Supporting Information

UV Resonance Raman Spectroscopy of the Supramolecular Ligand Guanidiniocarbonyl Indole (GCI) with 244 nm Laser Excitation

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Table S1 contains the theoretical vibrational spectrum of GCI ethyl amide in the single protonated form calculated at the B3LYP/6-311++G(d,p) level of theory employing the GAUSSIAN 2016 program package.[1] All normal modes together with their wavenumber values and Raman activities are listed.

Table S1: Calculated Raman spectrum of GCI ethyl amide in the single protonated form. Level of theory: B3LYP/6-311++G(d,p)

Mode	Wavenumber [cm^{-1}]	Raman activity
1	19.37	1.614
2	37.13	2.241
3	41.89	1.999
4	46.39	3.739
5	58.38	1.407
6	74.66	0.869
7	87.27	1.491
8	120.26	0.145
9	140.34	2.586
10	169.17	1.154
11	199.20	0.631
12	218.14	4.363

13	250.76	5.217
14	254.79	0.466
15	273.92	2.986
16	274.65	0.586
17	294.32	2.158
18	314.39	6.605
19	327.04	0.665
20	358.66	1.124
21	384.27	2.129
22	403.84	4.364
23	421.13	2.702
24	457.32	2.466
25	466.73	1.390
26	479.93	18.653
27	484.04	1.820
28	497.66	6.060
29	529.93	1.540
30	584.65	2.083
31	589.01	3.993
32	602.63	8.045
33	612.01	4.785
34	665.82	29.282
35	689.26	5.874
36	706.25	8.948
37	723.41	1.610
38	740.13	0.552
39	746.61	2.356
40	771.56	12.877
41	779.14	7.745
42	817.31	24.989
43	818.26	3.556
44	845.48	7.383
45	854.38	3.362
46	866.21	1.201
47	883.26	5.970
48	897.13	44.256
49	930.16	16.609
50	962.22	1.012
51	968.66	22.423
52	1011.33	29.625
53	1033.01	98.198
54	1045.78	17.852
55	1075.07	9.424
56	1079.29	21.142
57	1101.77	75.099
58	1114.88	74.764

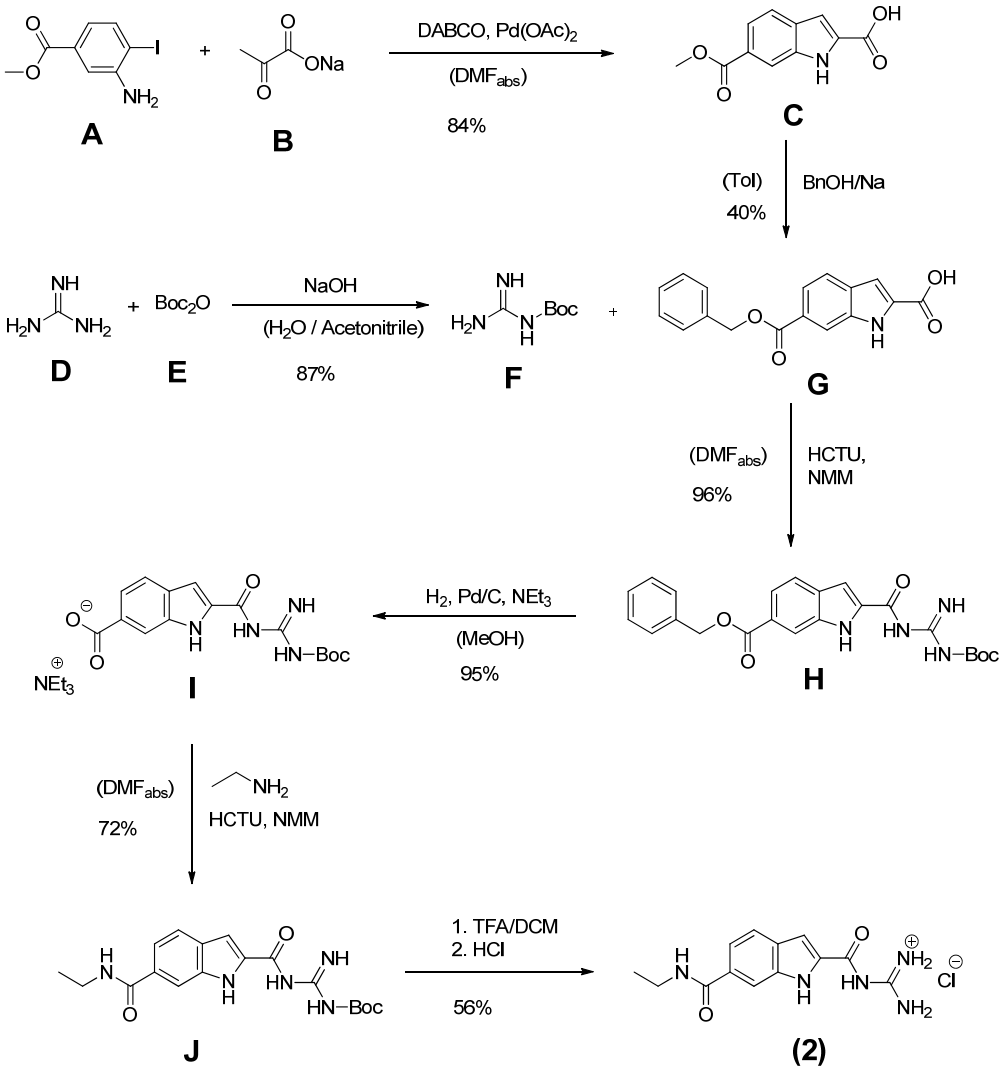
59	1122.24	4.698
60	1139.22	4.847
61	1182.98	10.770
62	1209.61	1.692
63	1227.57	45.250
64	1252.51	39.768
65	1272.77	467.632
66	1295.14	382.126
67	1335.60	23.526
68	1339.10	36.044
69	1348.12	19.142
70	1355.69	310.193
71	1369.28	0.408
72	1404.78	72.358
73	1442.05	11.384
74	1444.30	7.283
75	1456.31	53.538
76	1465.92	19.717
77	1467.95	49.036
78	1473.66	68.737
79	1500.97	558.777
80	1541.83	48.222
81	1552.73	4.742
82	1594.83	1217.006
83	1616.47	49.798
84	1648.92	3.871
85	1669.29	139.096
86	1694.70	759.733
87	2914.05	309.332
88	2915.87	80.694
89	2969.97	61.887
90	2996.46	150.032
91	2997.54	45.799
92	3065.37	50.758
93	3074.99	92.052
94	3094.07	149.837
95	3131.67	72.506
96	3264.98	91.400
97	3447.16	32.326
98	3461.32	45.783
99	3464.52	133.812
100	3485.00	65.610
101	3526.35	81.719
102	3571.65	62.884

General information

All solvents were distilled before use. Millipore water was obtained with a TKA MicroPure ultrapure water system. All other commercially available reagents were used as obtained unless otherwise specified. Reactions were monitored by TLC on silica gel plates (Macherey-Nagel POLYGRAM SIL G/UV254). Spots were visualized by UV light (254 nm and 366 nm). Reversed phase column chromatography was performed with an Armen Instrument Spot Flash Liquid Chromatography MPLC apparatus with RediSep C-18 Reversed Phase columns. Lyophilisation was done with a Christ Alpha 1-4 LD plus freeze dryer. The melting points were obtained with a Büchi Melting-Point B-540 apparatus with open end glass capillary tubes. The melting points are not corrected. The IR spectra were measured on a Varian 3100 FT-IR Excalibur Series. The low resolution ESI mass spectra were recorded with a Bruker amaZon SL and the high resolution ESI mass spectra with a Bruker maXis 4G UHR-TOF. Analytical HPLC was performed on a Dionex HPLC apparatus that consisted of a P680 pump, an ASI-100 automated sample injector and an UVD 340U photodiode array detector with a YMC ODS-AQ column (column size: 150 x 3.0 mm, particle size: 5 µm, pore size: 12 nm). The NMR spectra were measured with Bruker DMX 300, AV NEO 400, DRX 500 or AVHD 600 spectrometers. All measurements were recorded at room temperature using DMSO-d_6 as solvent. The chemical shifts are relative to the signals of DMSO-d_6 (δ ^{1}H = 2.50 ppm and δ ^{13}C = 39.5 ppm). The apparent coupling constants are given in Hertz (Hz). The description of the fine structure means: s = singlet, br. s = broad singlet, d = doublet, t = triplet, m = multiplet.

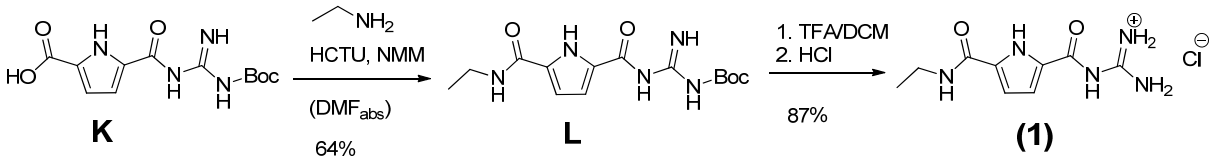
Synthesis

The GCI building block **I** was synthesized starting from commercially available methyl-3-amino-4-iodobenzoate **A** following a synthesis strategy inspired and adjusted from previous work [2]. Building block **I** was further functionalized with ethyl amine to achieve GCI ethyl amide **2.**

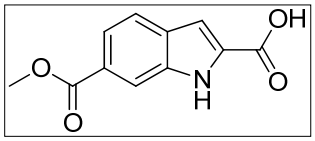


Scheme S1: Synthesis of GCI ethyl amide **2**.

The GCP ethyl amide **1** was synthesized starting from literature known GCP building block **K** [3].



