{11}H_{13}N_4O^+$: 217.1084.

7(8-^{2}H,15-^{2}H$_3$)-(*E*)-aplysinopsin hydroiodide (1r)



- **Yield:** 27%
- **^{1}H NMR (400 MHz, DMSO-d$_6$)** δ 11,5 (brs, 1H), 8.71 (d, *J* = 2.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.42 (dt$_{app}$, *J* = 7.8, 1.0 Hz, 1H), 7.16 (dt, 7.8, 1.2 Hz, 1H), 7.11 (dt, *J* = 8.0, 1.3 Hz, 1H), 6.71 (brs, 1H), 3.26 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 162.2, 150,6, 135.5, 127.6 (2C), 127.3, 126.3, 121.7, 119.4, 118.0, 111.7, 108.7, 30.6, 26.9.
- **IR (neat)** ν_{max} = 3294, 1707, 1690, 1608, 1516, 1460, 1429, 1386, 12401228, 1112, 1078, 1056.
- **HRMS ESI**: *m/z* [M+H]$^+$: 259.1492; calculated for $C_{14}H_{10}D_4N_4O^+$: 259.1491.

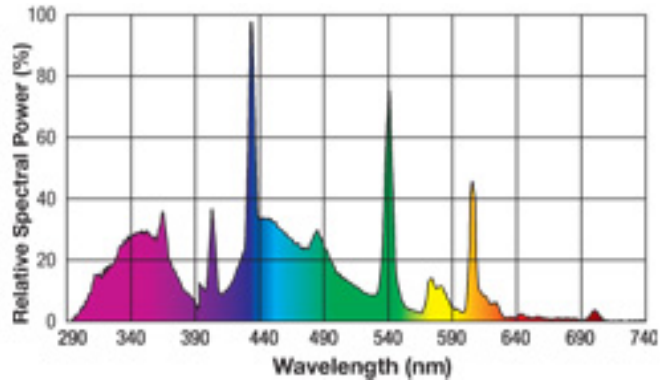
2.5. General procedure for the synthesis of the pseudo-dictazoles



Aplysinopsin-type hydroiodide monomer (x, 0.075 mMol) is solubilized in DMSO (12.5 mL). Organic solution is then added to MOPS buffer (pH = 6.5, 12.5 mL) affording solution #1.To a solution of st-DNA (97.5 mg, 150 mM) in MOPS buffer (pH = 6.5, 25.0 mL) in a crystalizing dish (diameter = 6 cm), solution #1 (25.0 mL) is then slowly added. Reaction mixture is stirred for 36 h under light irradiation (ReptiSun® 10.0 ZOOMED® UV-B, (10%) 26W lamps, distance = 5 cm). Crude mixture is concentrated to dryness under reduced pressure (2.10^{-2} mbar, 50°C for DMSO removal) and isopropanol (50 mL) is added. After 15 mn of stirring in an ultrasonic cleaner bath, DNA precipitates and suspension is centrifuged (3500 rpm, 6 min). The solution is collected and concentrated to dryness. The crude product is purified by preparative HPLC affording the titled product **4**.

Lamp characteristics

All experiments were conducted using the same UV-B enriched lamp [ReptiSun® 10.0 ZOOMED® UV-B, (10%)] providing 10% UV-B and 30% UV-A with a power of 26W. A typical emission spectrum of the lamp used along with its spectral features is furnished (Figure S1).



Brand	Model	UV-B	UV-A	Power
ZOOMED®	Reptisun® 10.0	10%	30%	26 W

***Figure S1**. Emission spectra of the ReptiSun® 10.0 ZOOMED® lamp.*

3. Calibration curves for conversion yields

3.1. Calibration curve of formylindole

Solutions of formylindole in methanol (1.0 mL) using ethyl *para*-hydroxybenzoate (1.0 mg) as internal standard were prepared from stock solutions (10.0 mg/mL). Solutions were analysed by analytical HPLC [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nM]. Formylindole: R_T = 11.70 mn; ethyl *para*-hydroxybenzoate: R_T = 14.10 mn.

***Table S1**. Calibration table of formylindole.*

ethyl *para*-hydoxybenzoate (mg/mL)	formylindole (equiv.)	formylindole (mg/mL)	ethyl *p*-hydroxybenzoate (integration #1)	formylindole (integration #2)	integation ratio (area #2 / area #1]
1	0,1	0,114	42155	11218	0,266
1	0,1	0,114	42281	11157	0,264
1	0,1	0,114	42413	11248	0,265
1	0,5	0,572	45568	49640	1,089
1	0,5	0,572	45328	49578	1,094
1	0,5	0,572	45291	49304	1,089
1	1	1,145	44363	108304	2,441
1	1	1,145	44634	107259	2,403
1	1	1,145	44325	105366	2,377
1	2	2,290	44781	201371	4,497
1	2	2,290	43954	199186	4,532
1	2	2,290	44010	200722	4,561

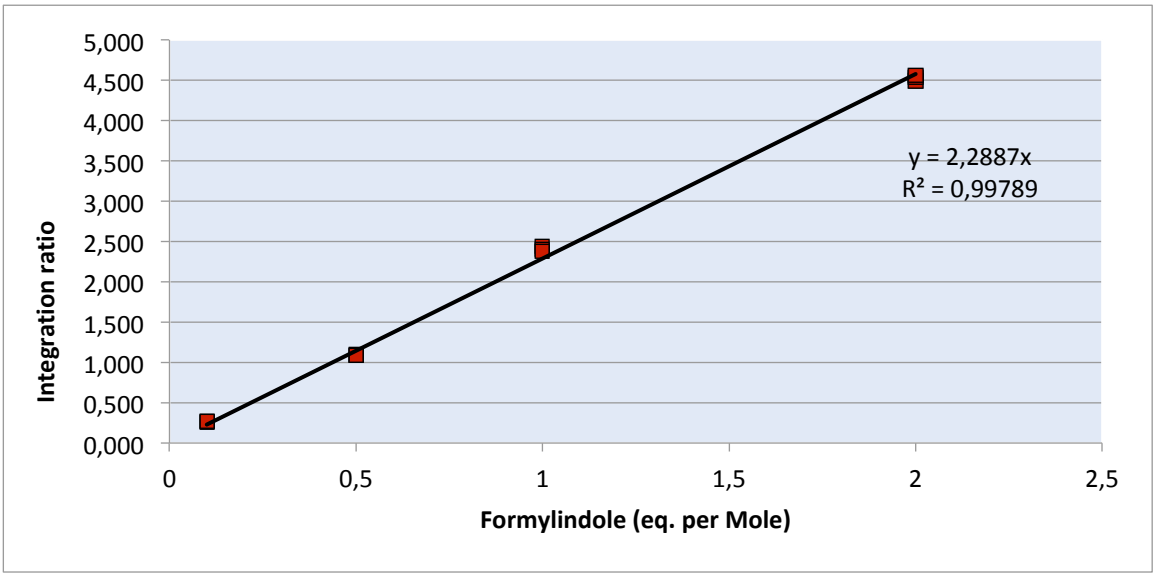


***Figure S2**. Calibration curve of formylindole*

3.2. Calibration curve of aplysinopsin (1a)

Solutions of (*E*)-aplysinopsin (**1a**) in methanol (1.0 mL) using ethyl *para*-hydroxybenzoate (1.0 mg) as internal standard were prepared from stock solutions (10.0 mg/mL). Solutions were analysed by analytical HPLC [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nM]. Aplysinopsin (**1a**): R_T = 8.50 mn; ethyl *para*-hydroxybenzoate: R_T = 14.10 mn.

***Table S2**. calibration table of aplysinopsin (**1a**)*

ethyl *para*-hydoxybenzoate (mg/mL)	aplysinopsin HI (equiv.)	aplysinopsin HI (mg/mL)	ethyl *p*-hydroxybenzoate (integration #1)	aplysinopsin HI (integration #2)	integation ratio (area #2 / area #1]
1	0,1	0,263	42956	17158	0,399
1	0,1	0,263	43037	17077	0,397
1	0,1	0,263	42478	17274	0,407
1	0,5	1,317	43720	78882	1,804
1	0,5	1,317	44095	79952	1,813
1	0,5	1,317	43849	79160	1,805
1	1	2,633	49890	170388	3,415
1	1	2,633	49729	171182	3,442
1	1	2,633	50503	172497	3,416
1	2	5,266	41988	308773	7,354
1	2	5,266	41868	308193	7,361
1	2	5,266	41816	307739	7,359

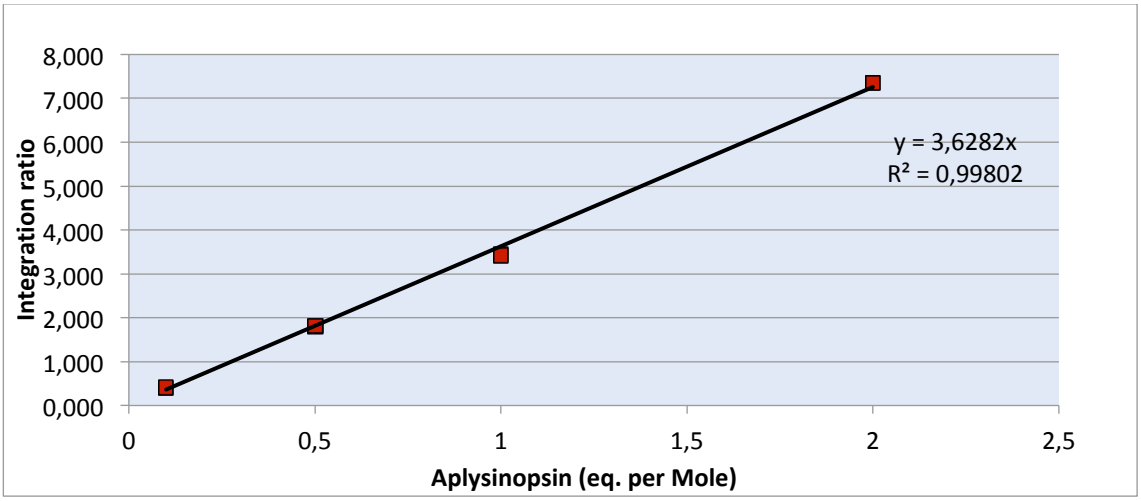


***Figure S3**. Calibration curve of aplysinopsin (**1a**)*

3.3. Calibration curve of pseudodictazole (4a)

Solutions of pseudodictazole (**4a**) in methanol (1.0 mL) using ethyl *para*-hydroxybenzoate (1.0 mg) as internal standard were prepared from stock solutions (20.0 mg/mL). Solutions were analysed by analytical HPLC [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nM]. Pseudodictazole (**4a**): R_T = 6.50 mn; ethyl *para*-hydroxybenzoate: R_T = 14.10 mn.

***Table S3**. Calibration table of pseudodictazole (**4a**)*

ethyl *para*-hydoxybenzoate (mg/mL)	pseudodictazole 2.HI (equiv.)	pseudodictazole 2.HI (mg/mL)	ethyl *p*-hydroxybenzoate (integration #1)	pseudodictazole 2.HI (integration #2)	integation ratio (area #2 / area #1]
1	0,1	0,527	46287	9333	0,202
1	0,1	0,527	45994	9282	0,202
1	0,1	0,527	46189	9360	0,203
1	0,25	1,317	46326	22592	0,488
1	0,25	1,317	46972	23193	0,494
1	0,25	1,317	46361	23102	0,498
1	0,5	2,633	46774	46206	0,988
1	0,5	2,633	46604	46987	1,008
1	0,5	2,633	46934	46955	1,000
1	1	5,266	46327	92571	1,998
1	1	5,266	46523	90773	1,951
1	1	5,266	45927	91809	1,999
1	2	10,533	45815	183148	3,998
1	2	10,533	46064	186855	4,056
1	2	10,533	46041	183447	3,984

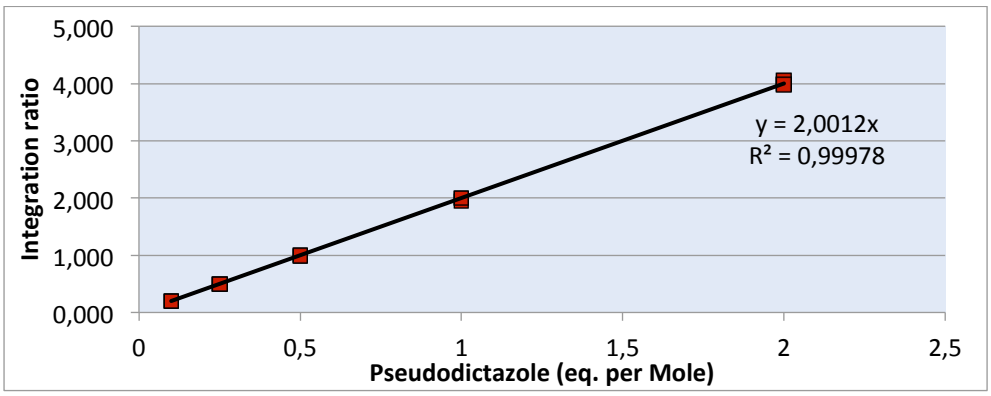
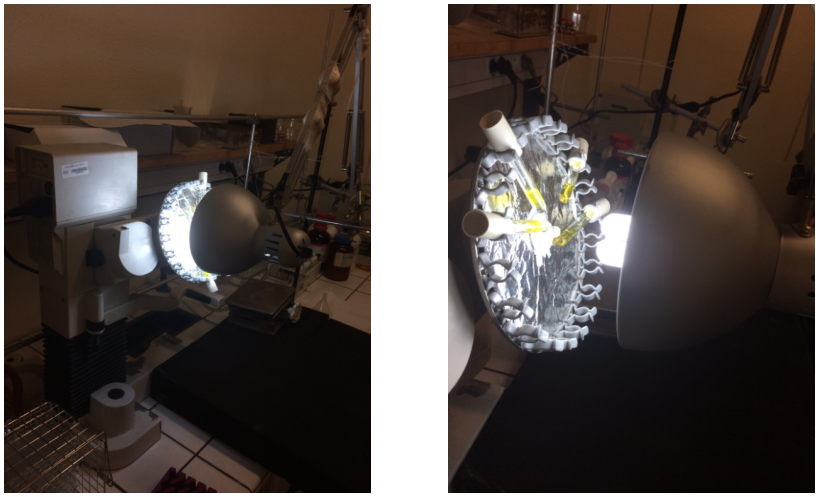


***Figure S4.** Calibration curve of pseudodictazole (**4a**)*

4. Optimization of the aplysinopsin dimerization

4.1. General procedures for dimerization of aplysinopsin (1a) with st-DNA (analytical scale)

(*E*)-aplysinopsin hydroiodide (**1a**, 11.5 mg, 0.03 mmol) and additive (see Table 4) are solubilized in DMF or DMSO (see relative percentage in Table 4). Organic solution is then added to MOPS buffer (pH = 6.5, final volume = 10.0 mL) affording solution #1. To a solution of st-DNA (3.9 mg/mL, 6 mM in base pairs) in MOPS buffer (pH = 6.5, 0.5 mL), a 2.5 mg/mL-solution of ethyl *para*-hydroxy benzoate (internal standard) in DMF (50 µL) is added affording solution #2. Solution #2 (0.5 mL) is transferred in a quartz tube and solution #1 (0.5 mL) is then slowly added. Reaction mixture is stirred by inversion (12 rpm, see device on picture 1) for 96 h under light irradiation (ReptiSun® 10.0 ZOOMED® UV-B, (10%) 26W lamps, distance = 8 cm). Crude mixture is concentrated to dryness under reduced pressure and isopropanol (2.00 mL) is added. After 5 mn of stirring in an ultrasonic cleaner bath, DNA precipitates and suspension is centrifuged (3500 rpm, 6 min). The solution is collected, concentrated to dryness and then analysed by analytical HPLC without any further purification (Figure 5, Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nM). Pseudodictazole (**4a**): R_T = 6.50 mn; aplysinopsin (**1a**): R_T = 8.50 mn; formylindole: R_T = 11.70 mn; ethyl *para*-hydroxybenzoate: R_T = 14.10 mn.



Picture S1. *Analytical scale device*

***Table S4**. Conditions screening for the dimerization of (E)-aplysinopsin (**1a**)*

Entry	st-DNA (mM BP)	C(1) (mM)	Additive (equiv.)	Co-solvent / V in stock solution #1	Co-solvent % total	Yield (%)[a]
1	3	1.5	-	-	-	0
2	3	1.5	-	DMF / 0.75 mL	3.75%	23
3	0	1.5	-	DMF / 0.75 mL	3.75%	0
4	3	1.5	-	DMF / 2.00 mL	10%	7
5	3	3.0	-	DMF / 0.75 mL	3.75%	17
6	3	4.5	-	DMF / 0.75 mL	3.75%	4
7	0	1.5	CuOTf (0.5)	DMF / 0.75 mL	3.75%	0
8	3	1.5	CuOTf (0.5)	DMF / 0.75 mL	3.75%	9
9	3	1.5	CuOTf (0.5)	DMF / 0.75 mL	3.75%	11
10	3	1.5	$Sc(OTf)_3$ (0.5)	DMF / 0.75 mL	3.75%	14
11	3	1.5	$Bi(OTf)_3$ (0.5)	DMF / 0.75 mL	3.75%	22
12	3	1.5	$Yb(OTf)_3$ (0.5)	DMF / 0.75 mL	3.75%	32
13	3	1.5	$Y(OTf)_3$ (0.5)	DMF / 0.75 mL	3.75%	49
14	3	1.5	$La(OTf)_3$ (0.5)	DMF / 0.75 mL	3.75%	56
15	3	1.5	-	DMSO / 0.75 mL	3.75%	28
16	3	1.5	-	DMSO / 2.00 mL	10%	39
16	3	1.5	-	DMSO / 5.00 mL	25%	56

[a] All reactions were performed in triplicate.

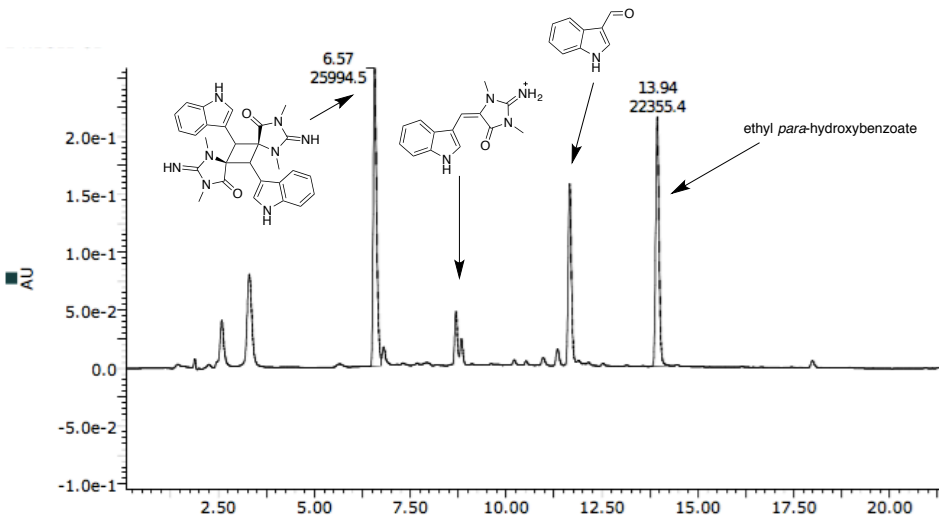
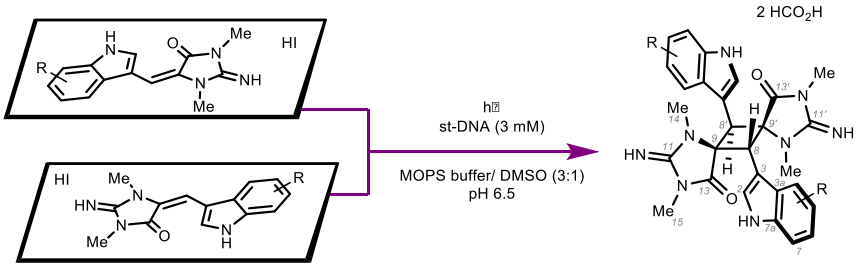
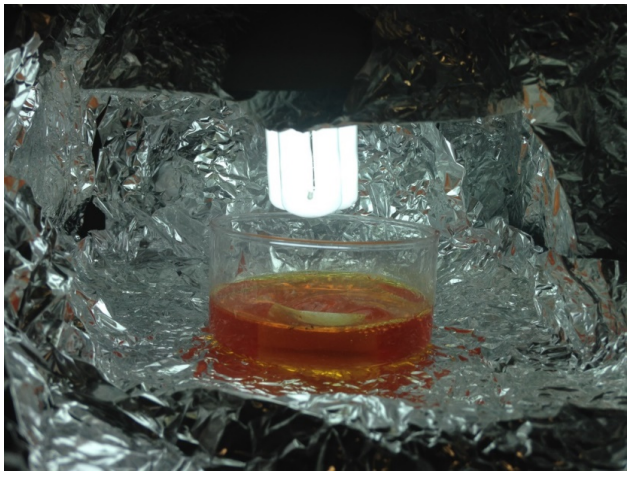


***Figure S5**. Diode array chromatogram of the homodimerization of (E)-aplysinopsin (**1a**) [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nm]*

4.2. Synthesis of pseudo-dictazole-type products (preparative scale)



General procedure: (*E*)-aplysinopsin-type substrate hydroiodide (0.15 mmol) and ethyl *p*-hydroxybenzoate [internal standard, 12.5 mg, 0.075 mmol) are solubilized in DMSO (25 mL). Organic solution is then added to MOPS buffer (pH = 6.5, 25 mL) affording solution #1. To a solution of st-DNA (3.9 mg/mL, 6 mM BP) in MOPS buffer (pH = 6.5, 50 mL) in a crystalizing dish (diameter = 14 cm), solution #1 (50 mL) is then slowly added. Reaction mixture is stirred for 48 h under light irradiation (ReptiSun® 10.0 ZOOMED® UV-B, (10%) 26W lamps, distance = 5 cm) (Picture S2). Crude mixture is concentrated to dryness under reduced pressure (2.10^{-2} mbar, 50 °C for DMSO removal) and isopropanol (100 mL) is added. After 15 mn of stirring in an ultrasonic cleaner bath, DNA precipitates and suspension is centrifuged (3500 rpm, 6 min). The solution is collected and concentrated to dryness. The crude product is purified by preparative HPLC (Sunfire® column) affording the titled products.