Scheme S2: Synthesis of GCP ethyl amide**1**.**6-(Methoxycarbonyl)-1H-indole-2-carboxylic (C)**



To freshly sublimed DABCO (6.074 g, 54.15 mmol, 3 eq.), sodium pyruvate **B** (5.959 g, 54,15 mmol, 3 eq.) and methyl 3-amino-4-iodobenzoate **A** (5 g, 18.05 mmol, 1 eq.), 260 mL degassed dry DMF was added under argon atmosphere. Palladium (II) acetate (0.33 g, 1.47 mmol, 0.08 eq.) was added and the mixture was heated to 105 °C for 19 h. After cooling to room temperature 150 mL water was added, the solution was acidified with 1 M HCl to pH = 2 and extracted with ethyl acetate (5x150 mL). The combined organic layers were washed with brine (2x150 mL) and water (2x150 mL), dried ($MgSO_4$) and the solvent was evaporated *in vacuo* to obtain a brown solid. The crude product was purified by column chromatography (SiO2, ethyl acetate/cyclohexane = 2/1 + 1% acetic acid) to give **C** (3.321 g, 15.15 mmol, 84%) as a yellow solid.

Molecular Formula: $C_{11}H_9NO_4$.

Molecular Mass: 219.193 g/mol.

^{1}H-NMR (300 MHz, DMSO-d_6): δ [ppm] = 13.14 (s, 1H, COO*H*), 12.17 (s, 1H, N*H*), 8.11 (s, 1H, *H*-7), 7.75 (d, *J* = 8.5 Hz, 1H, *H*-5), 7.65 (dd, *J* = 8.5, 1.5 Hz, 1H, *H*-4), 7.16 (dd, *J* = 2.0, 0.8 Hz, 1H, *H*-3), 3.87 (s, 3H, C*H*$_3$).

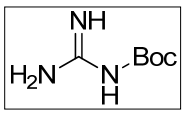
^{13}C-NMR (75 MHz, DMSO-d_6): δ [ppm] =166.79, 162.39, 131.61, 130.19, 125.07, 121.94, 120.94, 120.08, 114.51, 107.03, 51.93.

HR-MS: (pos. ESI, MeOH) m/z = 220.0616 ([M+H]$^+$, calc.: 220.0604).

FT-IR (ATR) ṽ [cm^{-1}] = 3648.66 (s), 2534.01 (w), 2159.88 (w), 2032.6 (w), 1683.55 (m), 1506.13 (m), 1438.64 (s), 1240 (m), 827.312 (s), 771.387 (s), 732.817 (s).

mp: 230 °C (decomposition).

N-^{t}Boc-guanidine (F)



The reaction was performed as described in literature [3]. A solution of ^{t}Boc$_2$O **E** (12.0 g, 55.0 mmol, 1 eq.) in acetonitrile (100 mL) was added very slowly over 8 h at 0 °C under vigorous stirring to a mixture of guanidinium chloride **D** (26.3 g, 275 mmol, 5 eq.) in an aqueous sodium hydroxide solution (12.0 g, 0.3 mol NaOH in 50 mL water). The resulting suspension was stirred at room temperature for additional 20 h. The acetonitrile was evaporated in vacuo and then 100 mL water was added. This aqueous suspension was extracted with ethyl acetate (3 times with 100 mL). The combined organic phases were washed with Brine (3 times with 100 mL), dried ($MgSO_4$) and evaporated *in vacuo*. The resulting white crystals were dried to yield 7.66 g (87%) of analytically pure **F**.

Molecular Formula: $C_6H_{13}N_3O_2$.

Molecular Mass: 159.186 g/mol.

^{1}H-NMR (300 MHz, DMSO-d_6): δ [ppm] = 6.88 (br.s 4H, N*H*), 1.33 (s, 9H, Boc-*H*).

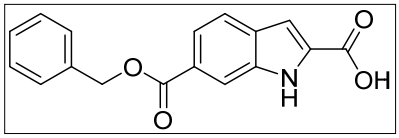
^{13}C-NMR (75 MHz, DMSO-d_6): δ [ppm] = 28.19, 75.49, 162.62, 163.25.

HR-MS: (pos. ESI, MeOH) m/z = 160.1081 ([M+H]$^+$, calc.: 160.1081).

FT-IR (ATR) $\tilde{\nu}$ [cm^{-1}] = 3408 (s), 1650 (s), 1540 (s), 1450 (m), 1311 (s), 1253 (m), 1142 (s), 1066 (s), 950 (w), 806 (m).

mp: 165 °C (decomposition).

6-((Benzyloxy)carbonyl)-1H-indole-2-carboxylic Acid (G)



A sodium benzylate solution, prepared from Sodium (0.603 g, 26.33 mmol, 5 eq.) in dry benzyl alcohol (300 mL), was added to a solution of **C** (1.071 g 4.88 mmol, 1 eq.) in dry benzyl alcohol (48 mL) and dry toluene (42 mL) under Argon. The resulting dark brown solution was stirred at 95 °C for 4 h. After cooling to room temperature 1 M hydrochlorid acid (26 mL) and water (60 mL) was added. The solution was extracted with chloroform (3 x 100 mL) and the solvent evaporated *in vacuo*. The crude product was purified by flash chromatography (RP-18 MeOH/H_2O, 40% MeOH to 100% MeOH, gradient) to give **G** (0.589 g, 2.03 mmol, 40%) as a yellow solid.

Molecular Formula: $C_{17}H_{13}NO_4$.

Molecular Mass: 295.289 g/mol.

^{1}H-NMR (300 MHz, DMSO-d_6): δ [ppm] = 13.23 (s, 1H, COO*H*), 12.13 (s, 1H, N*H*), 8.15 (s, 1H, *H*-7), 7.76 (d, *J* = 8.5 Hz, 1H, *H*-5), 7.68 (dd, *J* = 8.5, 1.4 Hz,1H, *H*-4), 7.52 – 7.33 (m, 5H, Cbz-Ar-*H*), 7.19 – 7.11 (m, 1H, *H*-3), 5.37 (s, 2H, Cbz-CH_2).

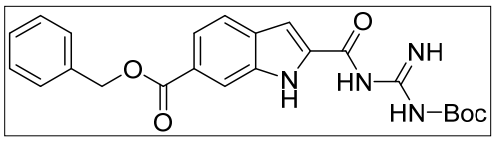
^{13}C-NMR (75 MHz, DMSO-d_6): δ [ppm] = 166.15, 162.33, 136.27, 131.71, 130.31, 128.49, 128.05, 127.95, 124.99, 122.02, 120.14, 114.64, 107.03, 65.99.

HR-MS: (pos. ESI, MeOH) m/z = 296.0920 ([M+H]$^+$, calc.: 296.0917).

FT-IR (ATR) $\tilde{\nu}$ [cm^{-1}] = 3853.08 (s), 3748.94 (s), 3675.66 (s), 3648.66 (s), 3253.32 (w), 2528.22 (w), 2159.88 (w), 2034.53 (w), 1673.91 (m), 1324.86 (m), 1241.93 (m), 1207.22 (m).

mp: 209 °C (decomposition).

Benzyl 2-((N-(tert-butoxycarbonyl)carbamimidoyl)carbamoyl)-1H-indole-6-carboxylate (H)



A mixture of the benzyl ester **G** (0.358 g, 1.256 mmol, 1 eq.), HCTU (1.0392 g, 2.512 mmol, 2 eq.) and NMM (0.5082 g, 5.024 mmol, 4 eq.) was stirred in DMF abs. (15 mL) at room temperature for 15 min. ^{t}Boc-guanidine **F** (0.299 g, 1.884 mmol, 1.5 eq.) was added and the resulting solution stirred at 40 °C. After 19 h the solution was poured into vigorously stirred water (150 mL) at 0 °C. A slightly yellow solid precipitated. The product was filtered, washed with cold water and dried *in vacuo*, yielding **H** as a slightly yellow solid (0.528 g, 1.21 mmol, 96%).

Molecular Formula: $C_{23}H_{24}N_4O_5$.

Molecular Mass: 436.47 g/mol.

^{1}H-NMR (400 MHz, DMSO-d_6): δ [ppm] = 11.82 (s, 1H, N*H*), 10.99 (s, 1H, N*H*), 9.48 (s, 1H, N*H*), 8.63 (s, 1H, N*H*), 8.17 (s, 1H, *H*-7), 7.73 (d, J = 8.5 Hz, 1H, *H*-5), 7.65 (dd, *J* = 8.5, 1.4 Hz, 1H, *H*-4), 7.51 – 7.33 (m, 5H, Cbz-Ar-*H*), 7.18 (s, 1H, *H*-3), 5.36 (s, 2H, Cbz-C*H*$_2$), 1.48 (s, 9H, Boc-C*H*$_3$).

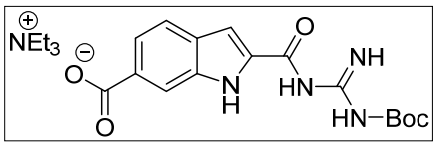
^{13}C-NMR (101 MHz, DMSO-d_6): δ [ppm] = 166.33, 158.64, 136.42, 136.16, 130.72, 128.55, 128.09, 128.00, 121.88, 119.96, 114.62, 105.64, 65.96, 27.73.

HR-MS: (pos. ESI, MeOH) m/z = 437.1815 ([M+H]$^+$, calc.: 437.1819).

FT-IR: (ATR) ṽ [cm-1] = 3853.08 (s), 3748.94 (s), 3675.66 (s), 3648.66 (s), 2524.36 (w), 2159.88 (w), 2030.68 (w), 1700.91 (m), 1617.98 (m), 1558.2 (m), 1540.85 (m), 1496.49 (m), 1191.79 (m), 1145.51 (m), 1085.73 (m).

mp: 270 °C.

GCI building block (I)



A mixture of the benzyl ester **H** (0.528 g, 1.21 mmol, 1 eq.) and 10 % Pd/C (30 mg) in 150 mL methanol and 5 mL triethylamine was vigorously stirred under hydrogen atmosphere for 16 h. The resulting solution was filtered through a folded filter and washed with methanol/triethylamine. The solvent was removed under reduced pressure yielding the GCI building block **I** as an off-white solid (0.516 g, 1.15 mmol, 95%).

Molecular Formula: $C_{22}H_{33}N_5O_5$.

Molecular Mass: 447.528 g/mol.

^{1}H-NMR (400 MHz, DMSO-d_6): δ [ppm] = 11.76 (s, 1H, N*H*), 9.46 (s, 1H, N*H*), 8.63 (s, 1H, N*H*), 8.09 (d, J = 0.8 Hz, 1H, *H*-7), 7.68 – 7.59 (m, 2H, *H*-5, *H*-4), 7.17 (s, 1H, *H*-3), 2.65 (q, J = 7.2 Hz, 6H, NEt_3-C*H*$_2$), 1.48 (s, 9H, Boc-C*H*$_3$), 1.02 (t, J = 7.2 Hz, 9H, NEt_3-C*H*$_3$).

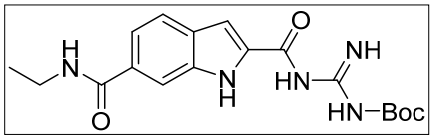
^{13}C-NMR (101 MHz, DMSO-d_6): δ [ppm] = 168.79, 158.59, 136.43, 129.77, 121.21, 120.51, 114.37, 105.70, 81.50, 45.37, 27.75, 10.48.

HR-MS: (pos. ESI, MeOH) m/z = 347.1351 ([M+H]$^+$, calc.: 347.1350).

FT-IR (ATR) ṽ [cm^{-1}] = 3648.66 (s), 2979.48 (w), 2159.88 (w), 1718.26 (s), 1540.85 (m), 1496.49 (m), 1365.35 (m), 1321 (w), 1238.08 (w), 1147.44 (w), 790.671 (s), 746.317 (s).

mp: 138 °C.

Boc-GCI ethyl amide (J)



The GCI building block **I** (79 mg, 0.223 mmol, 1 eq.) and HCTU (184.2 mg, 0.446 mmol, 2 eq.) was solved in DMF abs. (10 mL) and NMM (90.23 mg, 0.892 mmol, 4 eq.) was added. After stirring the solution for 20 min at room temperature ethylamine (15 mg, 0.335 mmol, 1.5 eq.) was added and the resulting solution was stirred at room temperature for 17 h. The solvent was removed with reduced pressure and the crude product was purified by flash chromatography (RP-18 MeOH/H2O, 20% MeOH to 100% MeOH, gradient) to give **J** (60.1 mg, 0.161 mmol, 72%) as a white solid.

Molecular Formula: $C_{18}H_{23}N_5O_4$.

Molecular Mass: 373.406 g/mol.

^{1}H-NMR (300 MHz, DMSO-d_6): δ [ppm] = 11.73 (s, 1H, N*H*), 10.96 (s, 1H, N*H*), 9.43 (s, 1H, N*H*), 8.61 (s, 1H, N*H*), 8.41 (t, *J* = 5.5 Hz, 1H, *H*-7), 7.95 (s, 1H, *H*-3), 7.65 (d, *J* = 8.4 Hz, 1H, *H*-5), 7.52 (dd, J = 8.5 Hz, 1H, *H*-4), 7.17 (s, 1H, N*H*), 3.35 – 3.24 (m, 2H, C*H*$_2$), 1.47 (s, 9H, Boc-C*H*$_3$), 1.13 (t, *J* = 7.2 Hz, 3H, C*H*$_3$).

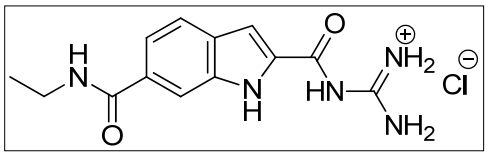
^{13}C-NMR (300 MHz, DMSO-d_6): δ [ppm]= 159.58, 158.79, 155.42, 132.97, 125.22, 115.90, 112.17, 33.62, 14.58.

HR-MS: (pos. ESI, MeOH) m/z = 374.1826 ([M+H]$^+$, calc.: 374.1823).

FT-IR (ATR) ṽ [cm-1]: 3357.46 (w), 2979.48 (w), 1725.98 (w), 1625.7 (m), 1546.63 (m), 1511.92 (m), 1369.21 (w), 1326.79 (m), 1241.93 (s), 1147.44 (s), 844.669 (m), 752.102 (w), 632.537 (m), 609.396 (m).

mp: 124 °C (decomposition).

GCl ethyl amide (2)



To a solution of Boc-GCl ethyl amide **J** (221 mg, 0.592 mmol, 1 eq.) in 15 mL DCM 15 mL TFA was added and the solution was stirred at room temperature for 3 h. The solvent was evaporated in vacuo to receive the off-white crude product, which was purified by flash chromatography (RP 18 MeOH/H2O + 0.1% TFA, 10% MeOH + 0.1% TFA to 100% MeOH + 0.1% TFA, gradient) and treated several times with 1 M HCl with respective solvent removal to give the Chloride salt **2** (103 mg, 0.333 mmol, 56%) as a white solid with a purity of 97% (HPLC).

Molecular Formular: $C_{13}H_{16}ClN_5O_2$.

Molecular Mass: 309.751 g/mol.

^{1}H-NMR (600 MHz, DMSO-d_6): δ [ppm] = 12.36 (m, 2H, indole-N*H*, amide-N*H*), 8.76 (s, 2H, guanidine-N*H*), 8.54 (m, 3H, guanidine-N*H*), 7.99 (s, 1H, *H*-7), 7.95 (d,1H, *J* = 1.4 Hz, *H*-3), 7.76 (d, 1H, *J* = 8.5 Hz, *H*-5), 7.59 (dd, *J* = 8.5, 1.3 Hz, 1H, *H*-4), 3.33 – 3.27 (m, 2H, C*H*$_2$), 1.13 (t, 3H, *J* = 7.2 Hz, C*H*$_3$).

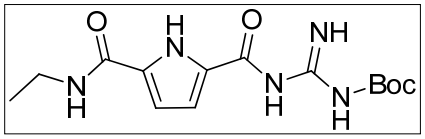
^{13}C-NMR (151 MHz, DMSO-d_6): δ [ppm] = 166.32, 161.06, 155.46, 137.37, 132.07, 130.24, 128.25, 122.17, 119.40, 112.44, 108.00, 34.14, 14.87.

HR-MS: (pos. ESI, MeOH) m/z = 274.1318 ([M+H]$^+$, calc.: 274.1299)

FT-IR (ATR) ṽ [cm^{-1}] = 3097.12 (w), 2159.88 (w), 2032.6 (w), 1683.55 (m), 1635.34 (m), 1606.41 (m), 1560.13 (m), 1498.42 (s), 1448.28 (s), 1413.57 (s), 1375 (s), 1322.93 (m), 1216.86 (w), 1081.87 (s), 836.955 (s), 727.032 (m), 632.537 (w).

mp: 189 °C.

Boc-GCP-ethyl amide (L)



GCP building block **K** (5 g, 12.6 mmol, 1 eq.), HCTU (10.42 g, 25.2 mmol, 2 eq.) and NMM (5.10 g, 50.4 mmol, 4 eq.) were dissolved in dry DMF (200 mL). After 15 min ethylamine [2M in THF] (9.5 mL, 18.9 mmol, 1.5 eq.) was added and the reaction mixture was stirred at room temperature for 16 h, extracted with chloroform (5 x 100 mL) and dried in vacuo. The crude product was purified by flash chromatography (RP-18 MeOH/H2O, 10% MeOH to 100% MeOH, gradient) to give **L** (2.62 g, 8.11 mmol, 64%) as a off-white solid.

Molecular Formula: $C_{14}H_{21}N_5O_4$.

Molecular Mass: 323.348 g/mol.

^{1}H-NMR (300 MHz, DMSO-d_6): δ [ppm] = 11.24 (s, 1H, N*H*), 10.85 (s, 1H, N*H*), 9.32 (s, 1H, N*H*), 8.57 (s, 1H, N*H*), 8.33 (s, 1H, N*H*), 6.81 (s, 1H, pyrrole-C*H*), 6.75 (d, J = 3.8 Hz, 1H, pyrrole-C*H*), 3.25 (q, *J* = 7.1, 5.6 Hz, 2H, CH_2), 1.45 (s, 9H, Boc-CH_3), 1.11 (t, *J* = 7.2 Hz, 3H, CH_3).

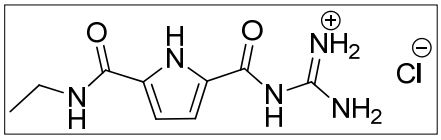
^{13}C-NMR (151 MHz, DMSO-d_6): δ [ppm] = 159.37, 158.43, 111.50, 48.60, 33.54, 27.77, 14.78.

HR-MS: (pos. ESI, MeOH) m/z = 324.1666 ([M+H]$^+$, calc.: 324.1666).

FT-IR (ATR) ṽ [cm^{-1}]: 3748.94 (s), 3648.66 (s), 3318.89 94. (w), 2977.55 (w), 2159.88 (w), 2030.68 (w), 1718.26 (s), 1621.84 (m), 1540.85 (m), 1455.99 (s), 1367.28 (s), 1286.29 (m), 1238.08 (w), 1141.65 (w), 842.74 (s), 748.245 (m);

mp: 138 °C.

GCP ethyl amide (1)



L (101 mg, 0.308 mmol, 1 eq.) was solved in Dichlormethane (2 mL). TFA (2 mL) was added and the reaction mixture was stirred at room temperature for 3 h. The solvent was removed with reduced pressure and the crude product was treated with 1 M HCl. The solvent was removed to receive **1** as a white solid (90 mg, 0.267 mmol, 87%) with a purity of 95% (HPLC).

Molecular Formula: $C_9H_{14}ClN_5O_2$.

Molecular Mass: 259.693 g/mol.

^{1}H-NMR (600 MHz, DMSO-d_6): δ [ppm] = 12.32 (s, 1H, pyrrole-N*H*), 11.32 (s, 1H, amide-N*H*), 8.55 – 8.12 (m, J = 11.2, 5.1 Hz, 5H, guanidine-N*H*), 7.16 (dd, J = 3.9, 2.3 Hz, 1H, pyrrole-C*H*), 6.85 (dd, J = 3.8, 2.4 Hz, 1H, pyrrole-C*H*), 3.33 – 3.21 (m, 2H, CH_2), 1.12 (t, J = 7.2 Hz, 3H, CH_3).