Picture S2. Large scale device

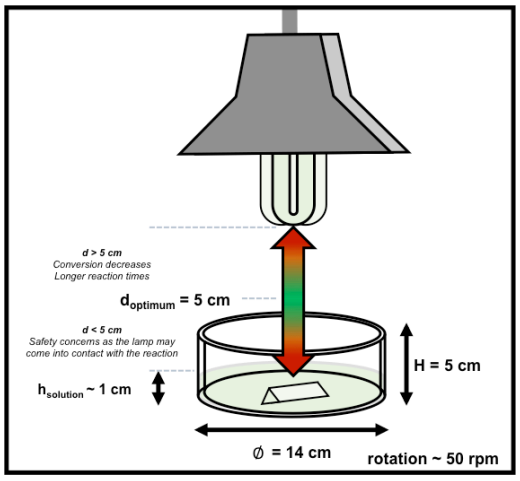


Figure S6. Large scale device

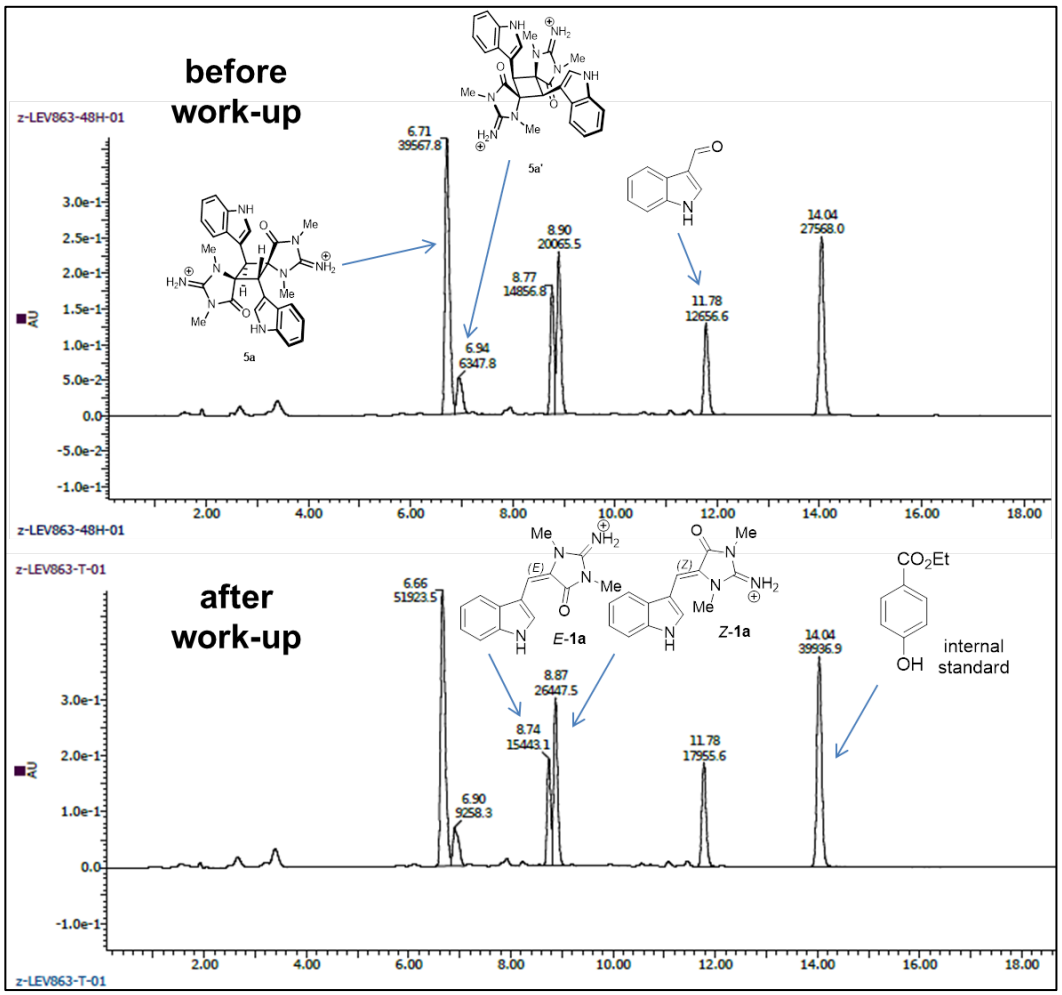
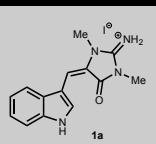
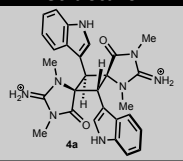
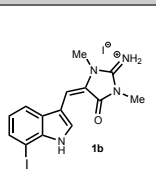
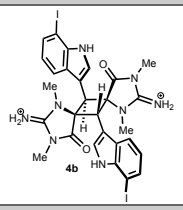
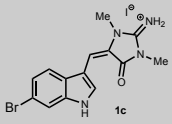
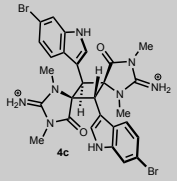
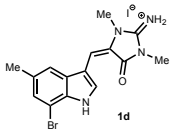
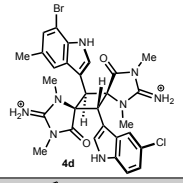
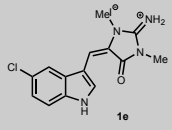
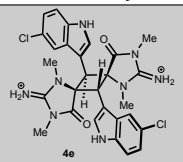
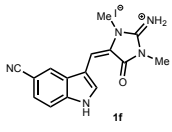
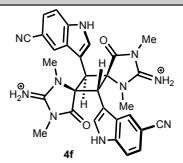
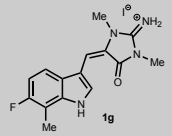
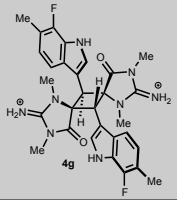
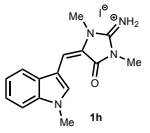
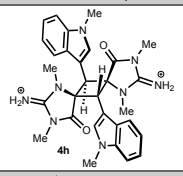
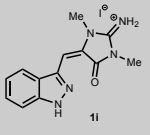
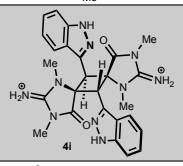
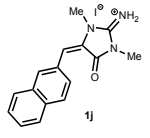
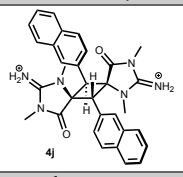
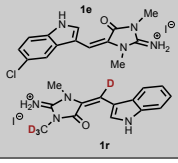
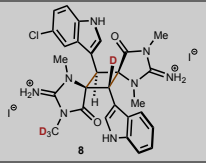
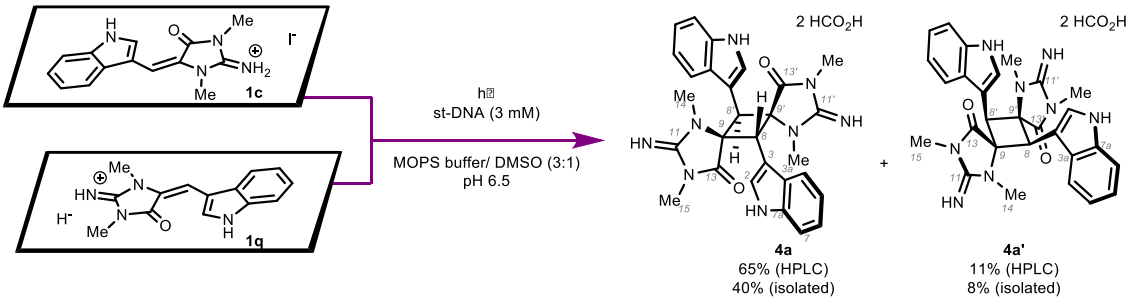


Figure S7. *Typical diode array chromatogram of the large scale homodimerization [ex: (E)-aplysinopsin (**1a**)] [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nm]*

Entry	Substrate	Dictazole-type structure	Conversion yield	Substrate not converted	"Formyl"-indole
1			**76%**	14%	10%
2			**68%**	24%	8%
3			**30%**	70%	-
4			**51%**	49%	-
5			**79%**	21%	-
6			**26%**	46%	28%
7			**57%**	43%	-
8			**83%**	17%	-
9			**78%**	22%	-
10			**88%**	12%	-
11			**46% [+34% 4e]: [+15% deutered-1a]**	[3% **1r**] [1% **1e**]	-

4.3. Description of dictazoles-type products

***Pseudo*-dictazole B (4a)**



Preparative HPLC purification: Sunfire® column (19 x 150 mm, 5 µm), 2% to 15% MeCN in H_2O (0.1% HCO_2H) affording the titled product **4a** (R_T = 12.50 min, 18.0 mg, 40%) and then **4a'** (R_T = 13.80 min, 2.2 mg, 8%).

Compound 4a:

- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 7.45 (dt, *J* = 8.2, 0.9 Hz, 2H), 7.40 (d, *J* = 1.0 Hz, 2H), 7.19 (ddd, *J* = 8.2, 7.1, 1.1 Hz, 2H), 7.08 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 2H), 6.94 (dt, *J* = 8.0, 1.0 Hz, 2H), 5.54 (d, *J* = 1.1 Hz, 2H), 3.39 (s, 6H), 3.35 (s, 6H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 174.8, 159.2, 137.5, 127.8, 125.0, 123.8, 121.2, 117.2, 113.3, 104.5, 70.1, 48.4, 30.4, 27.4.
- **IR (neat)** ν_{max} = 3142, 1707, 1659, 1589, 1460, 1388.
- **HRMS ESI**: *m/z* $[M+H]^+$: 509.2395; calculated for $C_{28}H_{29}N_8O_2^+$: 509.2411.

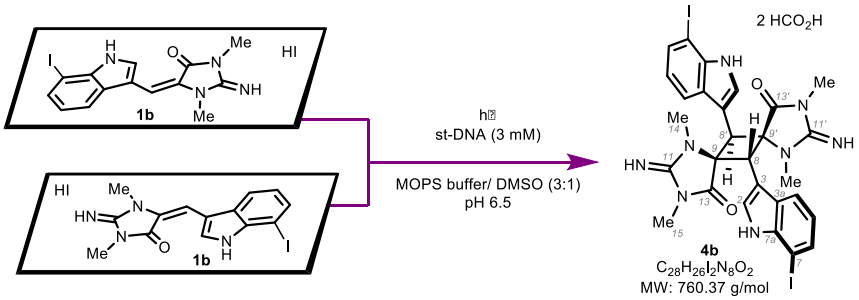
Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.40	2	d	*J* = 1.0 Hz	125.0
3/3'	-				104.5
3a/3a'	-				127.8
4/4'	6.94	2	dt	*J* = 8.0, 1.0 Hz	113.3
5/5'	7.08	2	ddd	*J* = 8.0, 7.0, 1.0 Hz	121.2
6/6'	7.19	2	ddd	*J* = 8.2, 7.1, 1.1 Hz	123.8
7/7'	7.45	2	dt	*J* = 8.2, 0.9 Hz	117.2
7a/7a'	-				137.5
8/8'	5.54	2	d	*J* = 1.0 Hz	48.4
9/9'	-				70.1
11/11'	-				159.2
13/13'	-				174.8
14/14'	3.39	6	s		30.4
15/15'	3.35	6	s		27.4

***syn*-Diastereomer 4a':**

- **^{1}H NMR (400 MHz, Methanol-d_4)** δ 7.38 (dt, *J* = 8.2, 0.9 Hz, 2H), 7.14 (dt, *J* = 7.5, 1.3 Hz, 2H), 7.13 (s, 2H), 7.03 (dt, *J* = 7.9, 1.0 Hz, 2H), 6.97 (dt, *J* = 8.1, 1.1 Hz, 2H), 5.21 (s, 2H), 3.61 (s, 3H), 3.31 (s, 3H), 3.20 (s, 3H), 3.09 (s, 3H).
- **^{13}C NMR (101 MHz, Methanol-d_4)** δ 176.5, 174.8, 159.4, 157.4, 137.6, 127.9, 124.5, 123.4, 120.8, 117.7, 113.0, 105.6, 71.4, 69.6, 47.7, 31.7, 27.2, 27.0, 26.2.
- **IR (neat)** ν_{max} = 3142, 1707, 1659, 1589, 1460, 1388.
- **HRMS ESI**: *m/z* $[M+H]^+$: 509.2398; calculated for $C_{28}H_{29}N_8O_2^+$: 509.2411.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.13	2	s		124.5
3/3'	-				105.6
3a/3a'	-				127.9
4/4'	6.97	2	dt	*J* = 8.1, 1.1 Hz	117.6
5/5'	7.03	2	dt	*J* = 7.9, 1.0 Hz	120.8
6/6'	7.14	2	dt	*J* = 7.5, 1.3 Hz	123.4
7/7'	7.38	2	dt	*J* = 8.2, 0.9 Hz	113.0
7a/7a'					137.6
8/8'	5.21	2	s		47.7
9	-				71.4
9'	-				69.6
11	-				157.4
11'					159.4
13	-				174.8
13'	-				176.5
14	3.61	3	s		27.2
14'	3.20	3	s		31.7
15	3.09	3	s		26.2
15'	3.31	3	s		27.0

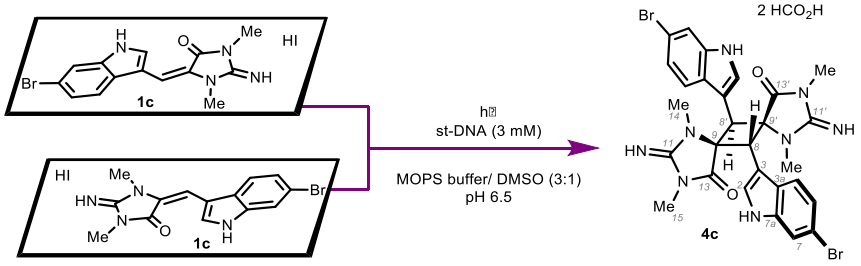
7,7'-Diiodo-*pseudo*-dictazole B (4b)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 60% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 7.10 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.50 (d, *J* = 7.4 Hz, 2H), 7.28 (d, *J* = 2.7 Hz, 2H), 6.90 (d, *J* = 7.9 Hz, 2H), 6.77 (t, *J* = 7.6 Hz, 2H), 5.21 (s, 2H), 3.23 (s, 6H), 2.99 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 173.6, 154.1, 137.2, 130.6, 127.2, 124.1, 121.0, 117.0, 107.2, 77.3, 67.0, 45.9, 28.4, 25.6.
- **IR (neat)** ν_{max} = 2962, 1736, 1648, 1490, 1425, 1130, 1087, 1056.
- **HRMS ESI**: *m/z* $[M+H]^+$: 761.0350; calculated for $C_{28}H_{27}N_8O_2I_2^+$: 761.041.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.28	2	d	*J* = 2.7 Hz	124.1
3/3'	-				107.2
3a/3a'	-				127.2
4/4'	6.90	2	d	*J* = 7.9 Hz	117.0
5/5'	6.77	2	t	*J* = 7.6 Hz	121.0
6/6'	7.50	2	d	*J* = 7.4 Hz	130.6
7/7'	-				77.3
7a/7a'	-				137.2
8/8'	5.21	2	s		45.9
9/9'	-				67.0
11/11'	-				154.1
13/13'	-				173.6
14/14'	3.23	6	s		28.4
15/15'	2.99	6	s		25.6

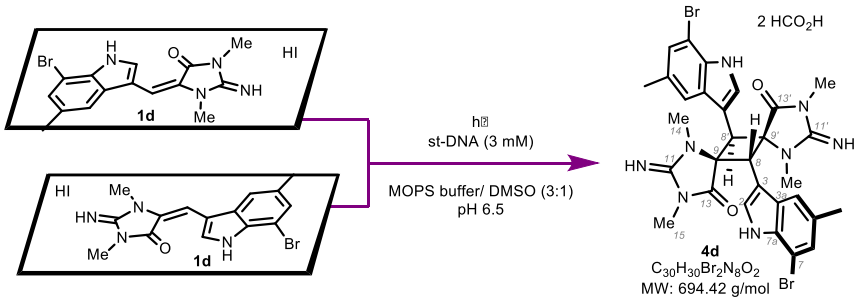
6,6'-Dibromo-*pseudo*-dictazole B (4c)



- **Preparative HPLC purification:** Xselect® column (2.1 x 150 mm, 3.5 µm), 1% to 40% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 9.00 min affording the titled product **2c** (8.1 mg, 14%).
- **^{1}H NMR (300 MHz, Methanol-d$_4$)** δ 7.58 (s, 2H), 7.36 (s, 2H), 7.15 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 5.43 (s, 2H), 3.30 (s, 6H), 3.28 (s, 6H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 174.9, 158.9, 138.3, 133.5, 127.0, 125.7, 124.1, 119.0, 116.9, 116.0, 105.7, 69.6, 48.1, 30.0, 27.0.
- **IR (neat)** ν_{max} = 3142, 1707, 1659, 1589, 1460, 1388.
- **HRMS ESI**: *m/z* $[M+H]^+$: 667.0575; calculated for $C_{28}H_{27}N_8O_2Br^{79}Br^{81}$: 667.0598.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.36	2	s		125.7
3/3'	-				105.7
3a/3a'	-				127.0
4/4'	6.84	2	d	*J* = 8.5 Hz	119.0
5/5'	7.15	2	d	*J* = 8.7 Hz	124.1
6/6'	-				116.9
7/7'	7.58	2	s		116.0
7a/7a'	-				138.3
8/8'	5.43	2	s		48.1
9/9'	-				69.6
11/11'	-				158.9
13/13'	-				174.9
14/14'	3.30	6	s		30.0
15/15'	3.28	6	s		27.0

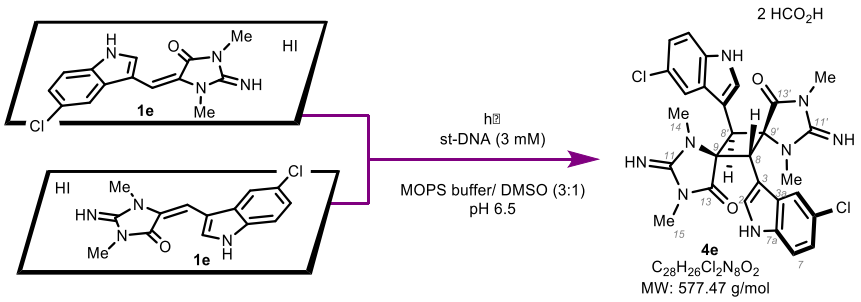
7,7'-Dibromo-5,5'-dimethyl-*pseudo*-dictazole B (4d)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 70% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 9.20 min.
- **^{1}H NMR (800 MHz, DMSO-d$_6$)** δ 7.18 (dd, *J* = 2.7, 1.2 Hz, 2H), 7.16 (d, *J* = 1.2 Hz, 2H), 6.62 (s, 2H), 5.14 (s, 2H), 3.18 (s, 6H), 2.97 (s, 6H), 2.29 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d$_6$)** δ 173.7, 153.9, 132.3, 129.5, 128.5, 125.4, 124.0, 116.4, 107.1, 104.0, 66.4, 46.0, 28.2, 25.4, 20.6.
- **IR (neat)** ν_{max} = 2917, 2852, 1742, 1653, 1485, 1448, 1425, 1390, 1189, 1189.
- **HRMS ESI**: *m/z* $[M+H]^+$: 695.0924; calculated for $C_{30}H_{31}N_8O_2{}^{79}Br^{81}Br^+$: 695.0911.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.18	2	dd	*J* = 2.7, 1.2 Hz	124.0
3/3'	-				107.1
3a/3a'					128.5
4/4'	6.62	2	s		116.4
5/5'	-				129.5
6/6'	7.16	2	dd	*J* = 1.2 Hz	125.4
7/7'	-				104.0
7a/7a'					132.3
8/8'	5.14	2	s		46.0
9/9'	-				66.4
11/11'	-				153.9
13/13'	-				173.7
14/14'	3.18	6	s		28.2
15/15'	2.97	6	s		25.4
5/5'-CH_3	2.29	6	s		20.6

5,5'-Dichloro-*pseudo*-dictazole B (4e)

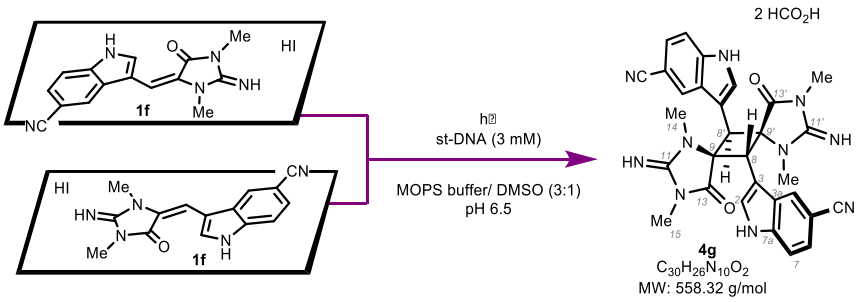


- **Preparative HPLC purification:** XSelect® column (19 x 150 mm, 5 µm), 5% to 50% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 9.20 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.39 (d, *J* = 8.6 Hz, 2H), 7.26 (d, *J* = 2.5 Hz, 2H), 7.08 (dd, *J* = 8.6, 2.0 Hz, 2H), 6.81 (d, *J* = 2.0 Hz, 2H), 5.15 (s, 2H), 3.13 (s, 6H), 3.03 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 173.8, 153.5, 134.0, 127.9, 124.3, 123.6, 121.5, 116.3, 113.2, 106.0, 66.3, 46.0, 28.2, 25.6.
- **IR (neat)** ν_{max} = 2980, 1738, 1660, 1424, 1191, 1076.
- **HRMS ESI**: *m/z* $[M+H]^+$: 577.1620; calculated for $C_{28}H_{27}N_8{}^{35}Cl^{37}ClO_3{}^+$: 577.1629

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.26	2	d	*J* = 2.6 Hz	124.3
3/3'	-				106.0
3a/3a'					127.9
4/4'	6.81	2	d	*J* = 2.0 Hz	116.3
5/5'	-				123.6
6/6'	7.08	2	dd	*J* = 8.6, 1.2 Hz	121.5
7/7'	7.39	2	s	*J* = 8.0 Hz	113.2
7a/7a'	-				134.0
8/8'	5.15	2	s		46.0
9/9'	-				66.3
11/11'	-				153.5
13/13'	-				173.8*
14/14'	3.13	6	s		28.2
15/15'	3.03	6	s		25.6