^{13}C-NMR (151 MHz, DMSO-d_6): δ [ppm] = 158.81, 155.03, 132.89, 125.22, 115.50, 112.09, 33.63, 14.58.

HR-MS: (pos. ESI, MeOH) m/z = 224.1148 ([M+H]$^+$, calc.: 224.1142).

FT-IR: (ATR) ṽ [cm^{-1}] = 3309.21 (w), 2159.88 (s), 1685.48 (w), 1633.41 (m), 1558.2 (m), 1473.35 (s), 1292.07 (s), 1203.36 (s), 1064.51 (s), 804.171 (s), 746.317 (m), 698.105 (m), 609.396 (m).

mp: 234 °C (decomposition).

NMR Spectra

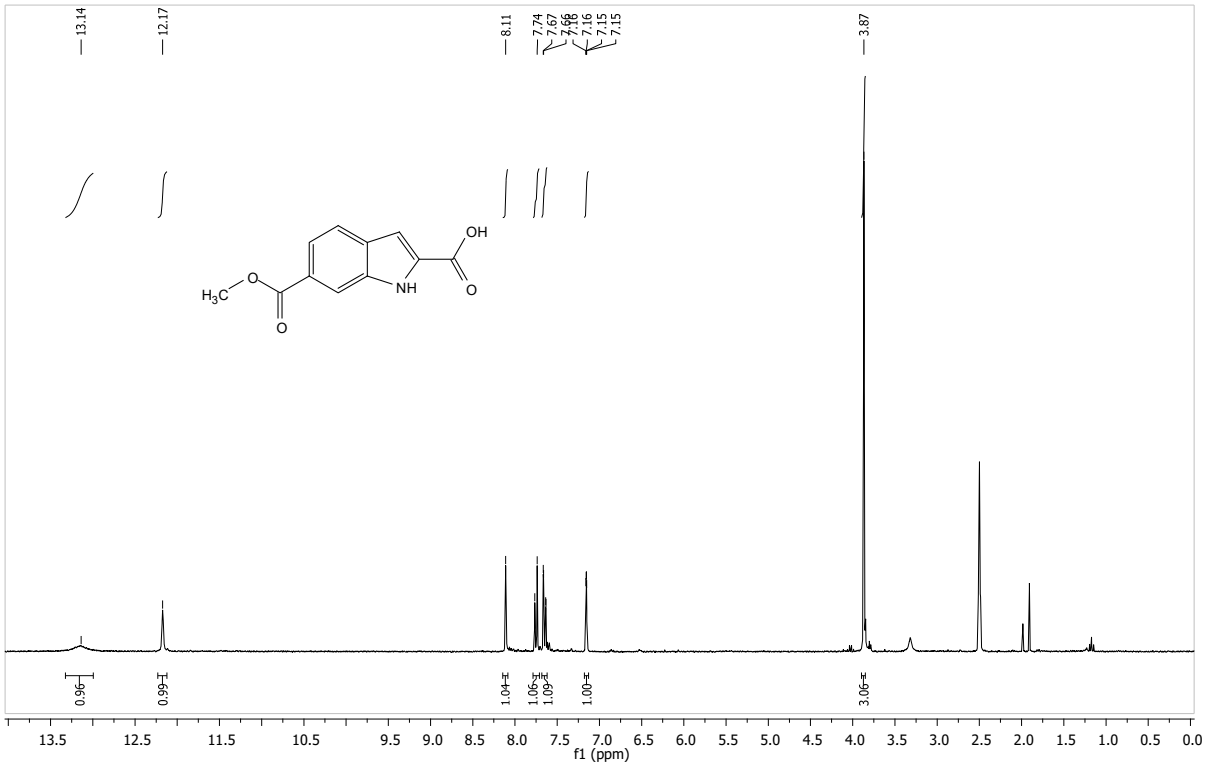


Figure S1: ^{1}H-NMR spectrum of **C** (300 MHz, DMSO-d_6).

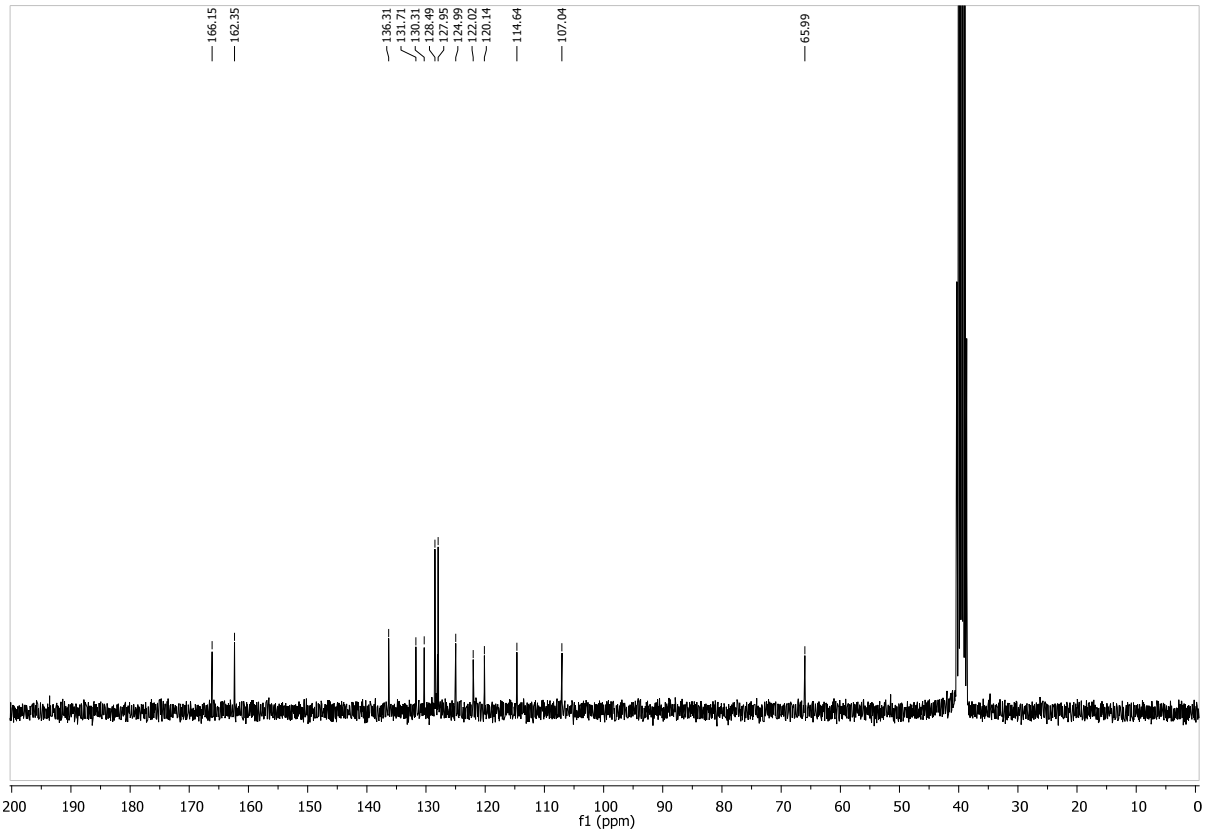


Figure S2: ^{13}C-NMR spectrum of **C** (75 MHz, DMSO-d_6).

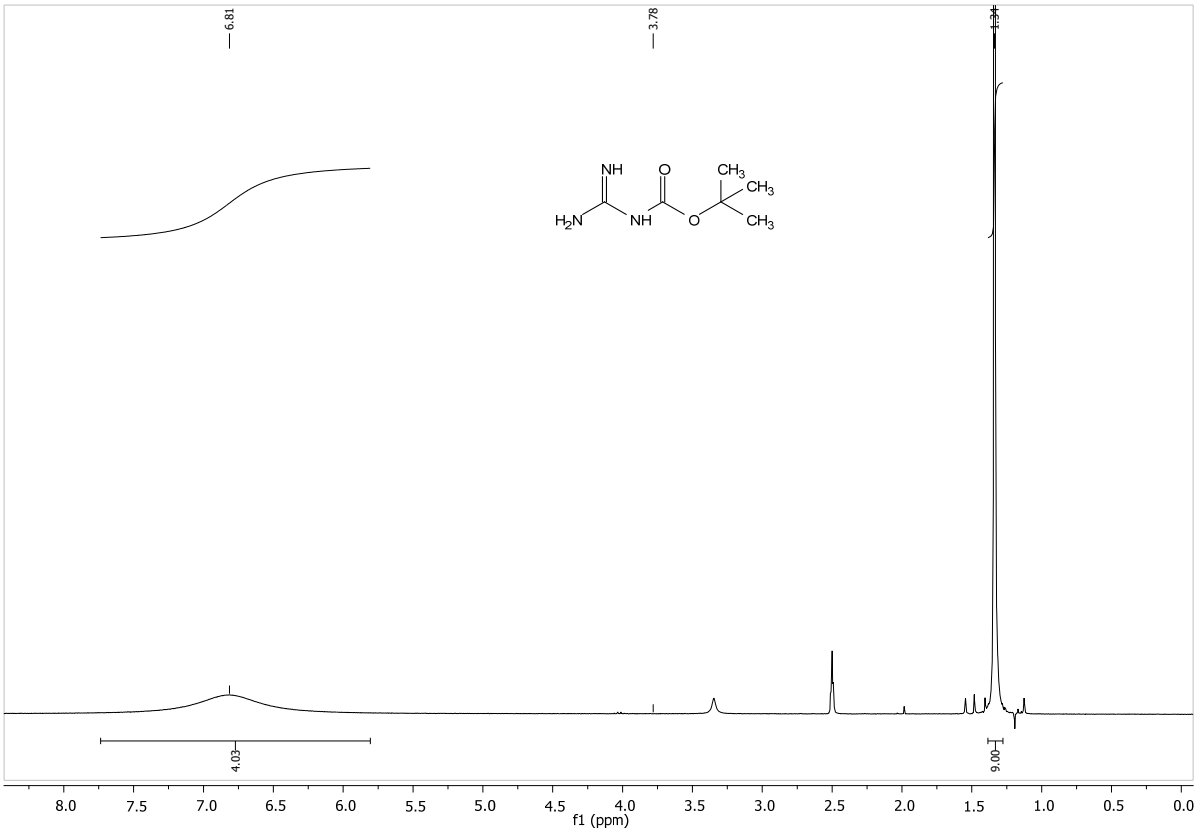


Figure S3: ^{1}H-NMR spectrum of **F** (300 MHz, DMSO-d_6).