* Attributed by 2D NMR

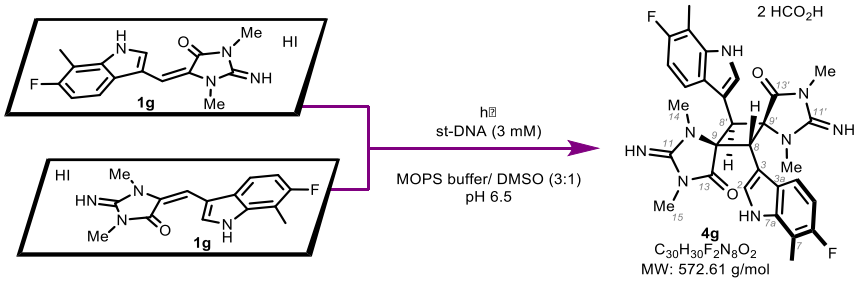
5,'-Dicyano-*pseudo*-dictazole B (4f)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 20% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 13.50 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.89 (d, *J* = 1.5 Hz, 2H), 7.52 (d, *J* = 2.2 Hz, 2H), 7.29 (dd, *J* = 8.3, 1.5 Hz, 2H), 7.00 (d, *J* = 8.3 Hz, 2H), 5.24 (s, 2H), 3.17 (s, 6H), 2.98 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 173.6, 153.7, 134.3, 129.8, 127.5, 121.7, 120.2, 118.0, 116.7, 107.0, 103.0, 66.7, 45.6, 28.2, 25.5.
- **IR (neat)** ν_{max} = 3047, 2913, 2218, 1745, 1658, 1476, 1404, 1206.
- **HRMS ESI**: *m/z* $[M+H]^+$: 559.2328; calculated for $C_{30}H_{27}N_{10}O_2^+$: 559.2313.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.52	2	d	*J* = 2.2 Hz	127.5
3/3'	-				107.0
3a/3a'					129.8
4/4'	7.89	2	d	*J* = 1.5 Hz	116.7
5/5'	-				103.0
6/6'	7.29	2	dd	*J* = 8.3, 1.5 Hz	121.7
7/7'	7.00	2	d	*J* = 8.3 Hz	118.0
7a/7a'					134.3
8/8'	5.24	2	s		45.6
9/9'	-				66.7
11/11'	-				153.7
13/13'	-				173.6
14/14'	3.17	6	s		28.2
15/15'	2.98	6	s		25.5
5/5'-CN	2.23	6	s		120.2

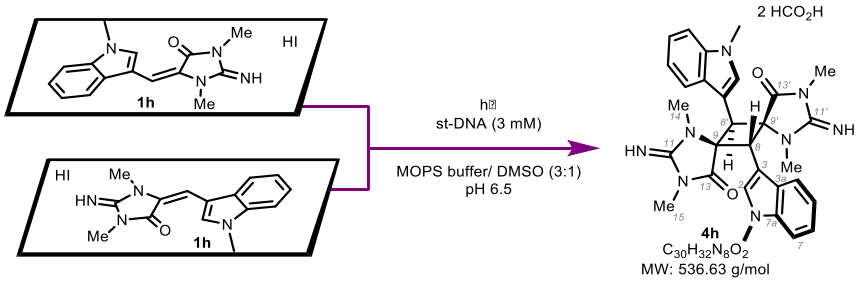
6,6'-Difluoro-7,7'-dimethyl-*pseudo*-dictazole B (4g)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 10% to 30% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 11.80 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.20 (s, 2H), 6.77 (t, *J* = 9.4 Hz, 2H), 6.67 (dd, *J* = 8.8, 4.7 Hz, 2H), 6.25 (s, 2H), 5.17 (s, 2H), 3.19 (s, 6H), 2.95 (s, 6H), 2.35 (d, *J* = 1.5 Hz, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 173.8, 156.8 (d, *J* = ~230 Hz), 154.1, 135.5, 123.2, 123.2, 114.8, 107.6, 106.6, 66.9, 45.8, 28.2, 25.4, 8.9 [attributed by 2D-NMR].
- **^{19}F NMR (753 MHz, DMSO-d_6)** δ –123.8 (s, 1F).
- **IR (neat)** ν_{max} = 2982, 1740, 1663, 1424, 1190, 1076.
- **HRMS ESI**: *m/z* $[M+H]^+$: 573.2546; calculated for $C_{30}H_{31}N_8O_2F_2^+$: 573.2533.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
1/1'	6.25	2	s		
2/2'	7.20	2	s		123.2
3/3'	-				107.7
3a/3a'					123.2
4/4'	6.67	2	dd	*J* = 8.8, 4.7 Hz	114.8
5/5'	6.77	2	t	*J* = 9.4 Hz	107.6
6/6'	-	-	-	*J* = ~230 Hz	156.8
7/7'	-				106.6
7a/7a'					135.5
8/8'	5.17	2	s		45.8
9/9'	-				66.9
11/11'	-				154.1
13/13'	-				173.8
14/14'	3.19	6	s		28.2
15/15'	2.95	6	s		25.4
7/7'-CH_3	2.23	6	s		8.9

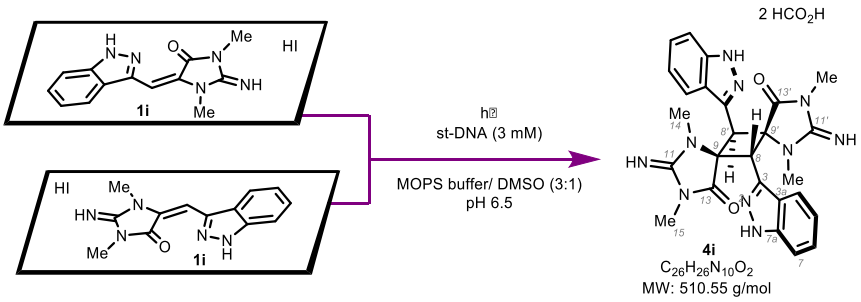
1,1'-Dimethyl-*pseudo*-dictazole B (4h)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 50% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 9.10 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.49 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 1.0 Hz, 2H), 7.22 (ddd, *J* = 8.1, 6.9, 1.0 Hz, 2H), 7.08 (ddd, *J* = 7.9, 7.0, 0.9 Hz, 2H), 6.88 (dt, *J* = 8.1, 0.9 Hz, 2H), 5.40 (d, *J* = 1.0 Hz, 2H), 3.82 (s, 6H), 3.38 (s, 6H), 3.27 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 172.8, 157.1, 136.0, 128.2, 126.6, 122.1, 119.8, 116.6, 110.4, 102.4, 68.2, 45.8, 32.7, 29.9, 27.1.
- **IR (neat)** ν_{max} = 2980, 1693, 1682, 1539, 1247, 1198, 1159.
- **HRMS ESI**: *m/z* $[M+H]^+$: 537.2732; calculated for $C_{30}H_{33}N_8O_2^+$: 537.2721.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2/2'	7.31	2	d	*J* = 1.0 Hz	128.2
3/3'	-				102.4
3a/3a'					126.6
4/4'	6.88	2	dt	*J* = 8.1, 0.9 Hz	116.6
5/5'	7.08	2	ddd	*J* = 7.9, 7.0, 0.9 Hz	119.8
6/6'	7.22	2	ddd	*J* = 8.1, 6.9, 1.0 Hz	122.1
7/7'	7.59	2	d	*J* = 8.3 Hz	110.4
7a/7a'					136.0
8/8'	5.40	2	d	*J* = 1.0 Hz	45.8
9/9'	-				68.2
11/11'	-				157.1
13/13'	-				172.8
14/14'	3.38	6	s		29.9
15/15'	3.27	6	s		27.1
1/1'-CH_3	3.82	6	s		32.7

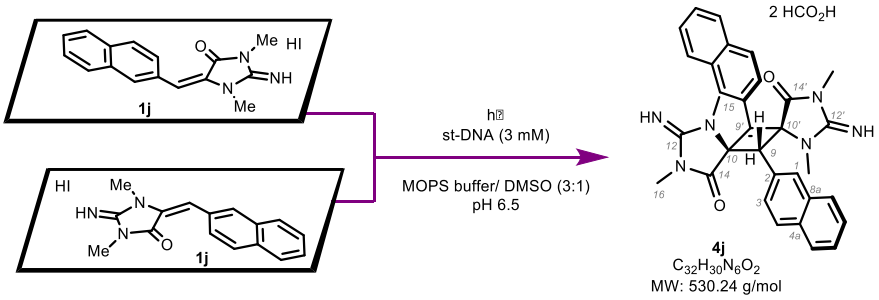
2,2'-Di-*aza*-*pseudo*-dictazole B (4i)



- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 60% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 6.00 min.
- **^{1}H NMR (800 MHz, DMSO-d$_6$)** δ 7.51 (d, *J* = 8.3 Hz, 2H), 7.35 (ddd, *J* = 8.3, 6.8, 1.1 Hz, 2H), 7.07-7.01 (m, 2H), 6.97 (d, *J* = 8.2 Hz, 2H), 5.44 (s, 2H), 3.12 (s, 6H), 2.97 (s, 6H)**.**
- **^{13}C NMR (200 MHz, DMSO-d$_6$)** δ 172.5, 159.8, 140.8, 136.6, 126.4, 120.5, 120.4, 118.1, 110.5, 64.7, 47.6, 26.1, 24.3.
- **IR (neat)** ν_{max} = 2980, 1773, 1743, 1703, 1496, 1459, 1431, 1200, 1088, 1024.
- **HRMS ESI**: *m/z* $[M+H]^+$: 511.2310; calculated for $C_{26}H_{27}N_{10}O_2^+$: 511.2313.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
3/3'	-				120.5
3a/3a'					136.6
4/4'	6.97	2	d	*J* = 8.2 Hz	116.6
5/5'	7.07-7.01	2	m		120.4
6/6'	7.35	2	ddd	*J* = 8.3, 6.8, 1.1 Hz	126.4
7/7'	7.51	2	d	*J* = 8.3 Hz	110.5
7a/7a'					140.8
8/8'	5.44	2	s		47.6
9/9'	-				64.7
11/11'	-				159.8
13/13'	-				172.5
14/14'	3.12	6	s		26.1
15/15'	2.97	6	s		24.3

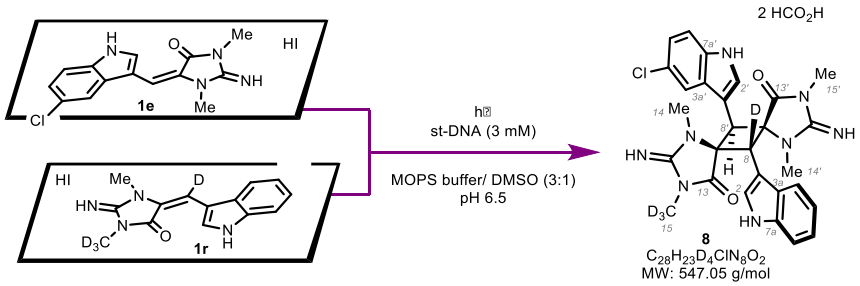
Compound (4j)



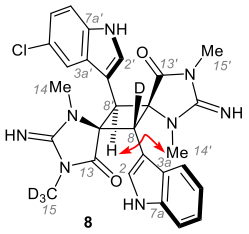
- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 50% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 10.10 min.
- **^{1}H NMR (800 MHz, DMSO-d_6)** δ 7.90-7.84 (m, 4H), 7.80 (d, *J* = 7.5 Hz, 2H), 7.55-7.50 (m, 4H), 7.34 (s, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 5.21 (s, 2H), 3.21 (s, 6H), 3.04 (s, 6H).
- **^{13}C NMR (200 MHz, DMSO-d_6)** δ 173.1, 153.5, 132.5, 131.8, 131.5, 128.0, 127.6, 127.4, 126.4, 126.0, 124.3, 124.0, 66.4, 51.1, 28.2, 25.6.
- **IR (neat)** ν_{max} = 3273, 1737, 1672, 1484, 1462, 1213, 1188,1078.
- **HRMS ESI**: *m/z* $[M+H]^+$: 531.2512; calculated for $C_{32}H_{31}N_6O_2^+$: 531.2503.

Assignment	^{1}H δ ppm	Integration	multiplicity	*J* (Hz)	^{13}C δ ppm
1/1'	7.34	2	s		124.3
2/2'	-				131.8
3/3'	6.87	2	d	*J* = 8.5 Hz	124.0
4/4'	7.87	2	m		128.0
4a/4a'	-				131.5
5/5'	7.88	2	m		127.4
6/6'	7.53	2	m		126.4
7/7'	5.53	2	m		126.0
8/8'	7.80	2	d	*J* = 7.5 Hz	127.6
8a/8a'	-				132.5
9/9'	5.21	2	s		51.1
10/10'	-				66.4
12/12'	-				153.5
14/14'	-				173.1
15/15'	3.21	6	s		28.2
16/16'	2.04	6	s		25.6

(8-^{2}H,15-^{2}H$_3$)-5'-Chloro-*pseudo*-dictazole B (8)



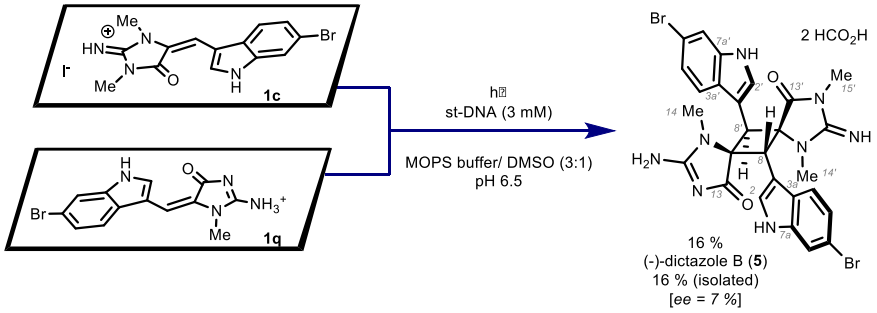
- **Preparative HPLC purification:** Sunfire® column (19 x 150 mm, 5 µm), 5% to 50% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min; R_T = 8.50 min.
- **^{1}H NMR (800 MHz, DMSO-d$_6$)** δ 7.39 (d, *J* = 8.5 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.27 (m, 1H), 7.24 (d, *J* = 2.6 Hz, 1H), 7.09-7.06 (m, 2H), 6.93 (brt, *J* = 7.4 Hz, 1H), 6.90 (d, *J* = 7.9 Hz, 1H), 6.81 (d, *J* = 2.0 Hz, 1H), 5.17 (s, 1H), 3.20 (s, 3H), 3.14 (s, 3H), 3.00 (s, 3H).
- **^{13}C NMR (200 MHz, DMSO-d$_6$)** δ 173.9 (2C), 153.9, 153.8,135.4, 134.0, 127.9, 126.9, 124.6, 123.5, 123.0, 121.5, 121.4, 119.0, 116.8, 116.3, 113.2, 111.7, 106.1, 105.7, 66.7, 66.6, 45.8, 28.1, 28.0, 25.4.
- **IR (neat)** ν_{max} = 2980, 1739, 1664, 1456, 1421, 1375, 1211, 1083.
- **HRMS ESI**: *m/z* [M+H]$^{+}$: 547.2294; calculated for $C_{28}{}^{1}H_{24}{}^{2}H_4N_8{}^{35}ClO_2{}^{+}$: 547.2269.



8

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2	7.24	1	d	2.6 Hz	123.0
3	-				105.7
3a	-				126.9
4	6.90	1	d	7.9 Hz	116.8
5	6.93	1	br t	7.4 Hz	119.0
6	7.07	1	m		121.5
7	7.37	1	d	8.1 Hz	111.7
7a	-				135.4
8	x				x
9	-				66.7
11	-				153.8
13	-				179.9
14	3.14	3			28.0
15	x				x
2'	7.27	1	m		124.6
3'	-				106.1
3a'	-				127.9
4'	6.81	1	d	2.0 Hz	116.3
5'	-				123.5
6'	7.08	1	m		121.4
7'	7.39	1	d	8.5 Hz	113.2
7a'	-				134.0
8'	5.17	1	s		45.8
9'	-				66.6
11'	-				153.9
13'	-				179.9
14'	3.20	3	s		28.1
15'	3.00	3	s		25.4

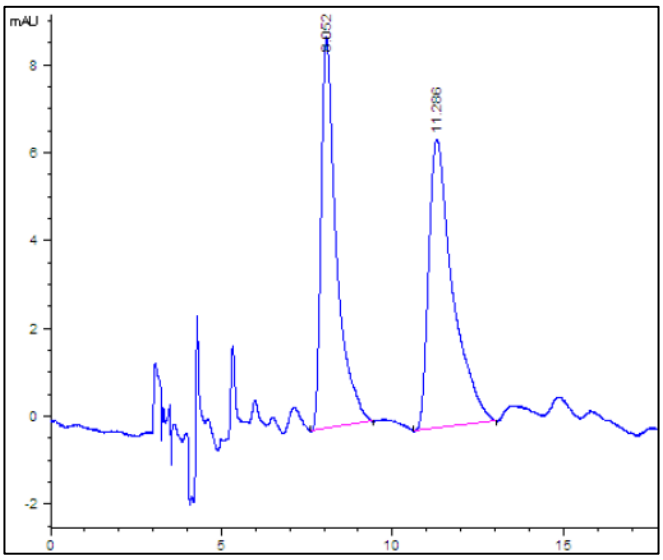
4.4. Synthesis of dictazole B (5)



6-bromo-(*E*)-aplysinopsin hydroiodide (**1c**, 34.5 mg, 0.075 mmol) and bromo-desmethyl-aplysinopsin (**1q**, 24.9 mg, 0.075 mmol) are solubilized in DMSO (25.0 mL). Organic solution is then added to MOPS buffer (pH = 6.5, 25.0 mL) affording solution #1. To a solution of st-DNA (3.9 mg/mL, 6 mM BP) in MOPS buffer (pH = 6.5, 50.0 mL) in a crystalizing dish (diameter = 14 cm), solution #1 (50.0 mL) is then slowly added. Reaction mixture is stirred for 36 h under light irradiation (ReptiSun® 10.0 ZOOMED® UV-B, (10%) 26W lamps, distance = 5 cm) (Picture S2). Crude mixture is concentrated to dryness under reduced pressure (2.10^{-2} mbar, 50°C for DMSO removal) and isopropanol (100 mL) is added. After 15 mn of stirring in an ultrasonic cleaner bath, DNA precipitates and suspension is centrifuged (3500 rpm, 6 min). The solution is collected and concentrated to dryness. The crude product is purified by preparative HPLC (XSelect® column, 15% to 30% MeCN in H_2O (0.1% HCO_2H) over 15 min at 17 mL/min) affording product **2c** (R_T = 5.90 min, 2.7 mg, 5%), the titled product **10** (R_T = 9.50 min, 9.0 mg, 16%). The enantiomeric excess was determined by HPLC (Chiralcel, IA, hexane/ethanol = 80: 20, 1.0 mL/min, 275 nm, R_T (minor) = 8.1 min, R_T (major) = 11.3 min); 7% ee.

- **^{1}H NMR (400 MHz, DMSO-d_6)** δ 11.18 (d, *J* = 2.8 Hz, 1H), 11.17 (d, *J* = 2.6 Hz, 1H), 7.56 (s, 1H), 7.55 (s, 1H), 7.44 (dd, *J* = 2.6, 1.1 Hz, 1H), 7.16 (dd, *J* = 2.6, 1.2 Hz, 1H), 7.06 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.03 (dd, *J* = 8.5, 1.8 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 1H), 6.80 (d, *J* = 8.5 Hz, 1H), 5.10 (s, 1H), 5.02 (s, 1H), 3.09 (s, 3H), 3.05 (s, 3H), 3.01 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d_6)** δ 188.3, 174.3, 170.1, 158.2, 136.5, 136.3, 126.3, 126.0, 124.6, 123.5, 121.8, 121.6, 119.2, 118.7, 114.3, 114.2 (2C), 114.0, 107.2, 107.0, 68.6 (2C), 46.0, 44.7, 28.5, 27.8, 25.5.
- **IR (neat)** ν_{max} = 3010, 1653, 1587, 1458, 1397.
- **HRMS ESI**: *m/z* $[M+H]^+$: 651.0464; calculated for $C_{27}H_{25}N_8O_2Br^{79}{}_2$: 651.0467.