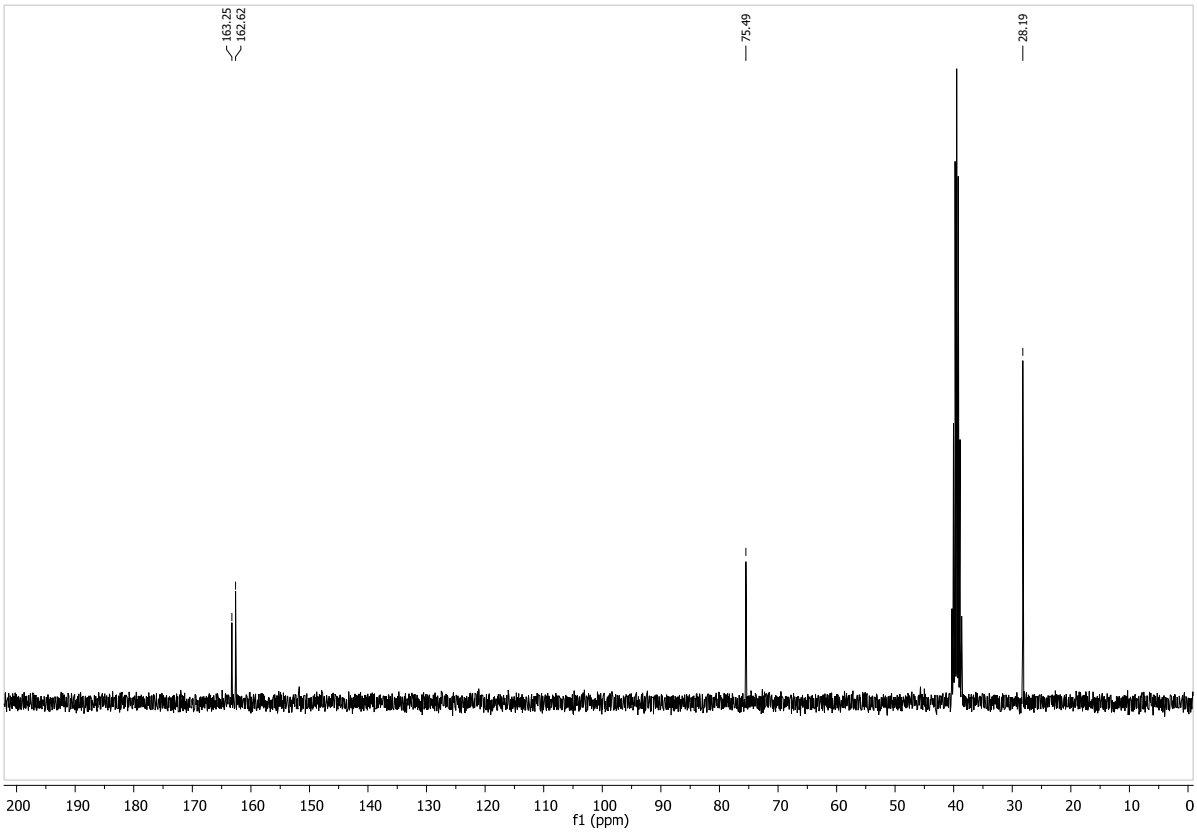


Figure S4: ^{13}C-NMR spectrum of **F** (75 MHz, DMSO-d_6).

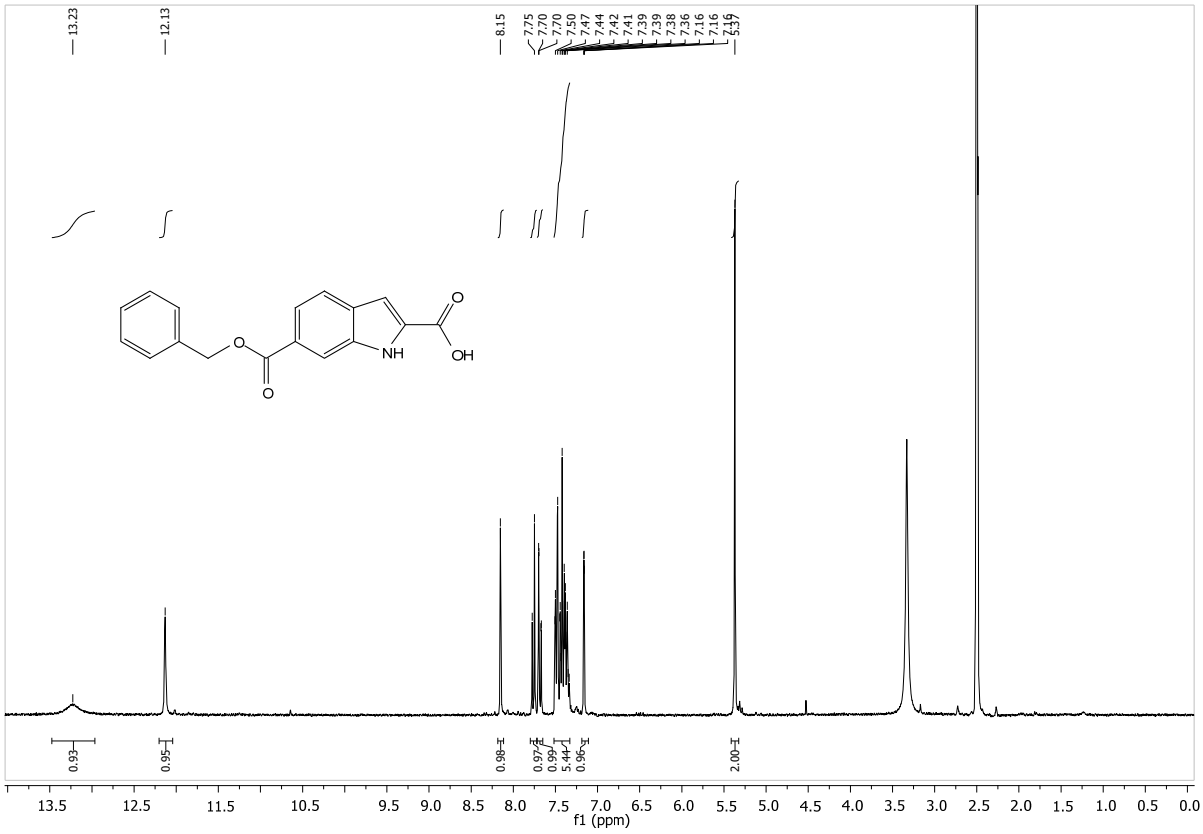


Figure S5: ^{1}H-NMR spectrum of **G** (300 MHz, DMSO-d_6).

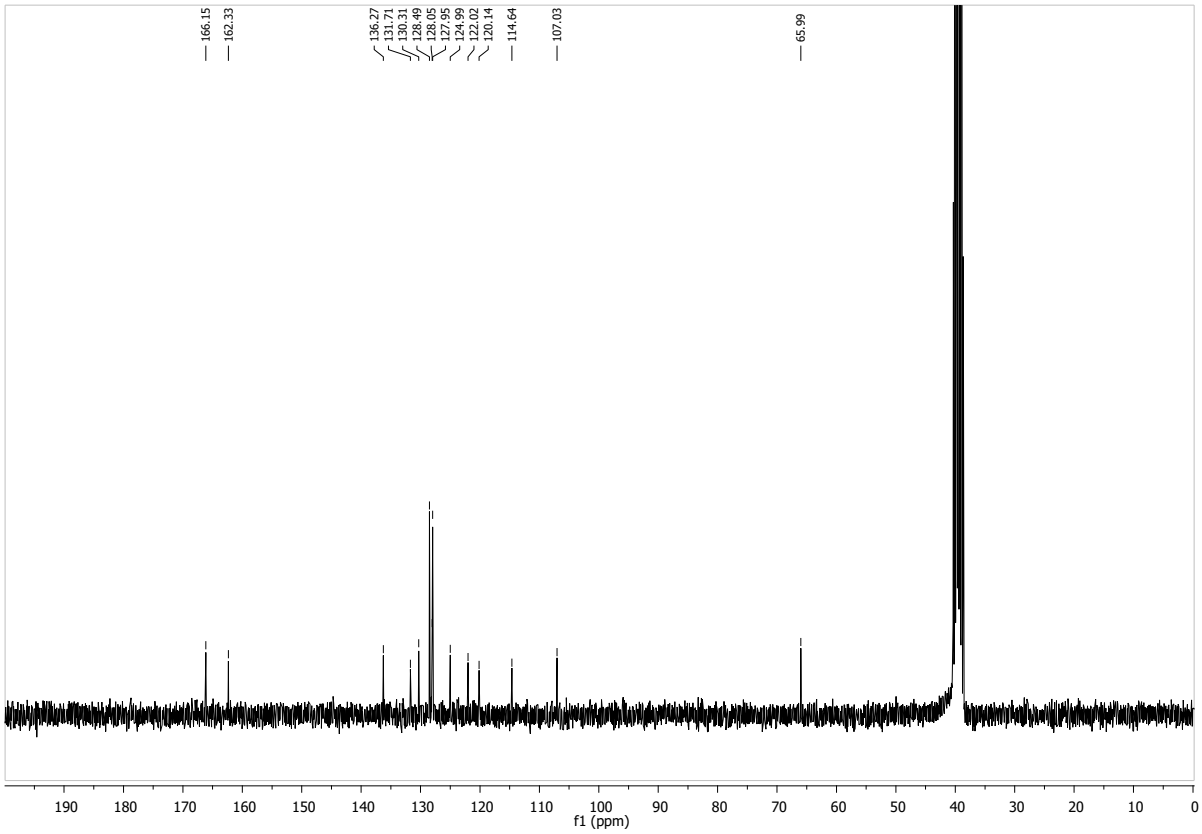


Figure S6: ^{13}C-NMR spectrum of **G** (75 MHz, DMSO-d_6).