Assignment	^{1}H δ ppm	integration	Multiplicity	*J* (Hz)	^{13}C δ ppm
1	11.18	1	d	*J* = 2.8 Hz	-
2	7.44	1	dd	*J* = 2.6, 1.1 Hz	124.6
3	-				107.0
3a	-				126.3
4	6.93	1	d	*J* = 8.5 Hz	118.7
5	7.06	1	dd	*J* = 8.5, 1.8 Hz	121.8
6	-				114.2
7	7.56	1	s		114.3
7a	-				136.3
8	5.03	1	s		44.7
9	-				58.6
11	-				170.1
13	-				188.3
14	3.01	3	s		27.8
1'	11.17	1	d	*J* = 2.6 Hz	-
2'	7.16	1	dd	*J* = 2.6, 1.2 Hz	123.5
3'	-				107.2
3a'	-				126.0
4'	6.93	1	d	*J* = 8.5 Hz	119.2
5'	7.06	1	dd	*J* = 8.5, 1.8 Hz	121.6
6'	-				114.2
7'	7.55	1	s		114.0
7a'	-				136.5
8'	5.06	1	s		46.0
9'	-				68.6
11'	-				158.2
13'	-				174.3
14'	3.09	3	s		28.5
15'	3.05	3	s		25.5



#	Time	Area	Height	Width	Area%	Symmetry
1	8.052	281.3	8.9	0.4515	46.556	0.479
2	11.286	323	6.6	0.6845	53.444	0.485

***Figure S8**. Chiral diode array chromatogram of Dictazole B (**5**) (Chiralcel, IA, hexane/ethanol = 80: 20, 1.0 mL/min, 275 nm)*

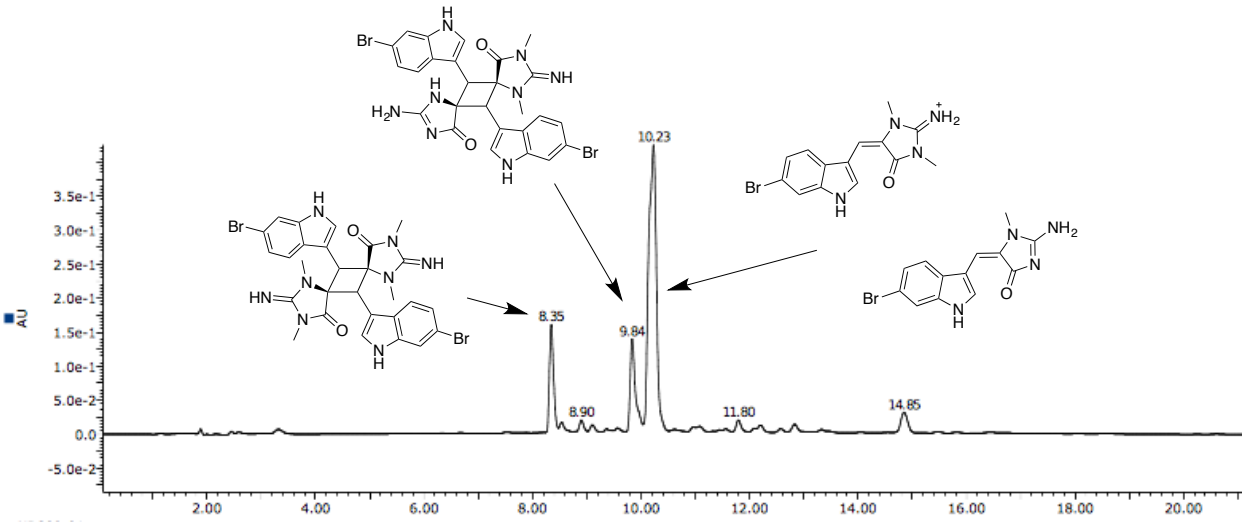
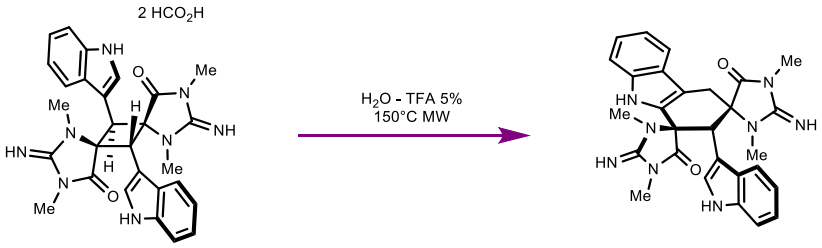
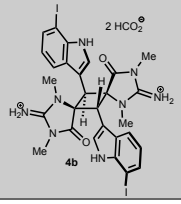
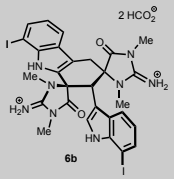
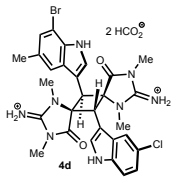
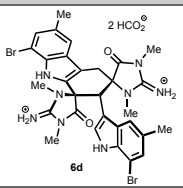
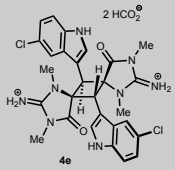
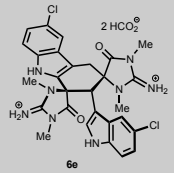
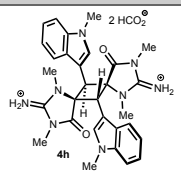
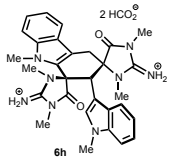


***Figure S9.** Diode array chromatogram of the large synthesis of Dictazole B (**5**) [Sunfire® C_{18} columns 5% to 75% MeCN in H_2O (0.1% HCO_2H) over 15 mn at 1 mL/mn, HRMS and UV detection at 280 nm]*

4.5. Synthesis of tubastrindole-type products



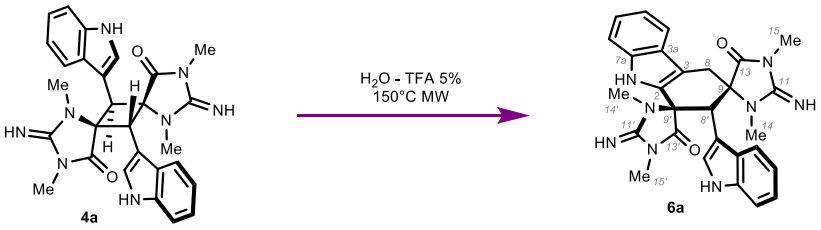
General *procedure.* A solution of dictazole-type product 4 (3 to 10 mg) in a 5%-aqueous solution of TFA (25 mL) is heated to 150 °C for 90 sec under microwave irradiation (max ~30W). After cooling the reaction mixture is concentrated under reduced pressure. The crude product is purified by preparative HPLC affording the titled product 6. [*Note: since our original report, our laboratory was equipped with a new microwave reactor and the reaction temperature needed to be re-optimized to 150 °C].*[3]

Entry	Substrate	Quantity of substrate	Tubastrindole-type product	Quantity of isolated product	Yield
1		10 mg		5.0 mg	50%
2		4 mg		1.6 mg	40%
3		3 mg		0.9 mg	30%
4		6.5 mg		1.8 mg	28%

[3] A. Skiredj, M. A. Beniddir, D. Joseph, K. Leblanc, G. Bernadat, L. Evanno and E. Poupon, *Org. Lett.*, 2014, **16**, 4980.

4.6. Description of tubastrindoles-type products

***rac*-Tubastrindole B (6a)**

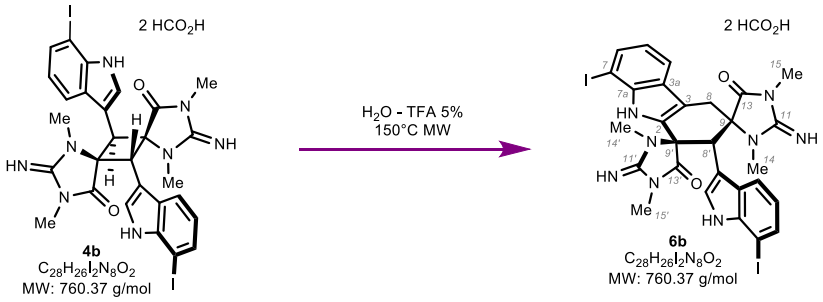


Tubastrindole B (**6a**) was prepared according to the previously reported procedure.[3]

- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 8.48 (s, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.41 (t$_{app}$, *J* = 7.3 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.18 (t, *J* = 7.2 Hz, 2H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.07 (s, 1H), 4.55 (s, 1H), 3.79 (d, *J* = 17.4 Hz, 1H), 3.60 (d, *J* = 17.4 Hz, 1H), 3.31 (s, 3H), 3.07 (s, 3H), 2.89 (s, 3H), 2.66 (s, 3H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 174.6, 172.9, 161.2, 158.3, 139.5, 136.7, 128.9, 126.3, 125.5, 124.9, 124.8, 123.6, 121.2 (2C), 120.0, 118.2, 114.5, 112.9, 112.7, 104.5, 72.2, 71.8, 44.8, 32.8, 27.8, 27.7, 26.4, 26.2.
- **IR (neat)** ν_{max} = 3318, 1769, 1690, 1589, 1460, 1388.
- **HRMS ESI**: *m/z* [M+H]$^{+}$: 509.2415; calculated for $C_{28}H_{29}N_8O_2$: 509.2413.

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2	-				124.9
3	-				114.5
3a	-				126.3
4	7.65	1	d	*J* = 8.0 Hz	120.0
5	7.18				121.2
6	7.21	1	t	*J* = 7.6 Hz	125.5
7	7.41	1	d	*J* = 7.3 Hz	112.9
7a	-				139.5
8α	3.60	1	d	*J* = 17.4 Hz	27.7
8β	3.79	1	d	*J* = 17.4 Hz	
9	-				72.2
11	-				161.2
13	-				174.6
14	3.31	3	s		32.8
15	2.66	3	s		26.4
2'	7.07	1	s		124.8
3'	-				104.5
3a'	-				128.9
4'	7.54	1	d	*J* = 8.0 Hz	118.2
5'	7.13	1	t	*J* = 7.5 Hz	121.2
6'	7.18	1	t	*J* = 7.3 Hz	123.6
7'	7.41	1	d	*J* = 7.3 Hz	112.7
7a'	-				136.7
8'	4.55	1	s		44.8
9'	-				71.8
11'	-				158.3
13'	-				172.9
14'	3.07	3	s		27.8
15'	2.89	3	s		26.2

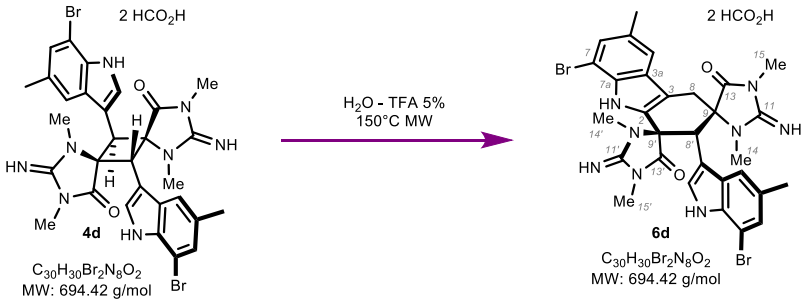
***rac*-7,7'-Diodo-tubastrindole (6b)**



- **Preparative HPLC purification:** Sunfire® column (10 x 150 mm, 5 µm), 5% to 50% MeCN in H_2O (0.1% HCO_2H) over 15 min at 1.0 mL/min; R_T = 10.5 min.
- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 7.70 (d, *J* = 7.5 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.13 (s, 1H), 6.97 (t, *J* = 7.8 Hz, 1H), 6.92 (t, *J* = 7.8 Hz, 1H), 4.44 (s, 1H), 3.74 (d, *J* = 17.3 Hz, 1H), 3.51 (d, *J* = 17.3 Hz, 1H), 3.23 (s, 3H), 3.04 (s, 3H), 2.83 (s, 3H), 2.66 (s, 3H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 174.7, 172.8, 160.8, 158.2, 141.6, 138.5, 135.0, 132.8, 129.3, 127.1, 126.0, 125.8, 122.9, 122.8, 120.1, 118.7, 116.4, 106.5, 77.0, 76.7, 71.5, 71.3, 45.5, 32.4, 28.0, 27.7, 26.3, 26.0.
- **IR (neat)** ν_{max} = 3387, 1654, 1550, 1489, 1458, 1199, 1075.
- **HRMS ESI**: *m/z* $[M+H]^+$: 761.0350; calculated for $C_{28}H_{27}N_8O_2I_2^+$: 761.0341.

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2	-				126.0
3	-				116.4
3a	-				127.1
4	7.66	1	d	*J* = 7.9 Hz	120.1
5	6.97	1	t	*J* = 7.8 Hz	122.82
6	7.70	1	d	*J* = 7.5 Hz	135.0
7	-	1	d		76.7
7a	-				141.6
8α	3.51	1	d	*J* = 17.3 Hz	28.0
8β	3.74	1	d	*J* = 17.3 Hz	
9	-				71.3
11	-				160.8
13	-				174.7
14	3.23	3	s		32.4
15	2.66	3	s		26.3
2'	7.13	1	s		125.8
3'	-				106.5
3a'	-				129.3
4'	7.55	1	d	*J* = 8.0 Hz	118.7
5'	6.92	1	br t	*J* = 7.8 Hz	122.87
6'	7.56	1	d	*J* = 7.4 Hz	132.8
7'	-				77.0
7a'	-				138.5
8'	4.44	1	s		45.5
9'	-				71.5
11'	-				158.2
13'	-				172.8
14'	3.04	3	s		27.7
15'	2.83	3	s		26.0

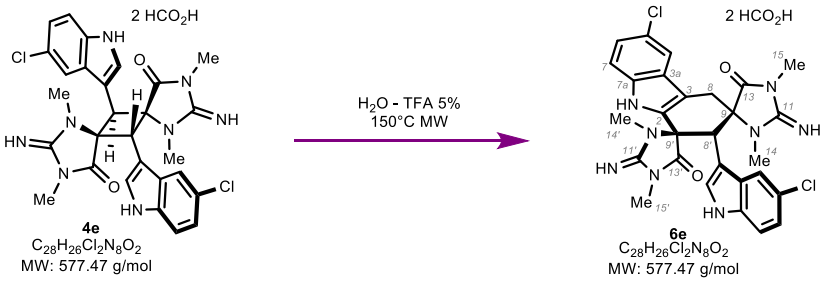
***rac*-7,7'-Dibromo-5,5'dimethyl-tubastrindole (6d)**



- **Preparative HPLC purification:** Sunfire® column (10 x 150 mm, 5 µm), 5% to 80% MeCN in H_2O (0.1% HCO_2H) over 15 min at 1.0 mL/min; R_T = 6.8 min.
- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 7.42 (t, *J* = 1.2 Hz, 1H), 7.33 (d, *J* = 1.2 Hz, 1H), 7.29 (t, *J* = 1.2 Hz, 1H), 7.19 (d, *J* = 1.2 Hz, 1H), 7.08 (s, 1H), 4.41 (s, 1H), 3.70 (d, *J* = 17.2 Hz, 1H), 3.45 (d, *J* = 17.2 Hz, 1H), 3.22 (s, 3H), 3.02 (s, 3H), 2.82 (s, 3H), 2.66 (s, 3H), 2.46 (s, 3H), 2.43 (s, 3H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 174.9, 172.9, 160.8, 158.1, 136.5, 133.6, 132.4, 132.3, 130.7, 129.4, 128.3, 127.5, 126.8, 126.1, 119.0, 117.5, 115.4, 105.9, 105.5 (2C), 71.5, 71.3, 45.3, 32.3, 27.9, 27.6, 26.3, 25.9, 21.4, 21.2.
- **IR (neat)** ν_{max} = 3240, 1743, 1651, 1558, 1483, 1427, 1207, 1045.
- **HRMS ESI**: *m/z* $[M+H]^+$: 695.0916; calculated for $C_{30}H_{31}N_8O_2{}^{79}Br^{81}Br^+$: 695.0911.

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2	-				126.8
3	-				115.4
3a	-				128.3
4	7.42	1	t	*J* = 1.2 Hz	119.0
5	-				132.4
6	7.33	1	d	*J* = 1.2 Hz	129.4
7	-				105.46
7a	-				136.5
8α	3.45	1	d	*J* = 17.2 Hz	27.9
8β	3.70	1	d	*J* = 17.2 Hz	
9	-				71.5
11	-				160.8
13	-				174.9
14	3.22	3	s		32.3
15	2.66	3	s		26.3
5-CH_3	2.46	3	s		21.2
2'	7.08	1	s		126.1
3'	-				105.9
3a'	-				130.6
4'	7.29	1	t	*J* = 1.2 Hz	117.5
5'	-				132.3
6'	7.16	1	d	*J* = 1.2 Hz	127.5
7'	-				105.49
7a'	-				133.6
8'	4.41	1	s		45.3
9'	-				71.3
11'	-				158.1
13'	-				172.9
14'	3.02	3	s		27.6
15'	2.82	3	s		25.9
5'-CH_3	2.43	3	s		21.4

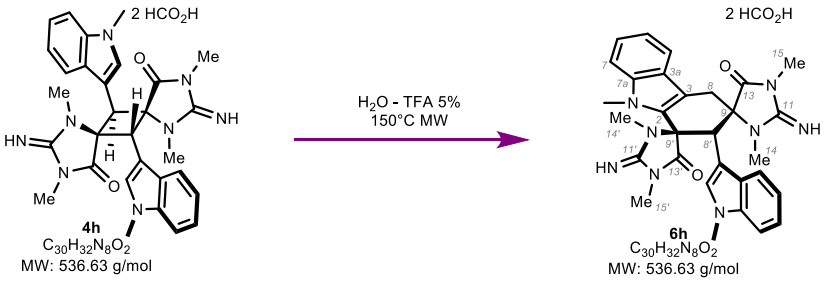
***rac*-6,6'-Chloro-tubastrindole (6e)**



- **Preparative HPLC purification:** Sunfire® column (10 x 150 mm, 5 µm), 5% to 60% MeCN in H_2O (0.1% HCO_2H) over 15 min at 1.0 mL/min; R_T = 8.0 min.
- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 7.65 (d, *J* = 2.0 Hz, 1H), 7.51 (d, *J* = 1.9 Hz, 1H), 7.38 (d, *J* = 8.7 Hz, 1H), 7.35 (d, *J* = 8.7 Hz, 1H), 7.25 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.13 (s, 1H), 7.11 (dd, *J* = 8.7, 2.0 Hz, 1H), 4.40 (s, 1H), 3.70 (d, *J* = 17.1 Hz, 1H), 3.46 (d, *J* = 17.1 Hz, 1H), 3.24 (s, 3H), 2.96 (s, 3H), 2.84 (s, 3H).
- **^{1}H NMR (400 MHz, DMSO-d$_4$)** δ 11.46 (s, 1H), 11.26 (s, 1H), 7.63 (d, *J* = 2.1 Hz, 1H), 7.37 (d, *J* = 2.0 Hz, 1H), 7.35 (d, *J* = 8.6 Hz, 1H), 7.32 (d, *J* = 8.6 Hz, 1H), 7.18 (dd, *J* = 8.6, 2.1 Hz, 1H), 7.05 (m, 1H), 7.02 (dd, *J* = 8.6, 2.0 Hz, 1H), 4.17 (s, 1H), 3.42 (d, *J* = 16.7 Hz, 1H), 3.28 (d, *J* = 16.7 Hz, 1H), 2.94 (s, 3H), 2.74 (s, 3H), 2.65 (s, 3H), 2.43 (s, 3H).
- **^{13}C NMR (101 MHz, DMSO-d$_6$)** δ 173.9, 171.3, 156.6, 153.4, 135.7, 133.1, 129.2, 129.1,126.4, 125.9, 123.7, 123.3, 122.7, 121.0, 118.2, 117.9, 113.1, 113.0, 112.0, 105.2, 68.8, 68.6, 42.6, 30.3, 26.8, 26.1, 25.2, 24.9 [attributed by 2D-NMR].
- **IR (neat)** ν_{max} = 1653, 1556, 1471, 1431, 1064.
- **HRMS ESI**: *m/z* $[M+H]^+$: 577.1633; calculated for $C_{28}H_{27}N_8{}^{35}Cl^{37}ClO_3{}^+$: 577.1629.

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
1	11.46	1	s		-
2	-				126.4
3	-				112.0
3a	-				129.1
4	7.63	1	d	*J* = 2.1 Hz	118.2
5	-				123.3
6	7.18	1	dd	*J* = 8.6, 2.1 Hz	122.7
7	7.35	1	d	*J* = 8.6 Hz	113.1
7a	-				135.7
8α	3.28	1	d	*J* = 16.7 Hz	26.8
8β	3.42	1	d	*J* = 16.7 Hz	
9	-				68.8
11	-				156.6
13	-				173.9
14	2.94	3	s		30.3
15	2.43	3	s		25.2
1'	11.26	1	s		-
2'	7.05	1	m		125.9
3'	-				105.2
3a'	-				129.2
4'	7.37	1	d	*J* = 2.0 Hz	117.9
5'	-				123.7
6'	7.02	1	dd	*J* = 8.6, 2.0 Hz	121.0
7'	7.32	1	d	*J* = 8.6 Hz	113.0
7a'	-				133.1
8'	4.17	1	s		42.6
9'	-				68.6
11'	-				153.4
13'	-				171.3
14'	2.74	3	s		26.1
15'	2.65	3	s		24.9

3.1 *rac*-1,1'-dimethyl-tubastrindole (6h)

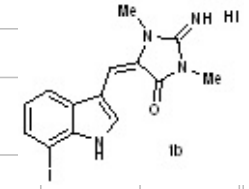


- **Preparative HPLC purification:** Sunfire® column (10 x 150 mm, 5 µm), 5% to 40% MeCN in H_2O (0.1% HCO_2H) over 15 min at 1.0 mL/min; R_T = 5.0 min.
- **^{1}H NMR (400 MHz, Methanol-d$_4$)** δ 7.69 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.52 (d, *J* = 8.3 Hz, 1H), 7.46 (d, *J* = 8.3 Hz, 1H), 7.41 (ddd, *J* = 8.3, 7.0, 1.1 Hz, 1H), 7.29 (ddd, *J* = 8.3, 7.0, 1.1 Hz, 1H), 7.27-7.19 (m, 2H), 6.98 (s, 1H), 4.61 (s, 1H), 3.83 (s, 3H), 3.81 (d, *J* = 17.4 Hz, 1H), 3.68 (d, *J* = 17.4 Hz, 1H), 3.48 (s, 3H), 3.29 (s, 3H), 3.19 (s, 3H), 2.87 (s, 3H), 2.76 (s, 3H).
- **^{13}C NMR (101 MHz, Methanol-d$_4$)** δ 173.8, 172.3, 161.9, 159.0, 140.5, 137.4, 129.3, 128.7, 127.6, 126.2, 124.2, 123.6, 121.9, 121.8, 120.3, 118.3, 116.0, 111.2, 111.1, 103.0, 72.4, 72.2, 45.9, 33.2, 32.9, 30.3, 28.3, 28.0, 26.7, 26.5.
- **IR (neat)** ν_{max} = 3005, 1778, 1672, 1545, 1469, 1364, 1203, 1132.
- **HRMS ESI**: *m/z* $[M+H]^+$: 537.2724; calculated for $C_{30}H_{33}N_8O_2^+$: 537.2721.

Assignment	^{1}H δ ppm	integration	multiplicity	*J* (Hz)	^{13}C δ ppm
2	-				123.6
3	-				116.0
3a	-				127.6
4	7.69	1	dd	*J* = 8.0, 1.0 Hz	120.3
5	7.41	1	ddd	*J* = 8.3, 7.0, 1.1 Hz	126.2
6	7.22	1	m		121.9
7	7.52	1	d	*J* = 8.3 Hz	111.2
7a	-				140.5
8α	3.68	1	d	*J* = 17.4 Hz	28.0
8β	3.81	1	d	*J* = 17.4 Hz	
9	-				72.2
11	-				161.9
13	-				173.8
14	3.29	3	s		32.9
15	2.76	3	s		26.7
N1-CH_3	3.48	3	s		30.3
2'	6.98	1	s		128.7
3'	-				103.0
3a'	-				129.3
4'	7.57	1	d	*J* = 8.1 Hz	118.3
5'	7.29	1	ddd	*J* = 8.3, 7.0, 1.1 Hz	124.2
6'	7.25	1	m		121.8
7'	7.46	1	d	*J* = 8.3 Hz	111.1
7a'	-				137.4
8'	4.61	1	s		45.9
9'	-				72.4
11'	-				159.0
13'	-				172.3
14'	3.19	3	s		28.3
15'	2.87	3	s		26.5
N1'-CH_3	3.83	3	s		33.2

5. ^{1}H and ^{13}C NMR spectra copies

LEV-966 - DMSO-D6



11.46
11.46
8.78
7.97
7.96
7.95
7.59
7.57
6.96
6.94
6.92
6.51
3.30
3.09

A (m)
11.46

B (s)
8.78

C (m)
7.96

D (d)
7.58

E (t)
6.94

F (s)
6.51

H (s)
3.09

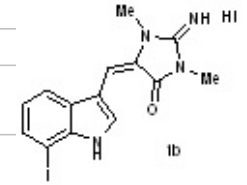
G (s)
3.30

0.96
1.00
1.00
1.23
1.05
1.02
4.68
3.39

12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.

f1 (ppm)

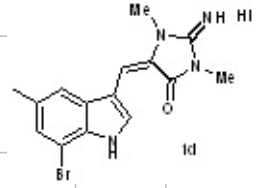
LEV-966 - DMSO-D6



161.81
150.76
137.39
130.76
128.64
128.16
126.30
121.39
118.37
109.95
104.60
77.24
27.28
25.09

f1 (ppm)

SO-510 - DMSO-D6

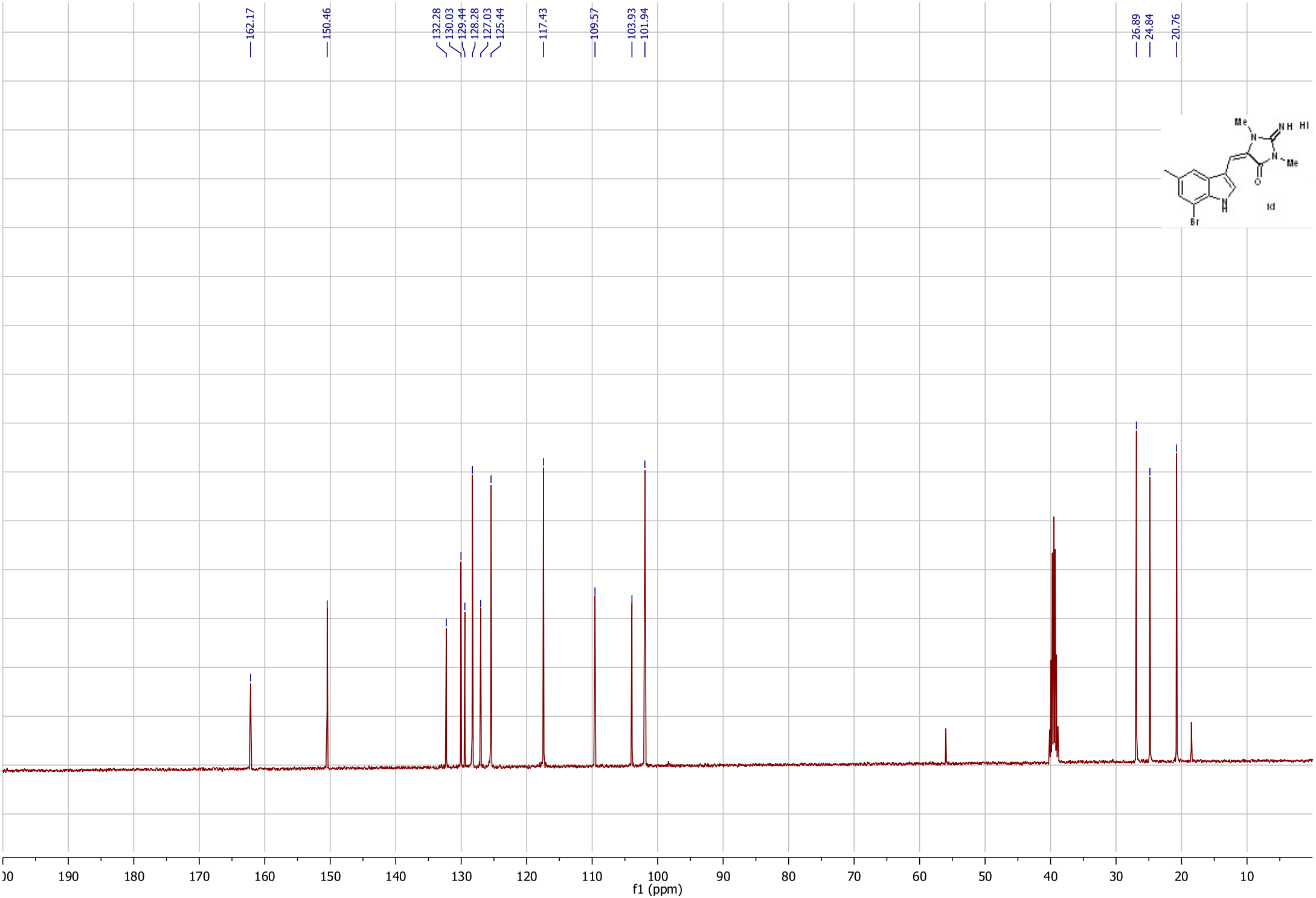


11.51
11.50
8.71
8.70
7.69
7.21
7.20
6.65
6.33
3.33
3.25
3.06
2.42

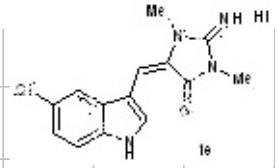
A (s) 11.51
B (d) 8.70
C (s) 7.69
D (s) 7.20
E (s) 6.65
F (s) 6.33
G (s) 3.25
H (s) 3.06
I (s) 2.42
J (s) 3.33

f1 (ppm)

SO-510 - DMSO-D6



SO-389 - DMSO-D6



11.61 11.61 11.61 | 8.74 8.74 | 8.03 8.02 | 7.44 7.42 7.15 7.15 7.13 7.13 | 6.44 | 3.26 | 3.06

A (m) 11.61

B (d) 8.74

C (d) 8.02

D (d) 7.43

E (dd) 7.14

F (s) 6.44

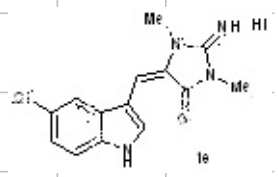
G (s) 3.26

H (s) 3.06

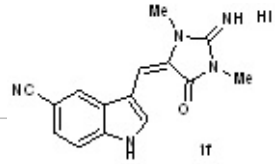
1.00 | 1.07 | 1.06 | 1.15 | 1.11 | 0.71 | 1.09 | 3.24 | 3.34

f1 (ppm)

SO-389 - DMSO-D6



SO-500 - DMSO-D6



11.95
8.89
8.89
8.09
8.09
8.07
8.07
7.93
7.93
7.45
7.44
7.43
7.42
6.74
6.42
3.26
3.06

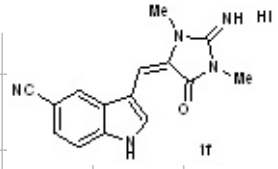
A (s) 11.95
B (d) 8.89
C (dd) 8.08
D (d) 7.93
E (dd) 7.44
F (s) 6.74
G (s) 6.42
H (s) 3.26
I (s) 3.06

1.00
1.05
1.07
1.07
1.10
0.90
1.08
3.29
3.33

12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

SO-500 - DMSO-D6

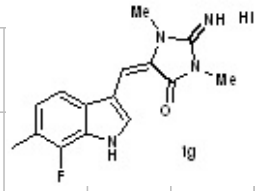


162.17
150.41
134.34
130.97
130.55
127.67
121.96
120.42
119.49
116.65
109.67
103.01
101.02
26.94
24.86

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

SO-509 - DMSO-D6



11.69
8.75
8.75
7.77
7.76
7.75
7.74
6.99
6.97
6.96
6.94
6.63
3.32
3.30
3.10

E (s)
11.69

A (d)
8.75

B (dd)
7.75

C (dd)
6.97

D (s)
6.63

G (s)
3.30

F (s)
3.32

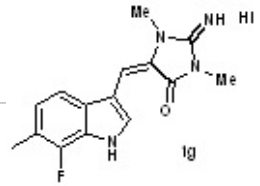
H (s)
3.10

1.04
1.00
1.03
1.01
1.03
3.35
6.74
3.15

12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

SO-509 - DMSO-D6



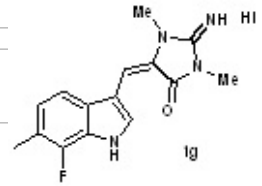
-127.36

A (s)
-127.36

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

f1 (ppm)

SO-509 - DMSO-D6

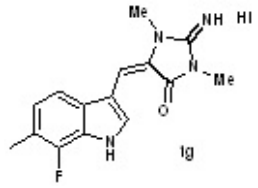


161.61
158.23
155.93
150.93
135.51
128.53
123.95
116.45
109.21
108.32
39.94
39.73
39.52
39.31
39.10
27.41
25.19

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

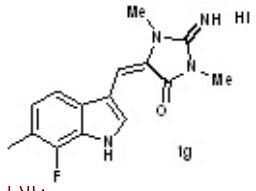
SO-509 - DMSO-D6 - HSQC



{3.15,2.95}
{3.40,3.40}
{3.30,3.31}
{3.15,3.15}
{7.01,7.01}
{6.94,6.94}
{8.84,8.84}

f2 (ppm)
f1 (ppm)

SO-509 - DMSO-D6 - HSQC

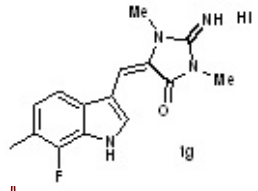


Ig

{3.35,27.35}
{6.99,108.42}
{6.97,108.28}
{6.73,107.31}
{7.77,116.51}
{8.79,128.85}

f2 (ppm)
f1 (ppm)

SO-509 - DMSO-D6 - HMBC

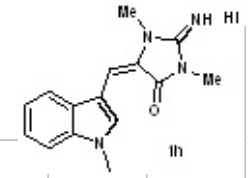


Ig

f2 (ppm)

f1 (ppm)

SO-439 - DMSO-D6



8.84 8.72 8.03 8.03 8.01 8.01 7.54 7.52 7.31 7.30 7.29 7.29 7.27 7.26 7.25 7.24 7.23 7.22 7.22 7.21 7.21 7.21 7.00 3.90 3.41 3.15 3.15

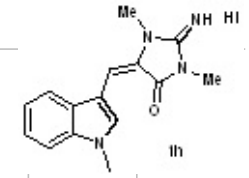
B (s) 8.72
E (td) 7.25
I (d) 3.15
A (s) 8.84
C (dd) 8.02
D (d) 7.53
F (s) 7.00
G (s) 3.90
H (s) 3.41

1.15 0.92 1.00 0.98 2.10 1.00 2.96 3.13 3.08

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

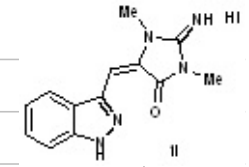
SO-439 - DMSO-D6



160.64
151.35
136.31
133.53
128.10
123.08
122.49
120.52
118.44
111.05
110.49
107.75
33.25
28.17
25.68

00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

SO-541 - DMSO-D6



7.84 7.82 7.74 7.56 7.54 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.33 7.33 7.31 7.31 7.31 7.31 7.29 7.29 7.20 7.19 7.18 7.18 7.17 7.16 7.16 7.11 7.11 7.09 7.09 7.09 7.07 7.07 6.56 6.40

3.47 3.26 3.09 3.05

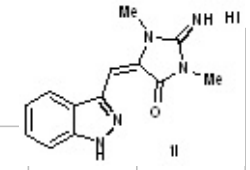
I (s) 7.74
C (ddd) 7.39
F (ddd) 7.09
H (s) 6.40
L (s) 3.09
A (d) 7.83
B (d) 7.55
E (ddd) 7.18
G (s) 6.56
J (s) 3.47
K (s) 3.26
D (ddd) 7.31
M (s) 3.05

1.03 0.30 1.39 1.03 0.53 1.24 0.55 1.01 0.30 3.00 1.25 2.83 1.35

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0

f1 (ppm)

SO-541 - DMSO-D6

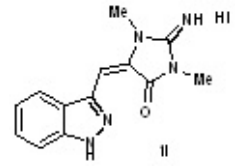


II

163.40
152.07
150.18
140.32
137.96
130.85
126.47
125.90
125.76
123.23
120.85
119.55
110.34
93.89
59.22
42.06
30.64
27.09
25.48
25.09

f1 (ppm)

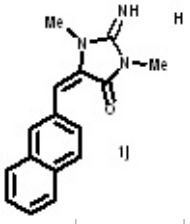
SO-541 - DMSO-D6 - HSQC



f2 (ppm)

f1 (ppm)

SO-415 - DMSO-D6



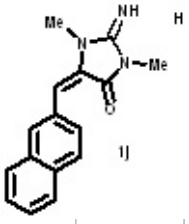
8.44 8.14 8.13 8.11 8.10 7.91 7.90 7.89 7.88 7.87 7.54 7.53 7.53 7.52 7.52 7.51 6.74 3.32 3.11

A (s) 8.44
B (dd) 8.12
C (m) 7.89
D (m) 7.52
E (s) 6.74
F (s) 3.32
G (s) 3.11

1.05 1.04 3.09 2.09 1.00 2.90 3.00

f1 (ppm)

SO-541 - DMSO-D6

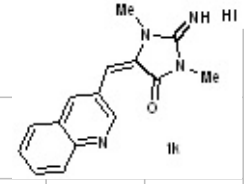


160.66
151.88
132.62
132.50
130.44
129.87
129.18
128.13
127.63
127.38
127.16
126.73
126.34
116.76
27.95
25.60

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

SO-quino - DMSO-D6



9.83 | 9.27 9.27 | 8.99 8.99 | 8.05 8.05 8.05 8.03 8.03 8.03 8.02 8.00 8.00 8.00 7.85 7.85 7.84 7.83 7.83 7.81 7.81 7.69 7.68 7.67 7.67 7.66 7.65 7.65 7.27 | 3.46 | 3.19

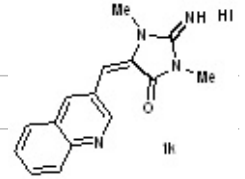
A (s) 9.83
B (d) 9.27
C (d) 8.99
D (t) 8.03
E (ddd) 7.83
F (ddd) 7.67
G (s) 7.27
H (s) 3.46
I (s) 3.19

1.14 | 1.00 | 1.07 | 2.03 | 1.03 | 0.96 | 0.98 | 3.31 | 2.97

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

SO-quino - DMSO-D6

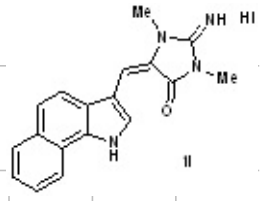


160.15
154.09
151.71
147.05
136.91
130.83
128.94
128.71
128.63
127.32
126.74
125.26
118.81
29.07
26.38

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

LEV-965 - DMSO-D6



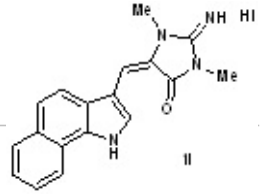
12.60
8.86
8.39
8.38
8.36
8.36
8.12
8.10
7.99
7.97
7.64
7.62
7.61
7.61
7.59
7.59
7.59
7.57
7.57
7.48
7.48
7.47
7.46
7.46
7.44
7.44
6.95
3.41
3.15

A (s) 12.60
B (s) 8.86
C (dd) 8.37
D (d) 8.11
E (d) 7.98
F (ddd) 7.46
G (s) 6.95
H (d) 7.63
I (ddd) 7.59
J (s) 3.41
K (s) 3.15

1.00
1.08
1.83
1.10
1.11
0.99
1.12
1.11
1.05
3.34
3.16

13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

LEV-965 - DMSO-D6



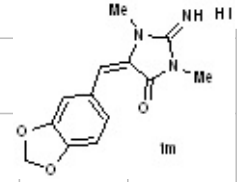
II

161.14
151.27
130.30
130.08
128.36
126.91
125.74
124.65
124.09
123.65
121.81
120.67
120.50
118.43
110.38
109.15
27.86
25.50

)0 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

f1 (ppm)

SO-460 - DMSO-D6



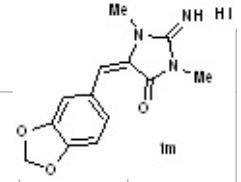
8.86
7.94
7.93
7.43
7.43
7.41
7.40
6.98
6.96
6.63
6.08
3.29
3.11

A (s) 8.86
B (d) 7.94
C (dd) 7.42
D (d) 6.97
E (s) 6.63
F (s) 6.08
G (s) 3.29
H (s) 3.11

0.96
1.00
1.04
1.12
0.99
2.22
3.44
2.88

f1 (ppm)

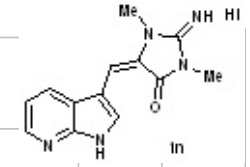
SO-460 - DMSO-D6



160.67
152.00
148.15
147.07
127.10
126.75
126.65
118.90
109.47
108.13
101.46
28.08
25.72

f1 (ppm)

SO-482 - DMSO-D6



12.33

8.93 8.92 8.87 8.45 8.43 8.33 8.33 8.32 8.32 8.31

7.25 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.04 7.04 7.03 7.01 7.00

3.43 3.42 3.17 3.16

A (s) 12.33

I (s) 8.87

B (d) 8.92

D (m) 8.32

C (d) 8.44

F (m) 7.02

E (m) 7.24

H (d) 3.17

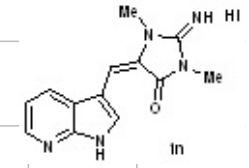
G (d) 3.42

1.00 1.31 1.01 1.15 1.15 1.16 1.14 3.46 3.39

f1 (ppm)

SO-482 - DMSO-D6

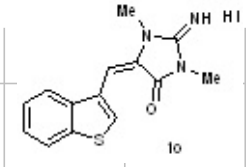
160.80
151.67
148.20
143.81
129.81
127.08
124.38
119.86
116.49
110.67
107.29
28.27
25.78



In

f1 (ppm)

SO-450 - DMSO-D6



8.75 | 8.10 | 8.10 | 8.08 | 8.03 | 8.02 | 8.01 | 7.49 | 7.49 | 7.47 | 7.47 | 7.47 | 7.45 | 7.45 | 7.44 | 7.43 | 7.42 | 7.41 | 7.40 | 7.40 | 7.03 | 6.37 | 3.29 | 3.06

A (s) 8.75

B (d) 8.09

C (d) 8.02

D (ddd) 7.47

I (m) 7.42

E (s) 7.03

F (s) 6.37

G (s) 3.29

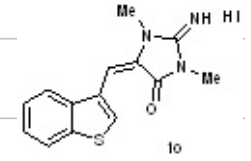
H (s) 3.06

1.00 | 1.22 | 1.14 | 1.28 | 1.15 | 0.89 | 1.17 | 2.99 | 3.82

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

SO-450 - DMSO-D6



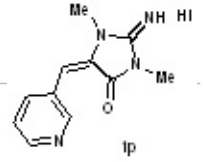
1o

162.32
150.63
139.24
139.01
132.14
128.18
126.63
124.99
124.80
123.37
122.26
100.94
27.52
25.49

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

BYM-pyridine - DMSO-D6



9.76
8.98 8.97 8.58 8.58 8.57 8.56 8.44 8.44 8.43 8.42 8.42 8.41
7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.48
7.09
3.42
3.16

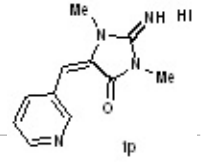
A (s) 9.76
B (d) 8.98
C (dd) 8.57
D (dt) 8.43
E (ddd) 7.49
F (s) 7.09
G (s) 3.42
H (s) 3.16

0.94 1.00 1.01 1.00 1.08 1.00 2.83 2.95

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

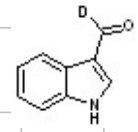
BYM-pyridine - DMSO-D6



160.08
154.11
151.03
149.88
136.99
128.93
127.89
123.26
118.66
29.03
26.34

f1 (ppm)

BYM-004 - DMSO-D6



12.11

8.28 8.27 8.11 8.11 8.09 8.08 7.53 7.52 7.52 7.50 7.50 7.49 7.29 7.28 7.26 7.26 7.24 7.23 7.21 7.21 7.19 7.19

A (s)
12.11

B (d)
8.28

C (dd)
8.10

D (m)
7.51

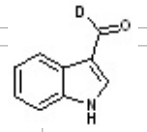
E (pd)
7.24

1.00 0.85 0.79 0.85 1.81

13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

f1 (ppm)

BYM-004 - DMSO-D6

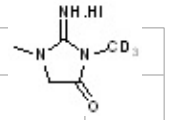


138.34
137.01
124.11
123.40
122.06
120.78
117.73
112.36

80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

BYM-001 - DMSO-D6



8.95

4.25

3.09

C (s)
8.95

B (s)
4.25

A (s)
3.09

3.73

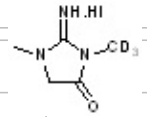
2.00

2.75

0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0

f1 (ppm)