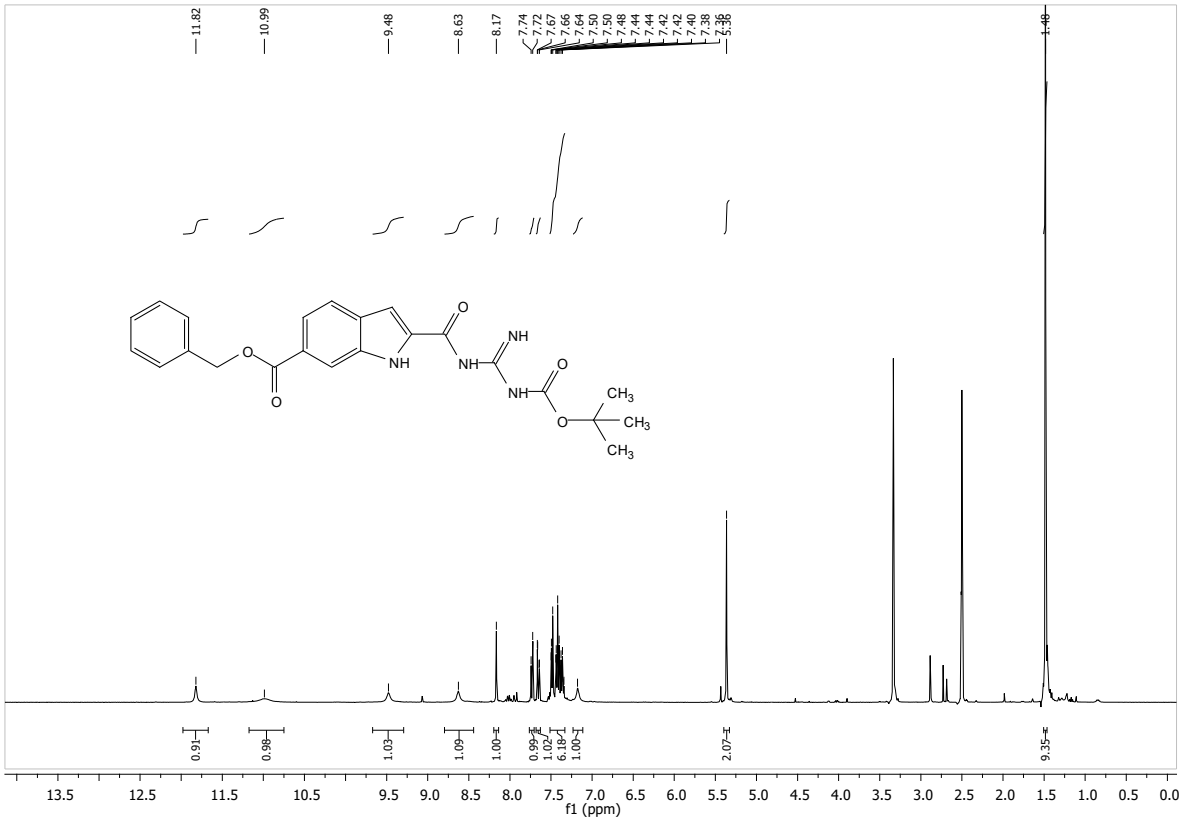


Figure S7: ^{1}H-NMR spectrum of **H** (400 MHz, DMSO-d_6).

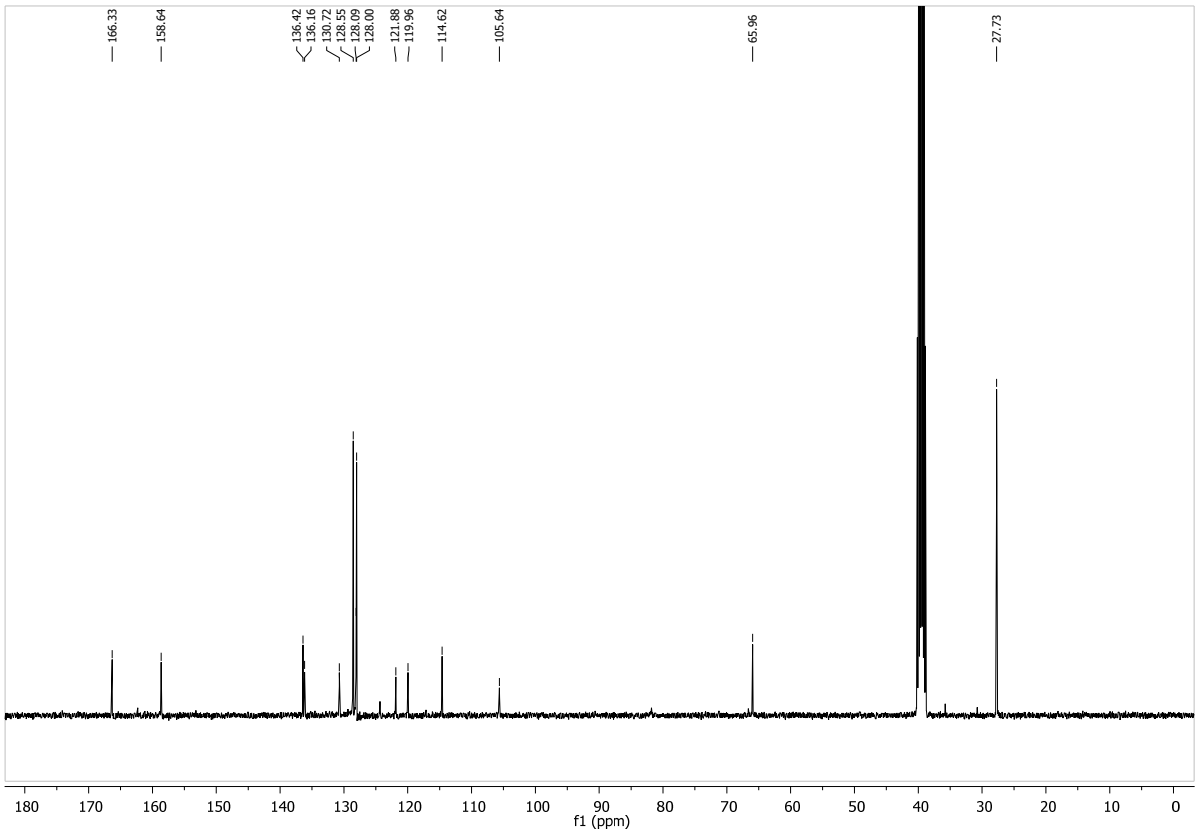


Figure S8: ^{13}C-NMR spectrum of **H** (101 MHz, DMSO-d_6).

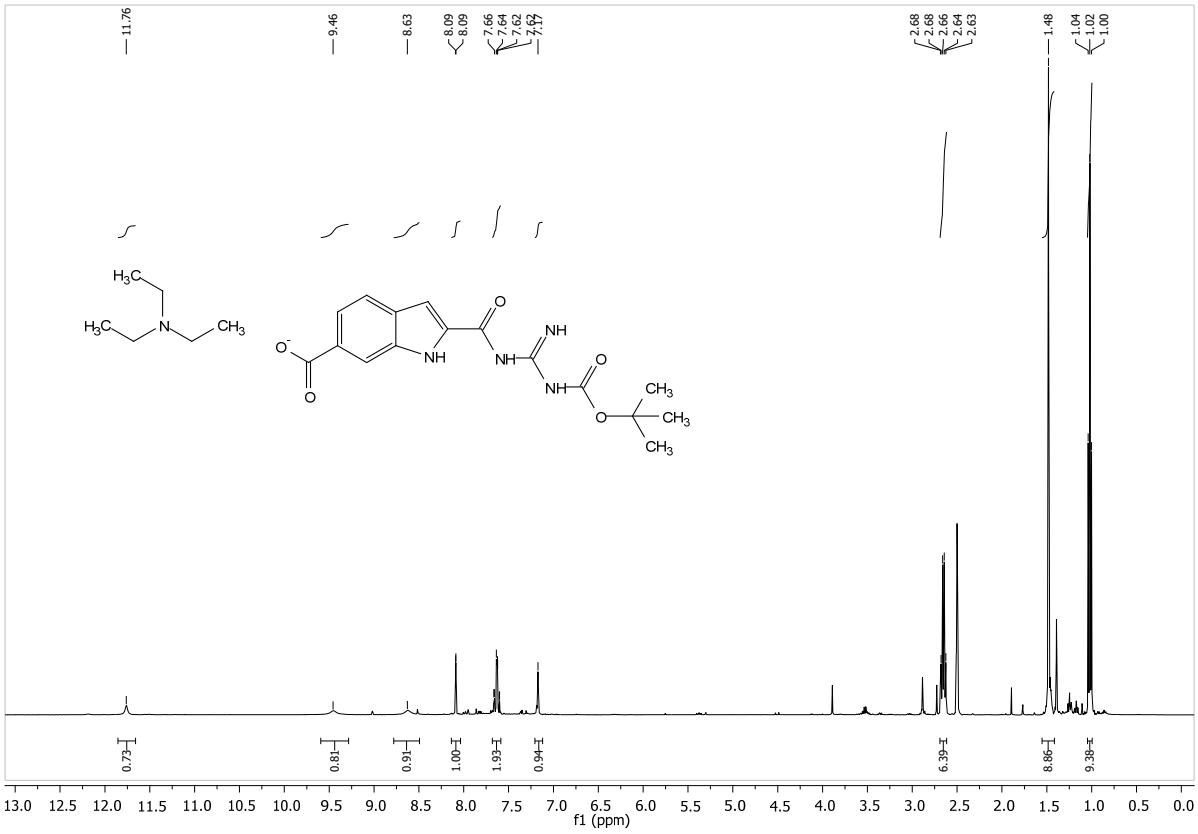


Figure S8: ^{1}H-NMR spectrum of **I** (400 MHz, DMSO-d_6).