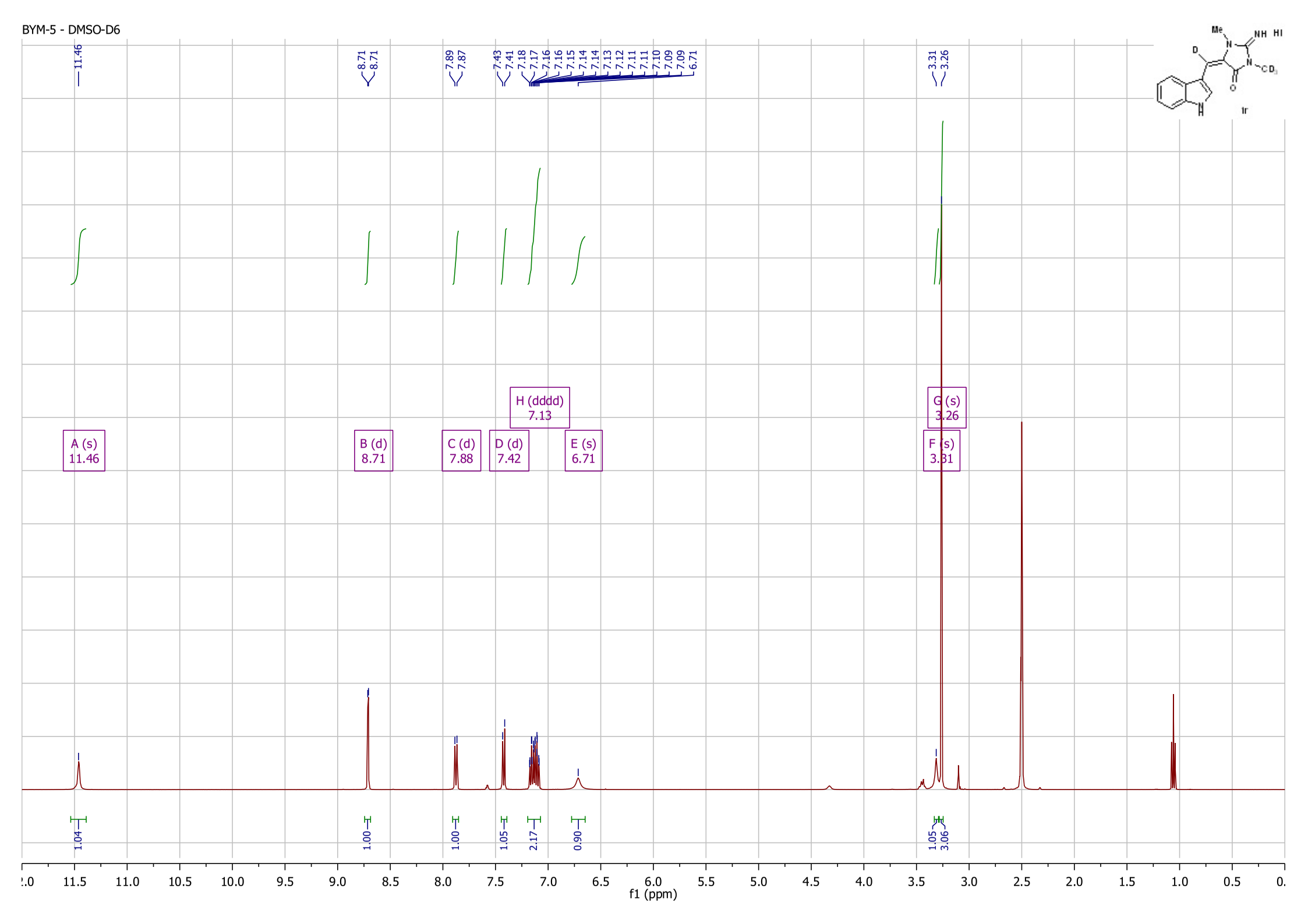
BYM-001 - DMSO-D6



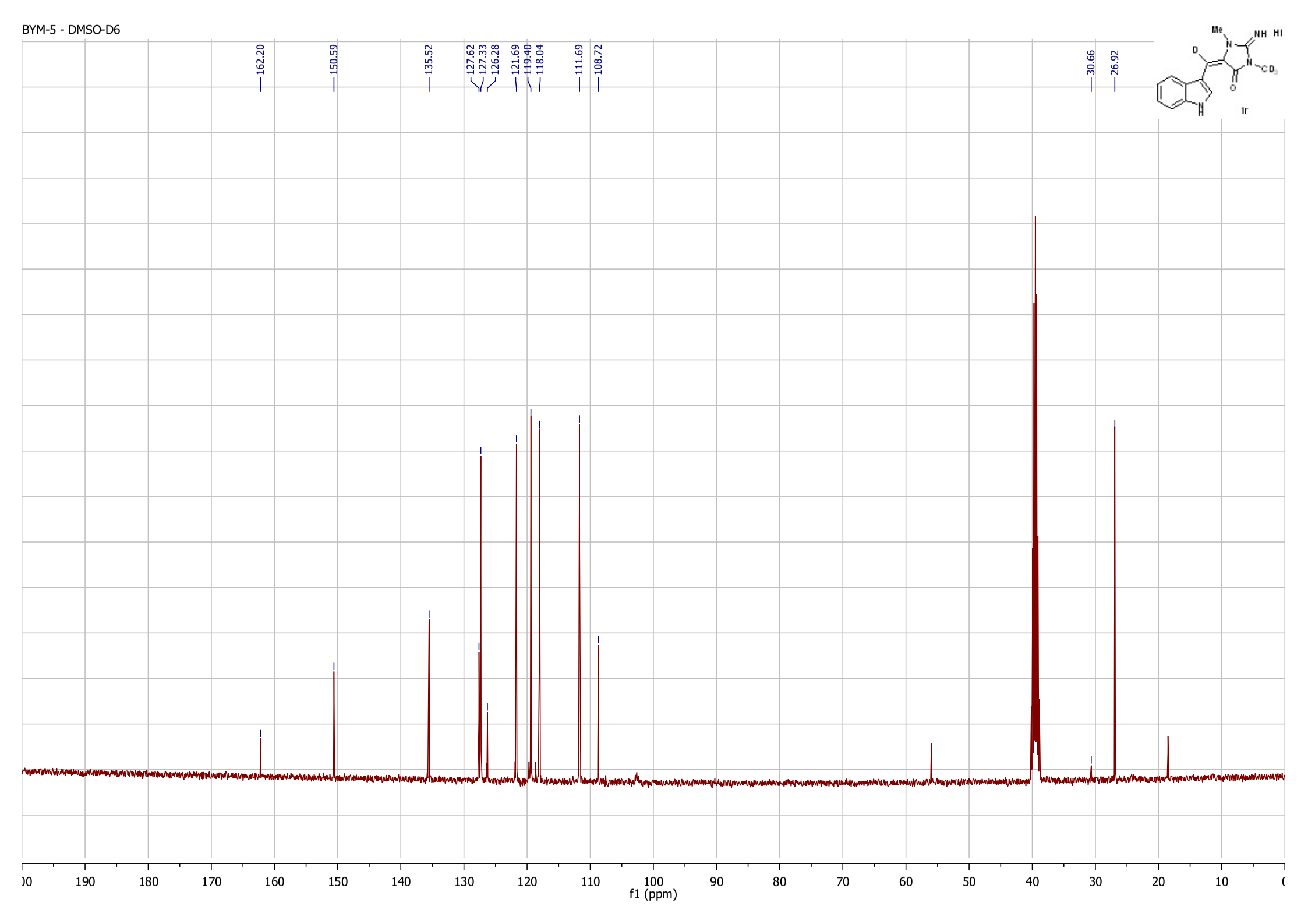
169.63
157.72
53.28
31.77

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

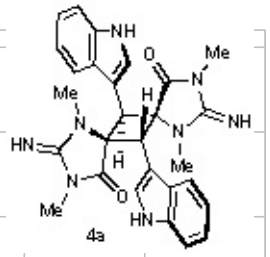
f1 (ppm)


BYM-5 - DMSO-D6
11.46
8.71
8.71
7.89
7.87
7.43
7.41
7.18
7.17
7.16
7.16
7.15
7.14
7.14
7.13
7.12
7.11
7.11
7.10
7.09
7.09
6.71
3.31
3.26
A (s)
11.46
B (d)
8.71
C (d)
7.88
D (d)
7.42
H (dddd)
7.13
E (s)
6.71
G (s)
3.26
F (s)
3.31
1.04
1.00
1.00
1.05
2.17
0.90
1.05
3.06
f1 (ppm)

BYM-5 - DMSO-D6


162.20
150.59
135.52
127.62
127.33
126.28
121.69
119.40
118.04
111.69
108.72
30.66
26.92
Me
NH HI
D
N
N
CD3
O
H
Ir
f1 (ppm)

ND-810.5 - Methanol-D4



7.46 7.46 7.45 7.44 7.44 7.44 7.43 7.40 7.40 7.21 7.21 7.19 7.19 7.19 7.17 7.17 7.10 7.10 7.08 7.08 7.08 7.06 7.06 6.96 6.95 6.95 6.94 6.93 6.93 5.54 5.54

3.39 3.35

B (d) 7.40

E (dt) 6.94

A (dt) 7.45

D (ddd) 7.08

C (ddd) 7.19

F (d) 5.54

H (s) 3.35

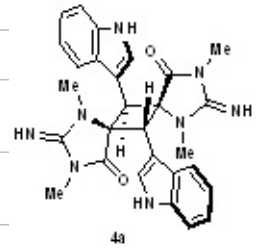
G (s) 3.39

2.01 2.00 2.27 2.17 2.07 2.04 6.38 6.81

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

ND-810.5 - Methanol-D4



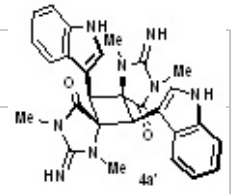
4a

174.77
159.20
137.53
127.83
124.98
123.76
121.17
117.22
113.28
104.44
70.10
58.83
30.38
27.41

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

LEV 860 E - Methanol-D4



8.42 | 7.39 7.37 7.16 7.16 7.15 7.14 7.14 7.13 7.13 7.12 7.05 7.05 7.03 7.03 7.03 7.01 7.01 6.99 6.98 6.98 6.97 6.96 6.96 | 5.21 5.21 | 3.61 | 3.20 3.09

I (s) 8.42

A (d) 7.38

B (m) 7.14

C (ddd) 7.03

D (dt) 6.97

E (d) 5.21

F (s) 3.61

G (s) 3.20

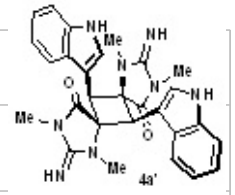
H (s) 3.09

3.25 | 2.00 | 4.02 | 2.14 | 2.02 | 1.95 | 2.64 | 2.54 | 2.48

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

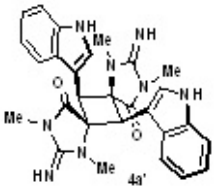
LEV 860 E - Methanol-D4



176.50
174.91
159.35
157.35
137.57
127.90
124.52
123.43
120.84
117.73
112.98
105.56
71.39
69.62
47.73
31.74
27.19
27.03
26.15

f1 (ppm)

LEV 860 E - Methanol-D4 HSQC



{3.61,26.77}

{3.31,26.77}

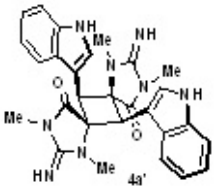
{3.09,25.93}

{3.20,31.74}

f2 (ppm)

f1 (ppm)

LEV 860 E - Methanol-D4 - HMBC

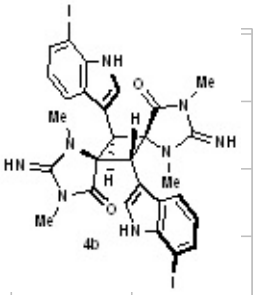


{3.63,157.35}
{3.34,159.45}
{3.23,159.45}
{3.12,157.35}
{3.12,172.49}
{3.34,176.50}

f1 (ppm)
140
145
150
155
160
165
170
175
180
185
190

f2 (ppm)
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7

SO-537-F2 - DMSO-D6



8.14 | 7.51 | 7.50 | 7.28 | 7.28 | 6.91 | 6.90 | 6.78 | 6.77 | 6.76 | 5.21 | 3.23 | 2.99

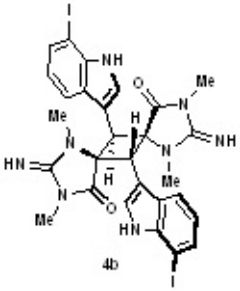
A (s) 8.14
B (d) 7.50
C (d) 7.28
D (d) 6.90
E (t) 6.77
F (s) 5.21
G (s) 3.23
H (s) 2.99

0.67 | 2.00 | 1.92 | 2.12 | 2.08 | 1.99 | 6.74 | 6.50

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

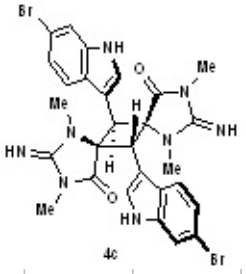
SO-537-F2 - DMSO-D6



173.61
154.12
137.24
130.63
127.22
124.05
121.02
116.97
107.24
77.32
66.99
45.91
28.40
25.62

f1 (ppm)

LEV-847c - Methanol-D4



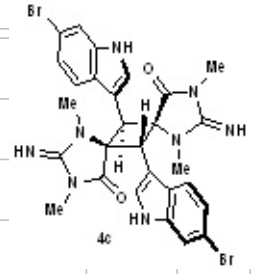
8.30
7.58
7.57
7.36
7.17
7.15
7.14
6.85
6.84
6.83
6.82
5.43
3.28

F (s) 8.30
A (s) 7.58
B (s) 7.36
C (d) 7.15
D (d) 6.84
E (s) 5.43

3.27
1.86
1.70
1.90
2.00
1.76

f1 (ppm)

LEV-847c - Methanol-D4

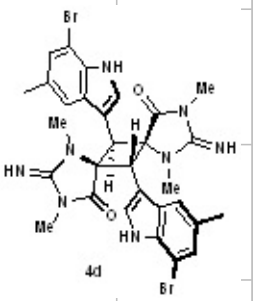


174.89
138.32
133.53
126.97
125.67
124.12
119.03
116.89
115.96
105.71
69.62
48.14
29.95
26.98

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

SO-538-F1 - DMSO-D6



7.18 7.18 7.18 7.17 7.16 7.16 6.62 5.14 5.14 3.18 2.97 2.29

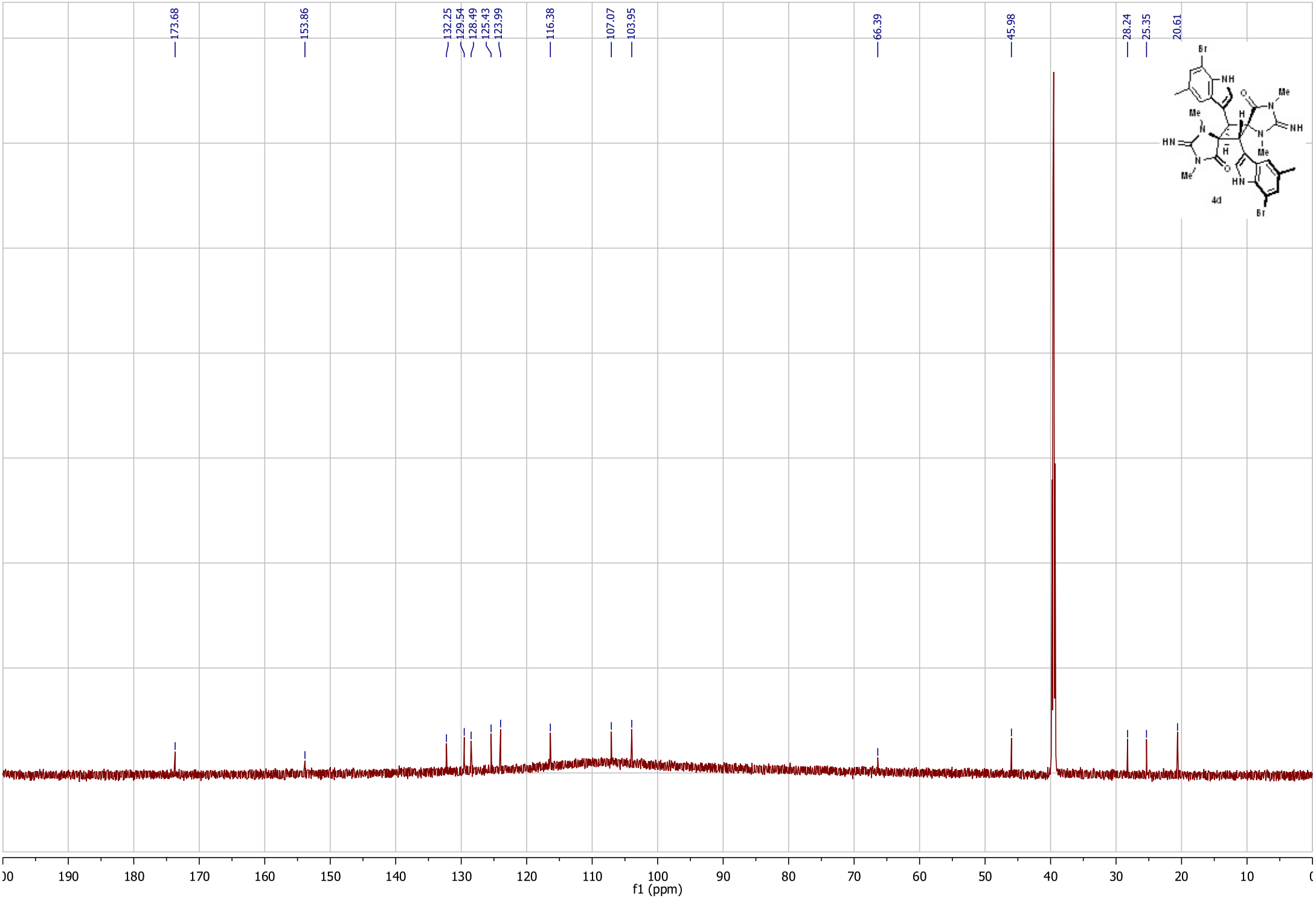
C (d) 7.16
B (dd) 7.18
D (s) 6.62
A (s) 5.14
E (s) 3.18
F (s) 2.97
G (s) 2.29

1.98 1.62 1.86 2.00 6.31 6.26 6.48

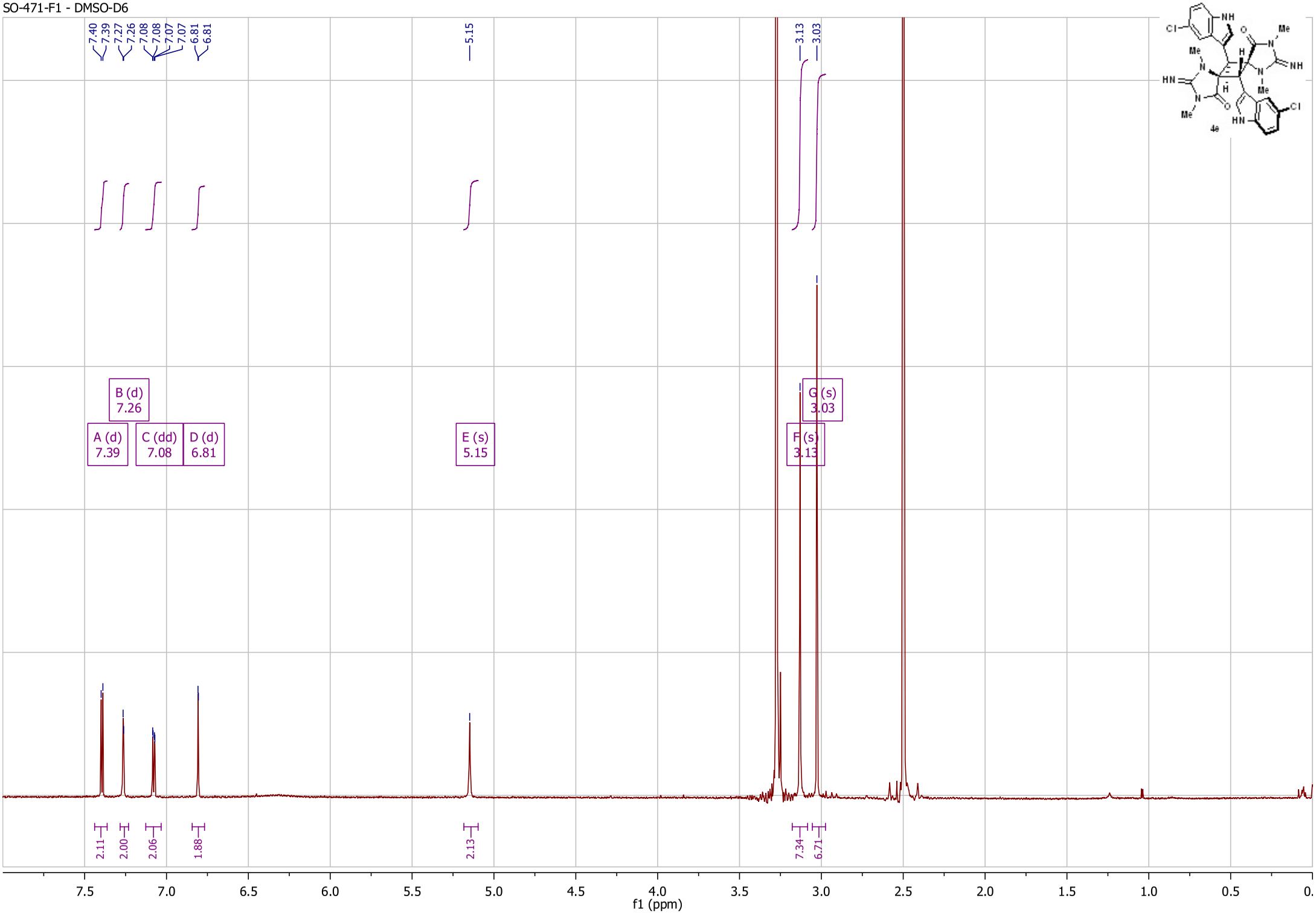
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

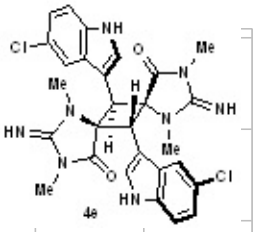
SO-538-F1 - DMSO-D6



SO-471-F1 - DMSO-D6



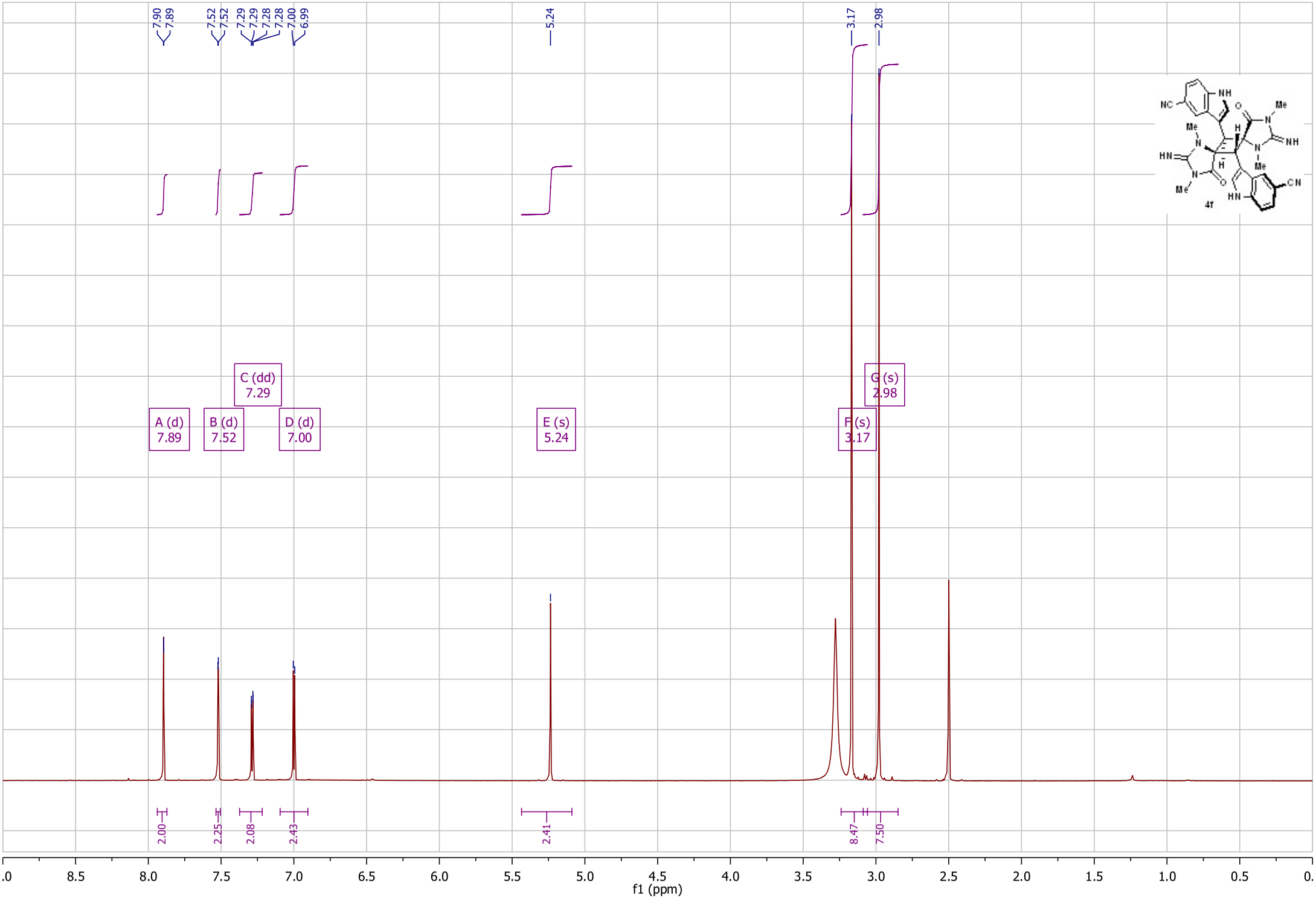
SO-399-F1 - DMSO-D6



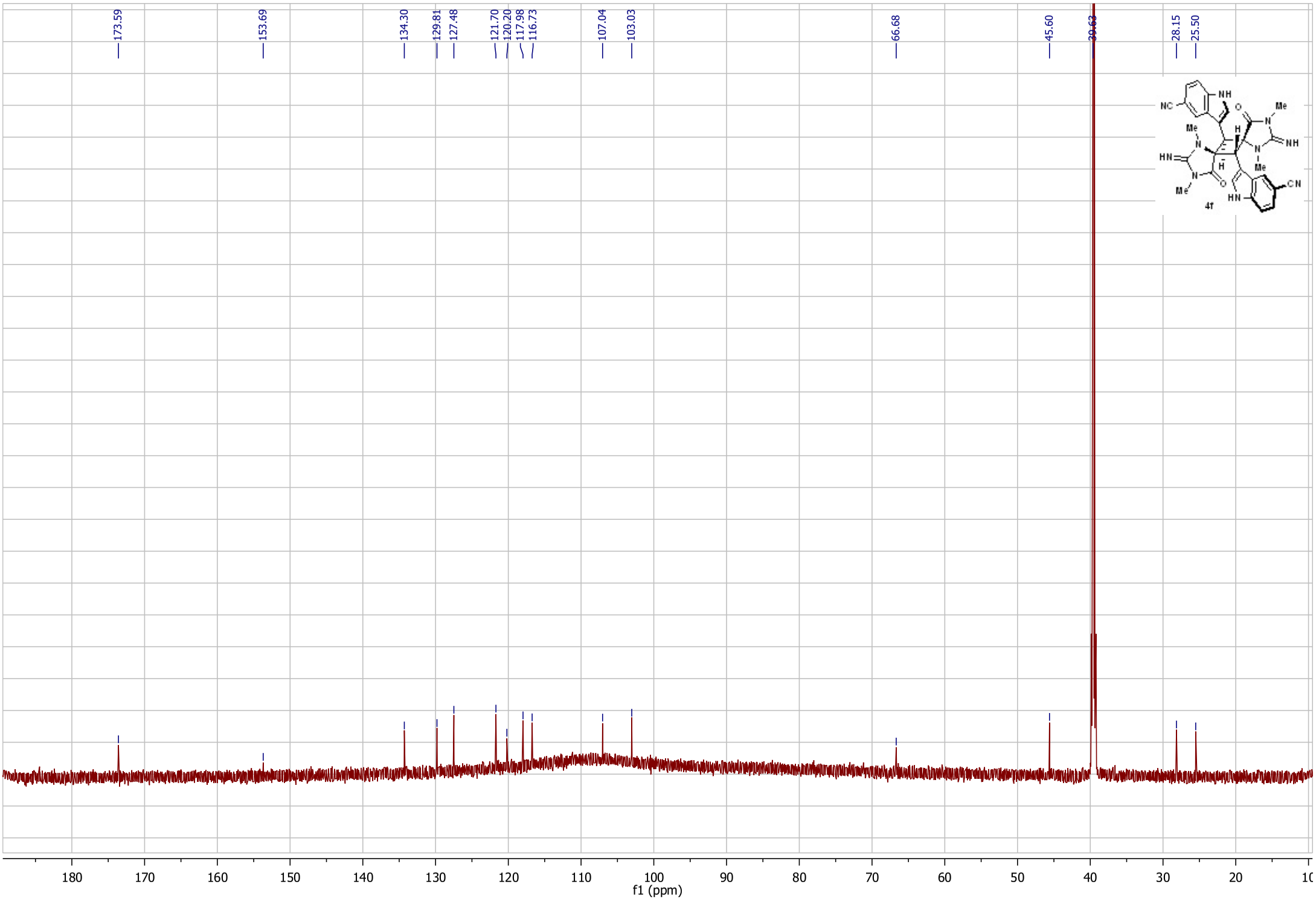
176.20
153.66
134.03
127.91
124.62
123.61
121.47
116.32
113.27
106.00
66.33
45.95
39.52
28.05
25.41

f1 (ppm)

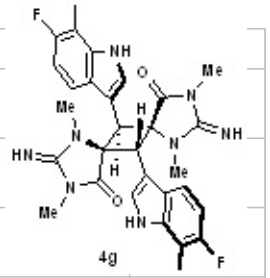
SO-529-F1 - DMSO-D6



SO-529-F1 - DMSO-D6



SO-535-F1 - DMSO-D6



7.20 | 6.78 6.77 6.76 6.68 6.67 6.67 6.66 | 6.25 | 5.17 | 3.27 3.19 | 2.95 | 2.35 2.34

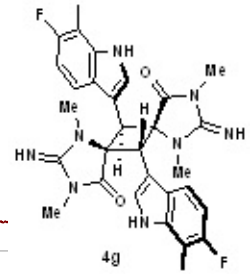
B (s) 7.20 | C (t) 6.77 | D (dd) 6.67 | E (s) 6.25 | A (s) 5.17 | F (s) 3.19 | G (s) 2.95 | H (d) 2.35

1.69 | 1.74 | 1.82 | 1.91 | 2.00 | 6.12 | 5.60 | 5.69

7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

SO-535-F1 - DMSO-D6 - HSQC

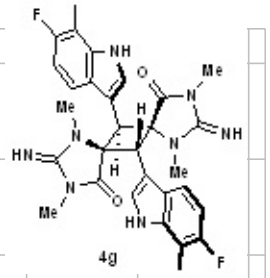


4g

{2.35,8.94}
{2.95,25.40}
{3.19,28.16}
{5.17,45.78}
{6.77,107.62}
{6.67,114.76}
{7.19,123.21}

f2 (ppm)
f1 (ppm)

SO-535-F1 - DMSO-D6



4g

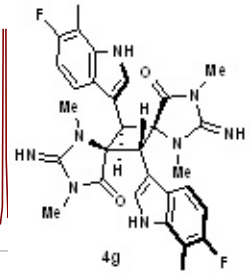
-75.76

-123.82

-30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220

f1 (ppm)

SO-535-F1 - DMSO-D6 - HMBC



4g

{3.19,66.94}

{6.67,107.65}

{2.34,106.63}

{6.77,123.21}

{2.35,135.48}

{3.19,154.13}

{2.95,154.13}

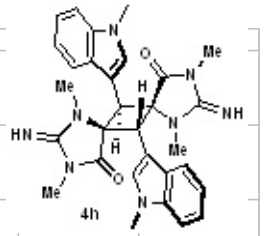
{2.35,157.34}

{2.95,173.83}

f2 (ppm)

f1 (ppm)

SO-444-F1 - DMSO-D6



7.50 7.49 7.31 7.31 7.23 7.23 7.22 7.22 7.22 7.21 7.21 7.09 7.09 7.08 7.08 7.08 7.07 7.07 6.89 6.89 6.89 6.88 6.88 6.88

5.40 5.40

3.82

3.38 3.27

B (d) 7.31

E (dt) 6.88

A (d) 7.49

D (ddd) 7.08

C (ddd) 7.22

F (d) 5.40

G (s) 3.82

I (s) 3.27

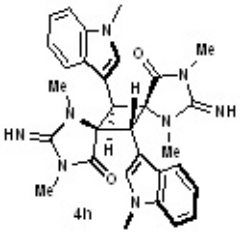
H (s) 3.38

2.00 2.46 3.09 2.90 2.76 2.71 8.50 9.39 8.82

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

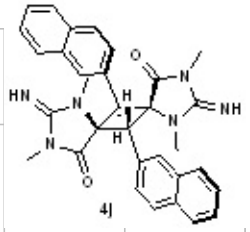
SO-444-F1 - DMSO-D6



172.84
157.05
136.01
128.20
126.63
122.10
119.75
116.55
110.37
102.41
68.24
45.80
32.67
29.89
27.13

f1 (ppm)

SO-461-F1 - DMSO-D6



7.88 7.87 7.87 7.86 7.80 7.79 7.52 7.51 7.34 6.87 6.86 5.21 3.21 3.04

H (dd) 7.87 | F (d) 7.52 | C (s) 3.04

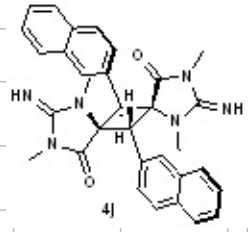
G (d) 7.80 | E (s) 7.34 | D (d) 6.87 | A (s) 5.21 | B (s) 3.21

3.96 2.02 3.26 2.02 1.91 2.00 6.22 6.69

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

SO-461-F1 - DMSO-D6

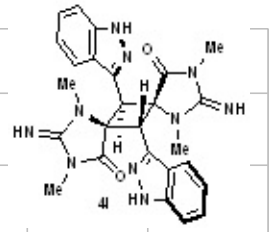


173.13
153.51
132.51
131.83
131.53
128.03
127.62
127.39
126.41
126.04
124.31
124.03
66.44
51.05
28.17
25.63

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

SO-545-F1 - DMSO-D6



7.51 7.50 7.36 7.35 7.35 7.35 7.34 7.34 7.33 7.06 7.05 7.04 6.97 6.96

5.44

3.12 2.99 2.97

B (ddd) 7.35

D (d) 6.97

A (d) 7.51

C (m) 7.05

E (s) 5.44

H (s) 3.12

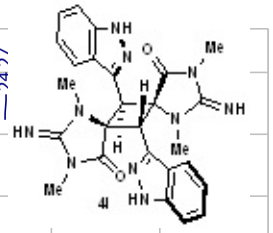
G (s) 2.97

2.85 3.20 1.86 2.51 2.00 7.09 6.04

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

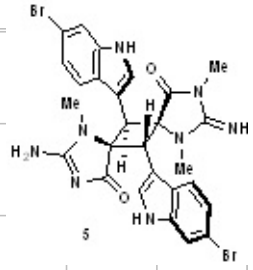
SO-545-F1 - DMSO-D6



172.55
158.95
140.83
136.57
126.38
120.61
120.46
118.13
110.47
64.68
47.58
42.09
26.04
24.27

f1 (ppm)

ND-808-F8 - DMSO-D6



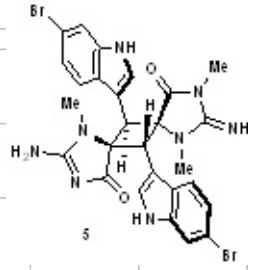
11.19 11.18 11.17 11.16 7.56 7.55 7.44 7.44 7.44 7.43 7.16 7.16 7.16 7.15 7.07 7.07 7.05 7.05 7.05 7.04 7.03 7.02 7.02 6.94 6.92 6.81 6.79 5.10 5.02 5.02 3.09 3.05 3.02 3.01

D (d) 11.17
C (d) 11.18
J (dd) 7.03
F (s) 7.55
I (dd) 7.06
E (s) 7.56
H (dd) 7.16
G (dd) 7.44
K (d) 6.93
L (d) 6.80
B (s) 5.02
A (s) 5.10
N (s) 3.05
M (s) 3.09
O (s) 3.01

1.06 1.20 1.26 1.68 0.96 0.97 1.01 1.12 0.94 0.98 1.00 0.69 2.87 2.98 3.10

f1 (ppm)

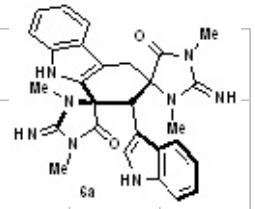
ND-808-F8 - DMSO-D6



188.25
174.30
170.13
165.80
158.19
136.47
136.33
126.33
125.97
124.58
123.46
121.76
121.59
119.15
118.65
114.25
114.18
114.00
107.17
106.95
68.62
46.03
44.74
28.44
27.79
25.45

f1 (ppm)

SO-tuba-Methanol-D4



8.48 7.67 7.65 7.43 7.41 7.39 7.33 7.33 7.31 7.31 7.30 7.29 7.29 7.20 7.18 7.17 7.16 7.14 7.12 7.07 4.55 3.81 3.77 3.62 3.58 3.31 3.07 2.89 2.66

A (s) 8.48
B (d) 7.66
C (t) 7.41
D (ddd) 7.31
E (t) 7.18
F (s) 7.12
G (s) 7.07
H (s) 4.55
I (d) 3.79
J (d) 3.60
K (s) 3.31
L (s) 3.07
M (s) 2.89
N (s) 2.66

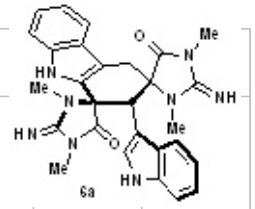
0.85 1.04 0.96 2.28 1.24 3.37 1.31 0.95 1.10 1.04 3.17 3.00 2.98 2.78

11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

SO-tuba-Methanol-D4

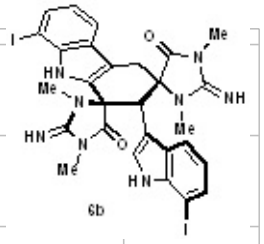
174.62
172.87
169.23
161.16
158.28
139.46
136.65
128.90
126.58
125.47
124.82
123.62
121.22
119.96
118.22
114.48
112.87
112.73
104.50
72.15
71.78
44.81
32.76
27.84
27.68
26.42
26.17



6a

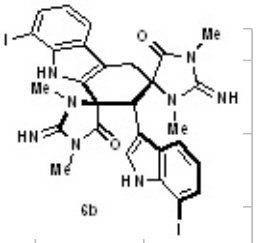
f1 (ppm)

LEV-1100-F4 - Methanol-D4



f1 (ppm)

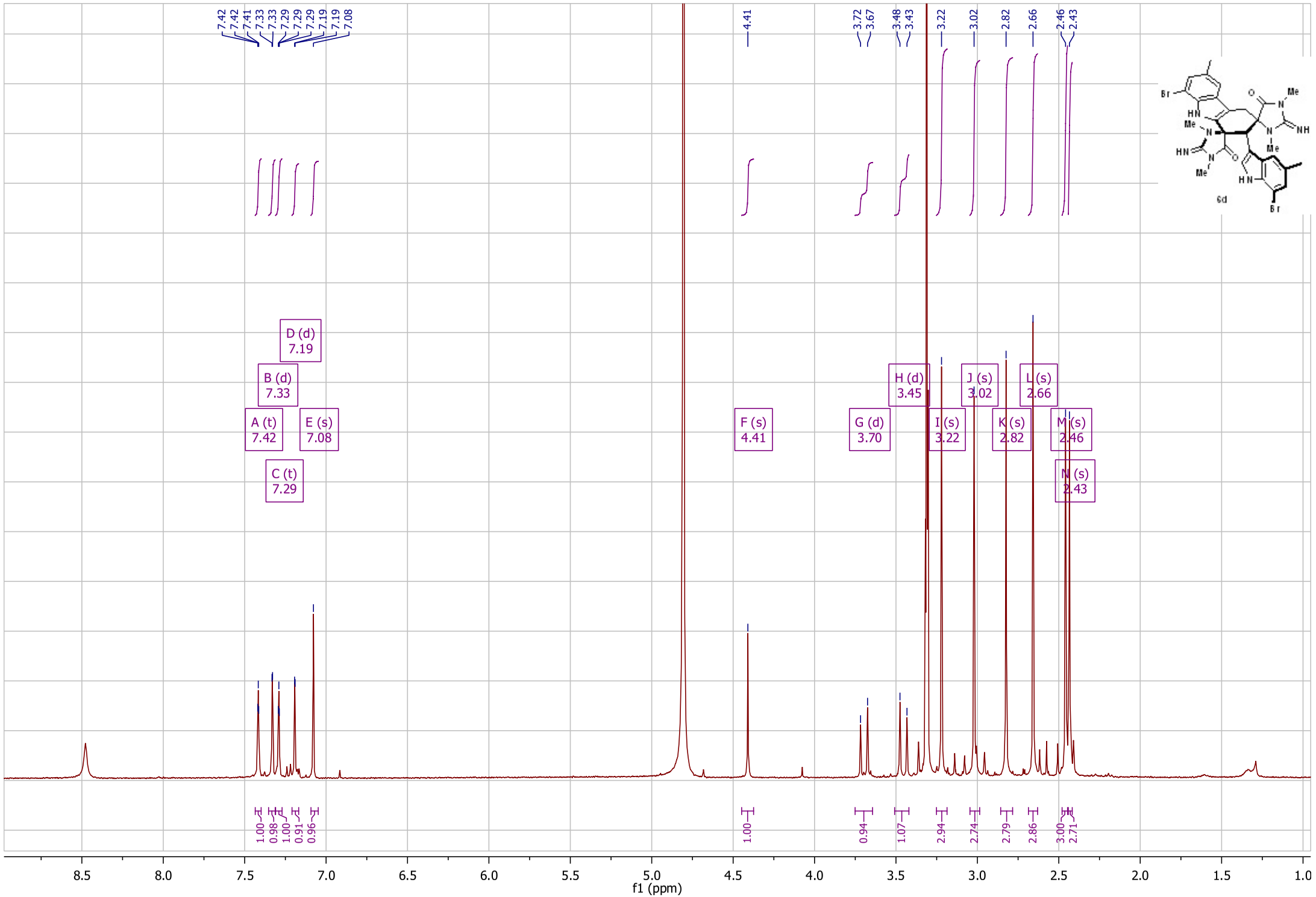
LEV-1100-F4 - Methanol-D4



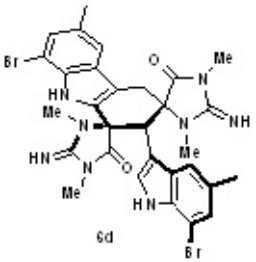
174.73
172.84
160.80
158.22
141.59
138.53
134.98
132.79
129.34
127.06
126.01
125.79
122.87
122.82
120.10
118.67
116.36
106.54
76.98
76.68
71.45
71.32
45.48
32.37
27.95
27.71
26.34
26.02

f1 (ppm)