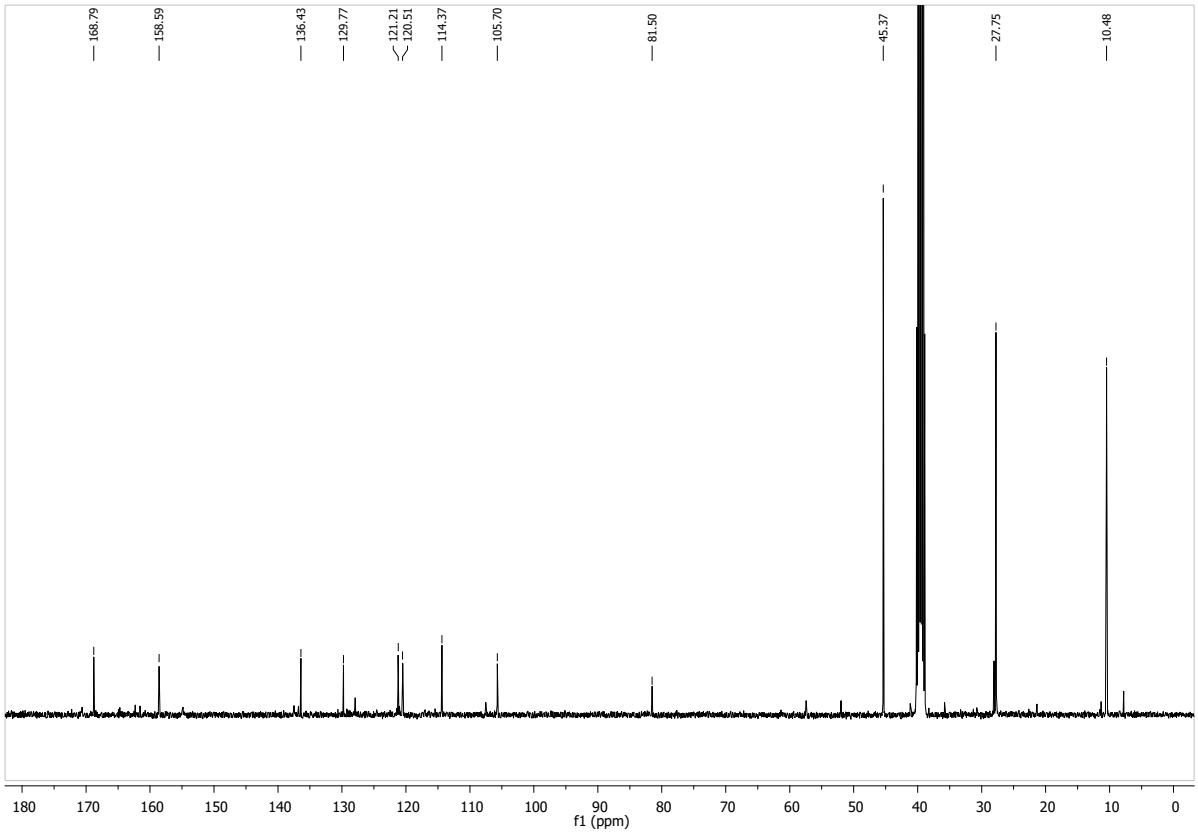


Figure S9: ^{13}C-NMR spectrum of **I** (101 MHz, DMSO-d_6).

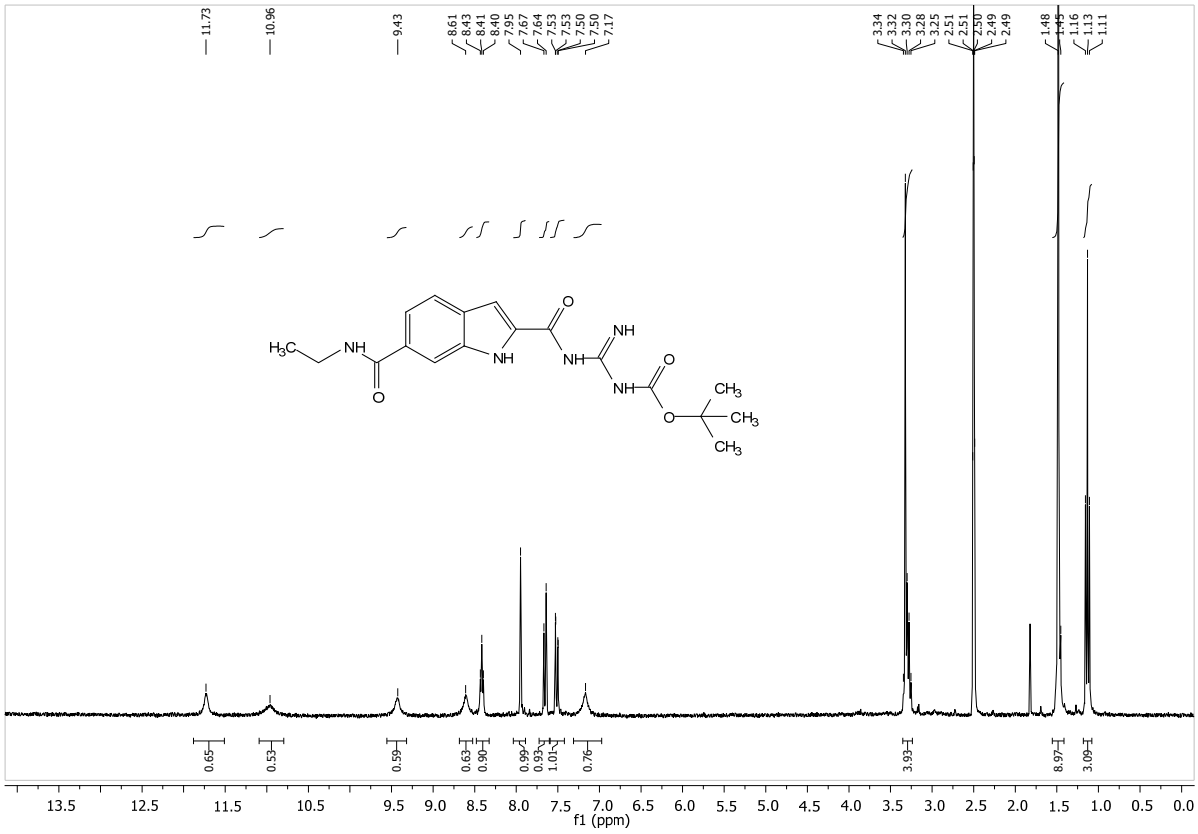


Figure S10: ^{1}H-NMR spectrum of **J** (300 MHz, DMSO-d_6).

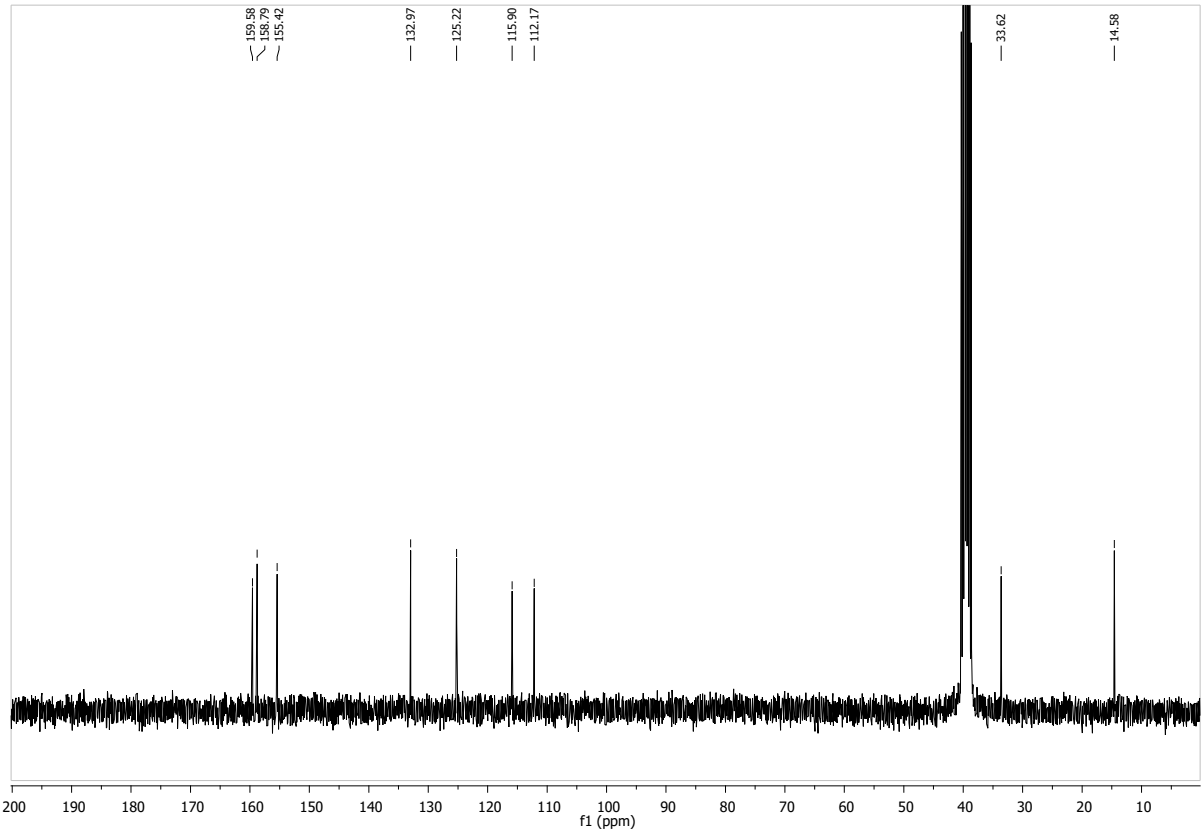


Figure S11: ^{13}C-NMR spectrum of **J** (75 MHz, DMSO-d_6).