LEV-1102-F4 - Methanol-D4



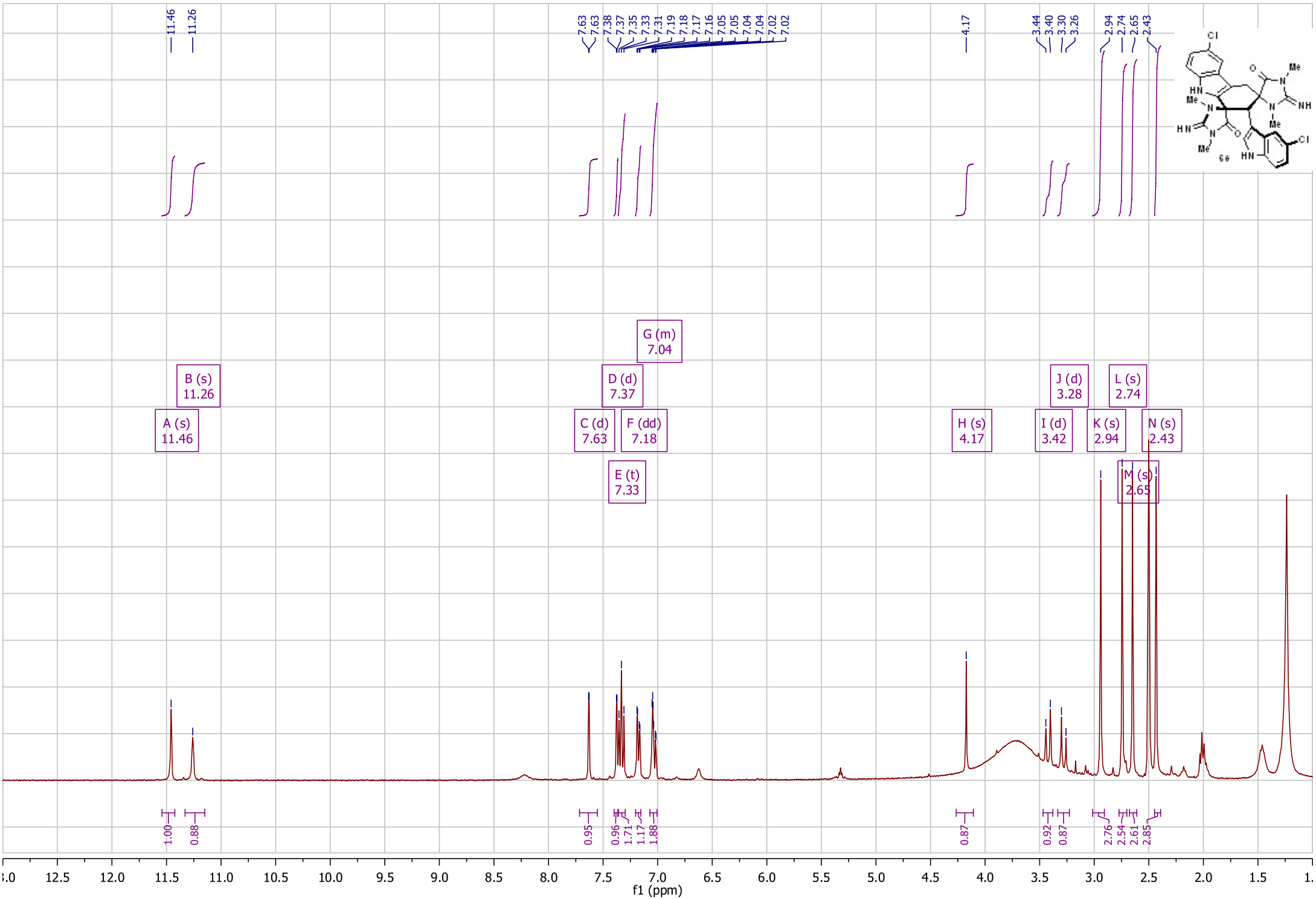
LEV-1102-F4 - Methanol-D4



174.88
172.92
160.76
158.06
136.46
133.57
132.36
132.27
130.66
129.40
128.25
127.50
126.81
126.13
118.97
117.54
115.39
105.93
105.49
105.46
71.54
71.26
45.28
32.32
27.91
27.59
26.29
25.93
21.38
21.16

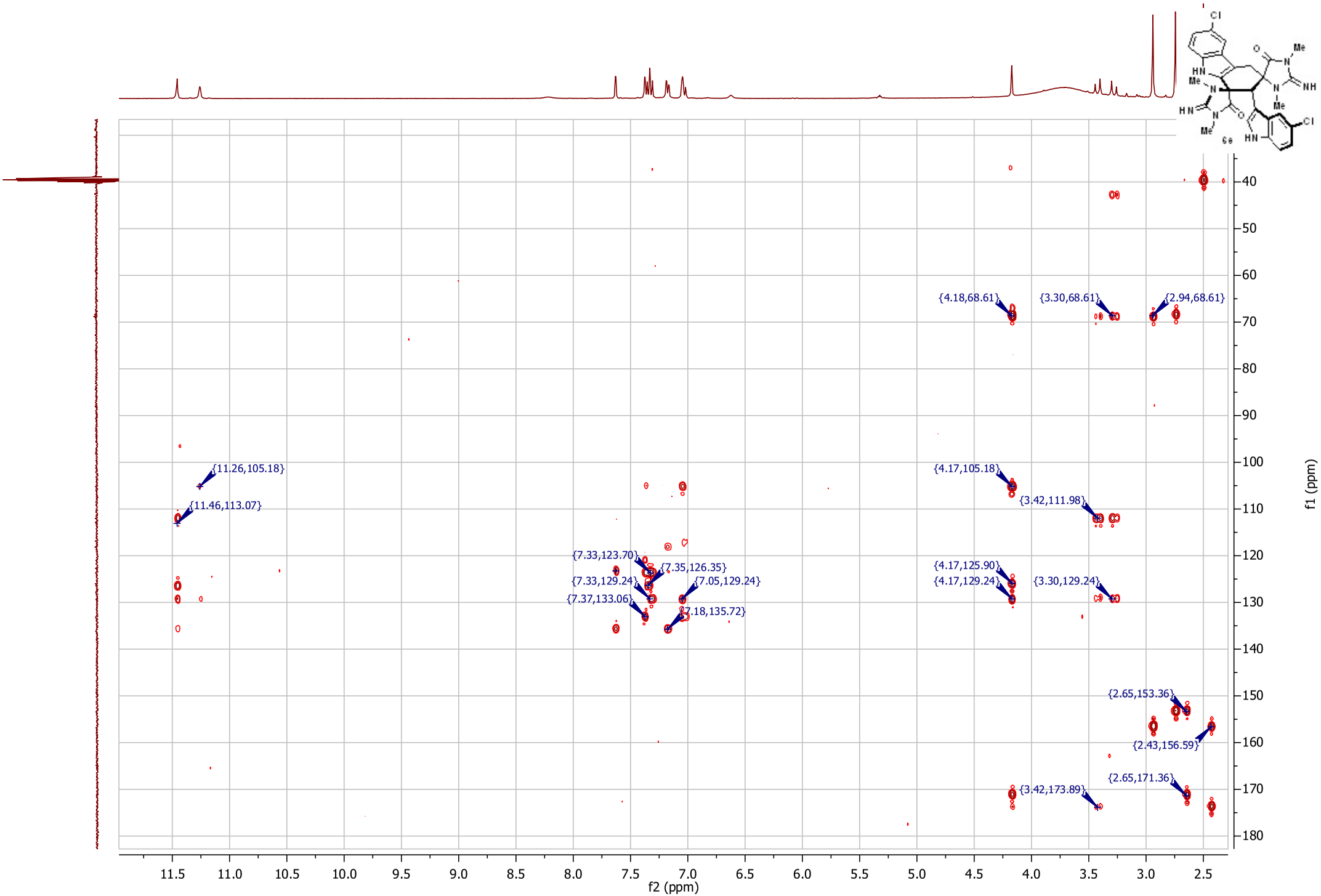
f1 (ppm)

LEV-1101 - DMSO-D6

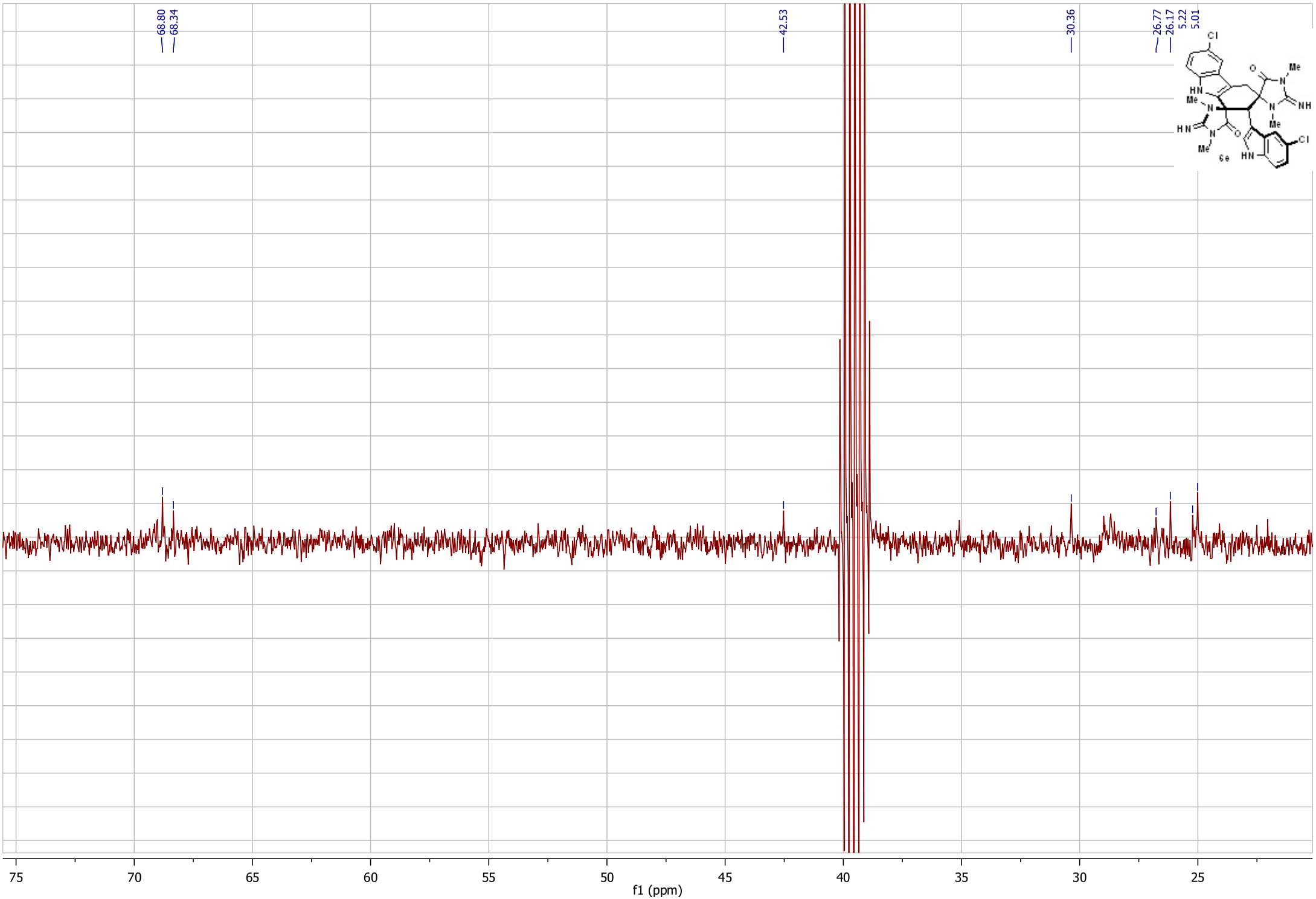


LEV-1101 - DMSO-D6 - HSQC

LEV-1101 - DMSO-D6 - HMBC

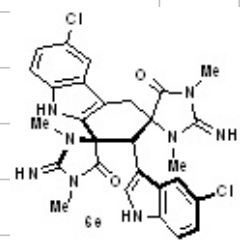


LEV-1101 - DMSO-D6



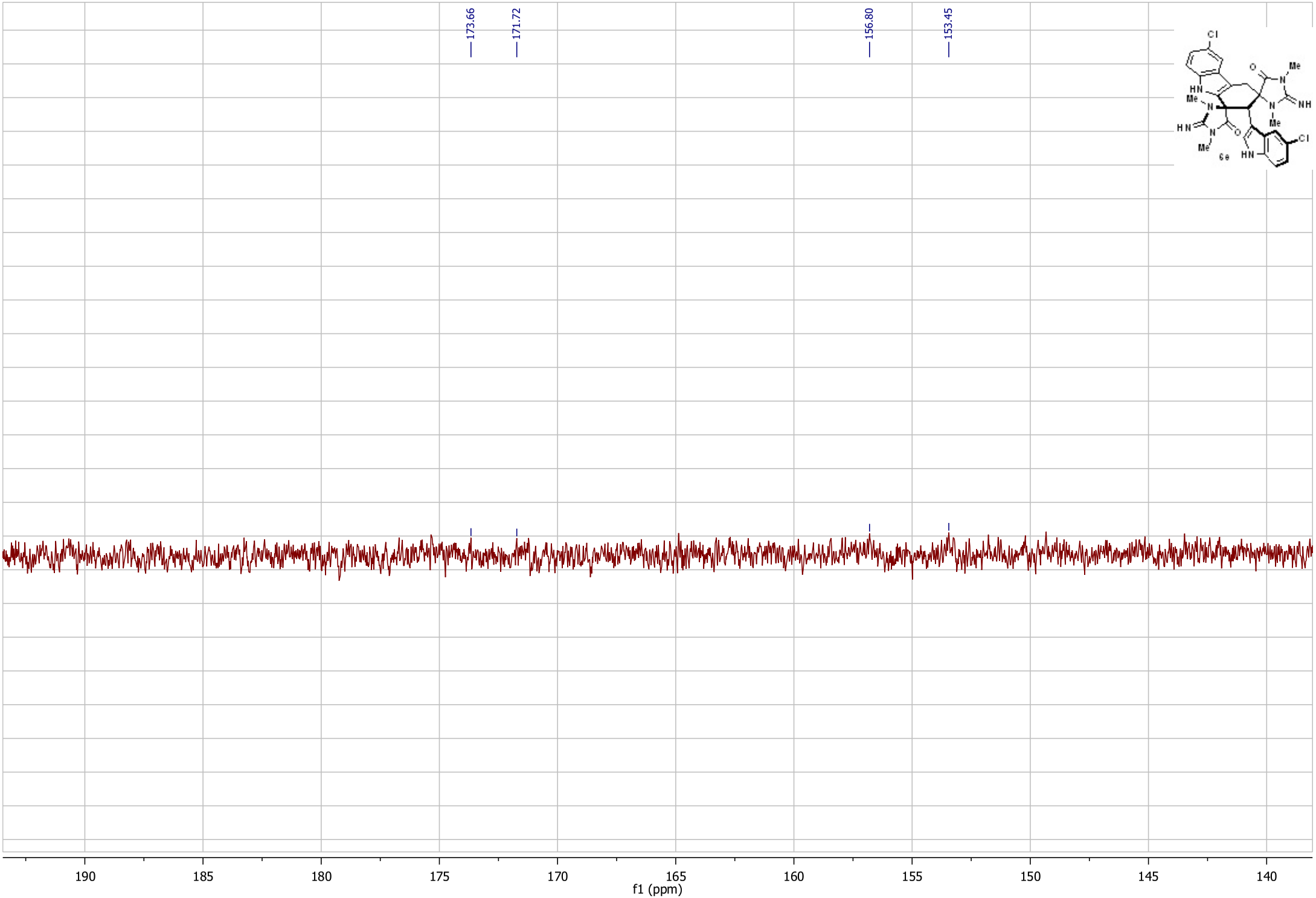
LEV-1101 - DMSO-D6

136.20
133.11
129.35
129.26
126.42
126.00
123.61
123.55
122.69
120.98
118.15
116.87
112.93
112.82
112.02
105.18

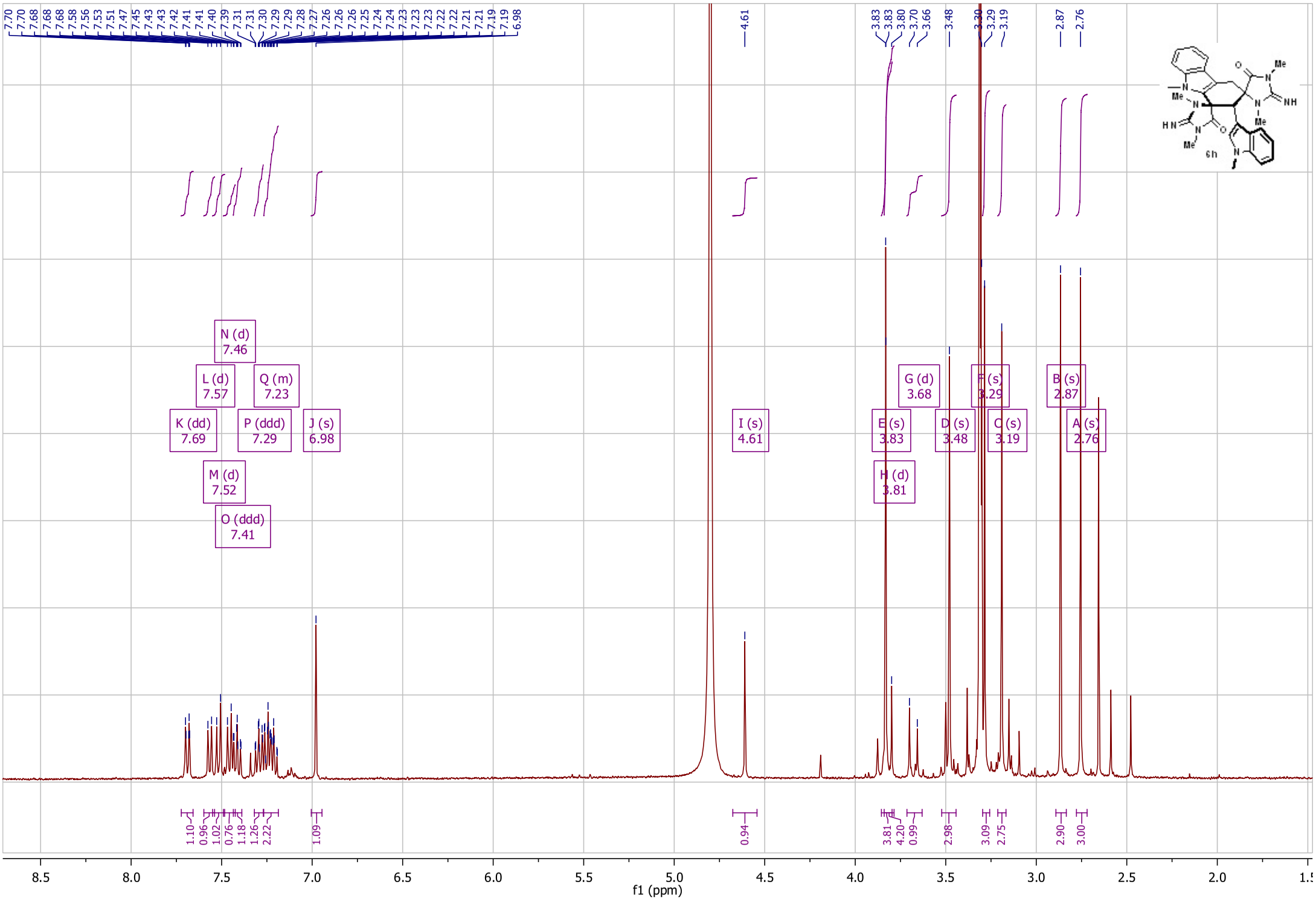


145
140
135
130
125
120
115
110
105
100
95
f1 (ppm)

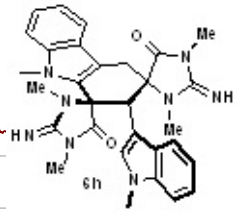
LEV-1101 - DMSO-D6



LEV-1104-F2 - Methanol-D4



LEV-1104-F2 - Methanol-D4 -COSY



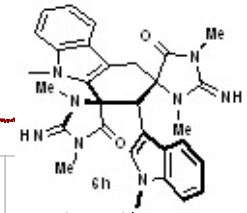
f2 (ppm)

8.10 8.05 8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85

f1 (ppm)

6.8 6.9 7.0 7.1 7.2 7.3 7.4 7.5 7.6 7.7 7.8 7.9 8.0 8.1 8.2

LEV-1104-F2 - Methanol-D4 - HSQC



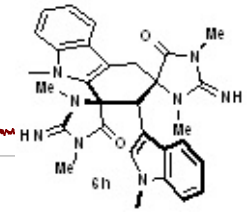
{2.86,26.45}

{2.76,26.45}

f2 (ppm)

f1 (ppm)

LEV-1104-F2 - Methanol-D4 - HMBC



{3.87,116.01}

{2.86,158.16}

{2.76,161.53}

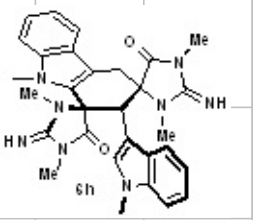
{2.76,174.01}

{2.86,172.52}

f2 (ppm)

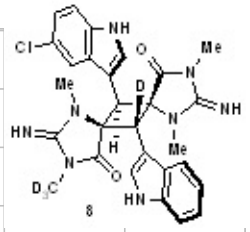
f1 (ppm)

LEV-1104-F2 - Methanol-D4



173.80
172.31
161.87
158.97
140.54
137.37
129.31
128.68
127.64
126.19
124.23
123.57
121.92
121.78
120.27
118.29
116.01
111.17
111.11
102.97
72.36
72.19
45.87
33.23
32.89
30.26
28.31
28.01
26.67
26.52

SO/BYM-583-F2 - DMSO-D6



7.40 7.39 7.37 7.36 7.27 7.27 7.26 7.24 7.24 7.08 7.08 7.07 7.07 7.06 7.06 6.94 6.93 6.92 6.90 6.89 6.82 6.81

5.17 5.16

3.20 3.14 3.00

D (d) 7.24
H (d) 6.81
B (d) 7.37
F (t) 6.93
A (d) 7.39
E (td) 7.07
C (m) 7.27
G (d) 6.90
I (d) 5.16
K (s) 3.14
J (s) 3.20
L (s) 3.00

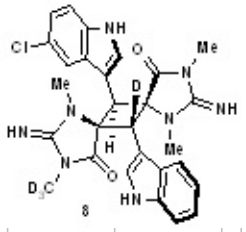
1.00 0.93 1.07 1.06 2.00 0.96 0.94 0.84 1.05 3.62 3.46 3.43

.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.

f1 (ppm)

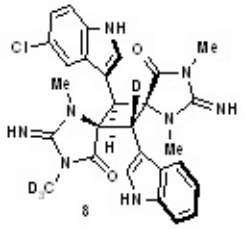
SO/BYM-583-F2 - DMSO-D6

135.44
134.01
127.91
126.91
124.58
123.59
123.01
121.49
121.44
118.97
116.80
116.31
113.23
111.66
106.05
105.86
45.84
28.14
28.04
25.42



f1 (ppm)

SO/BYM-583-F2 - DMSO-D6 - COSY



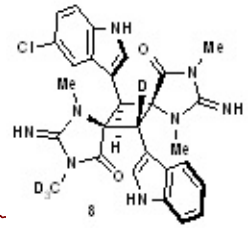
f1 (ppm)

6.7
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6

7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65

f2 (ppm)

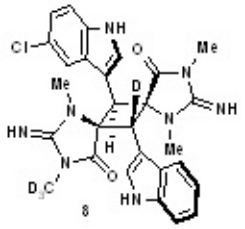
SO/BYM-583-F2 - DMSO-D6- HSQC



f2 (ppm)

f1 (ppm)

SO/BYM-583-F2 - DMSO-D6 - HMBC



{5.17,66.61}
{3.20,66.61}
{3.14,66.61}
{3.20,153.84}
{3.14,153.84}
{3.00,153.84}
{5.17,173.89}
{3.00,173.89}

f2 (ppm)
f1 (ppm)