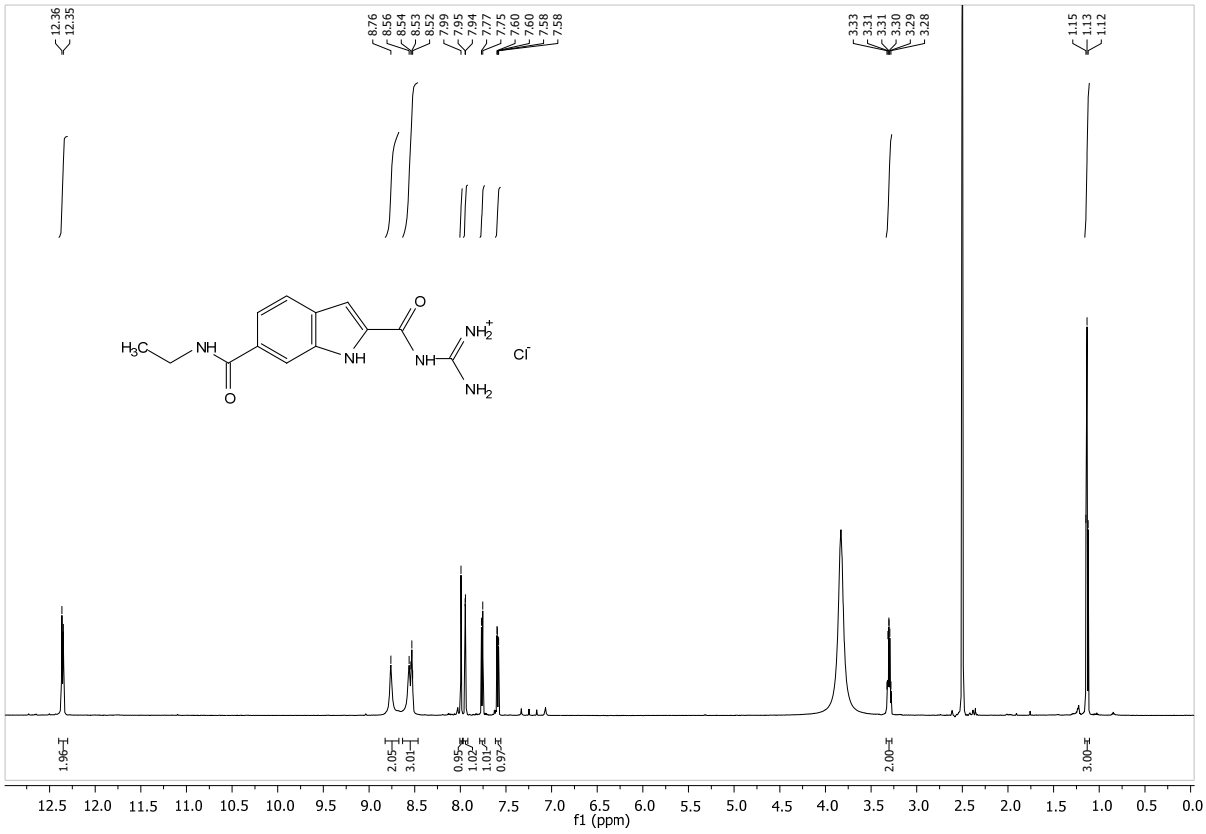


Figure S12: ^{1}H-NMR spectrum of **2** (600 MHz, DMSO-d_6).

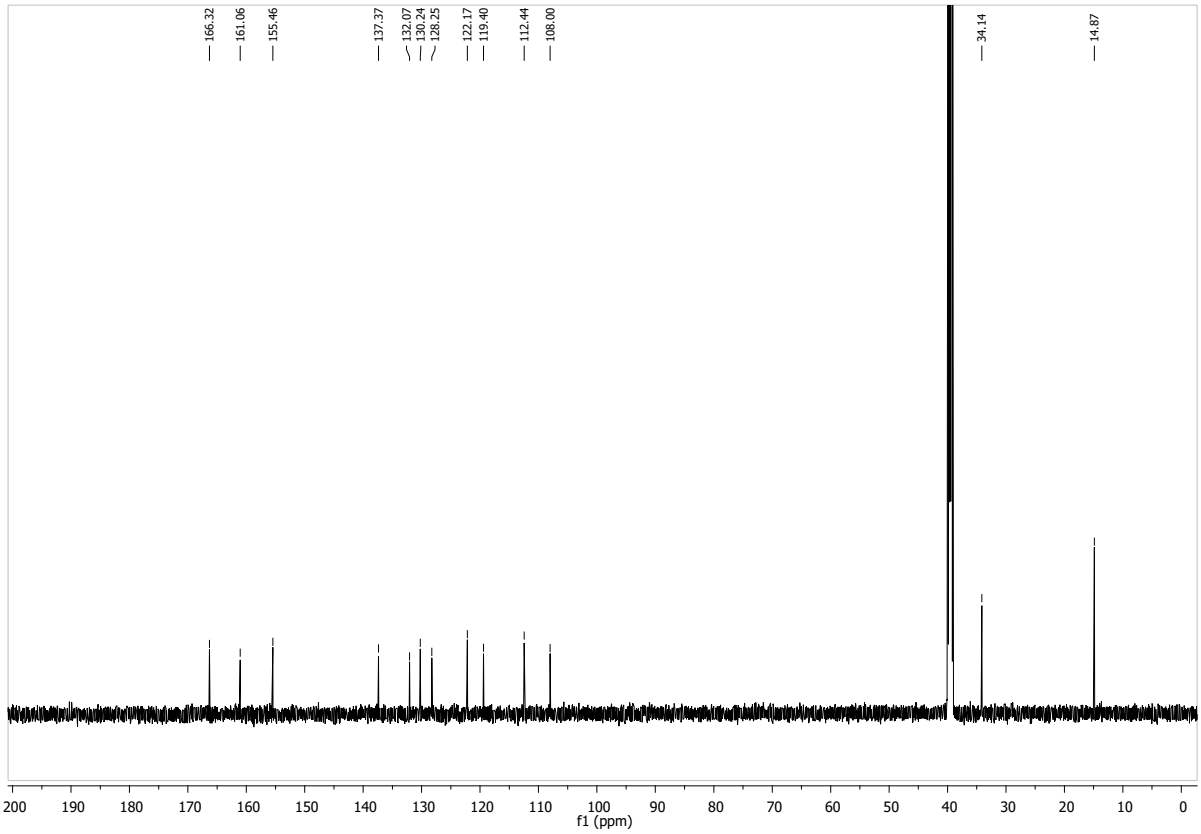


Figure S13: ^{13}C-NMR spectrum of **2** (151 MHz, DMSO-d_6).

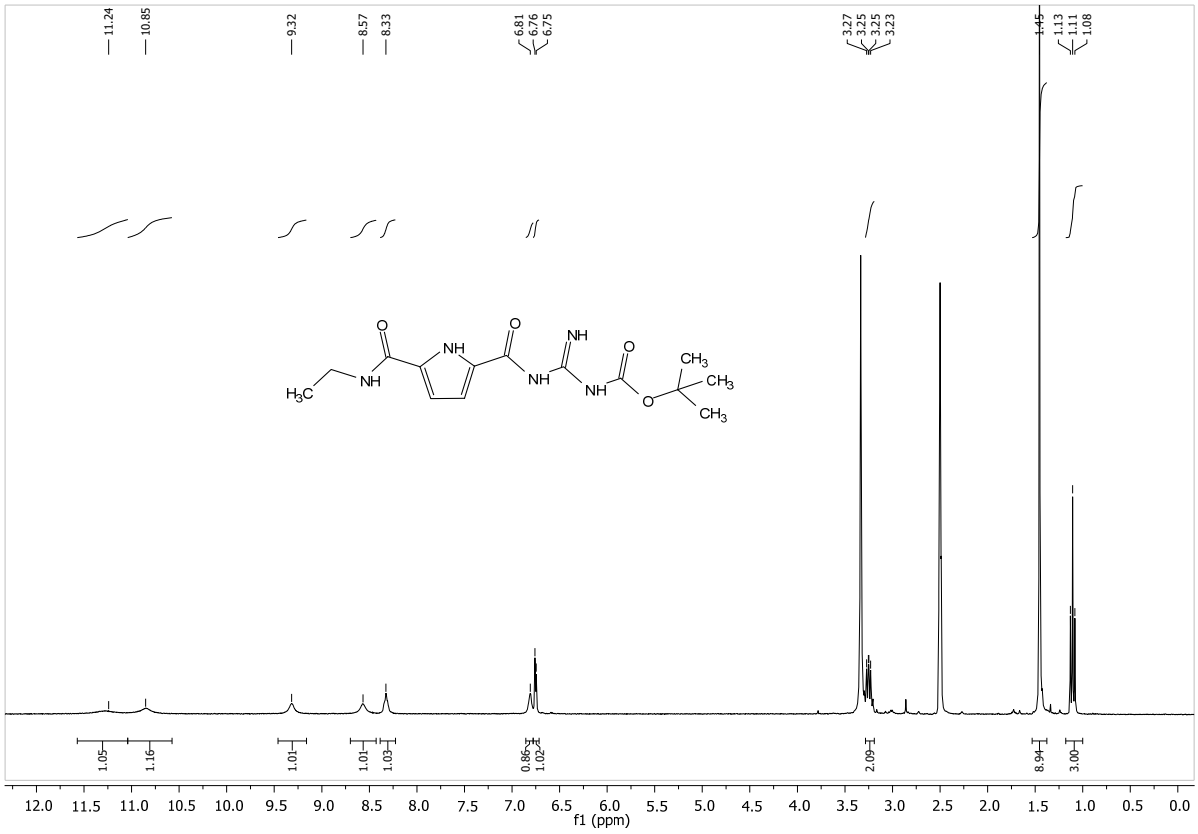


Figure S14: ^{1}H-NMR spectrum of **L** (300 MHz, DMSO-d_6).

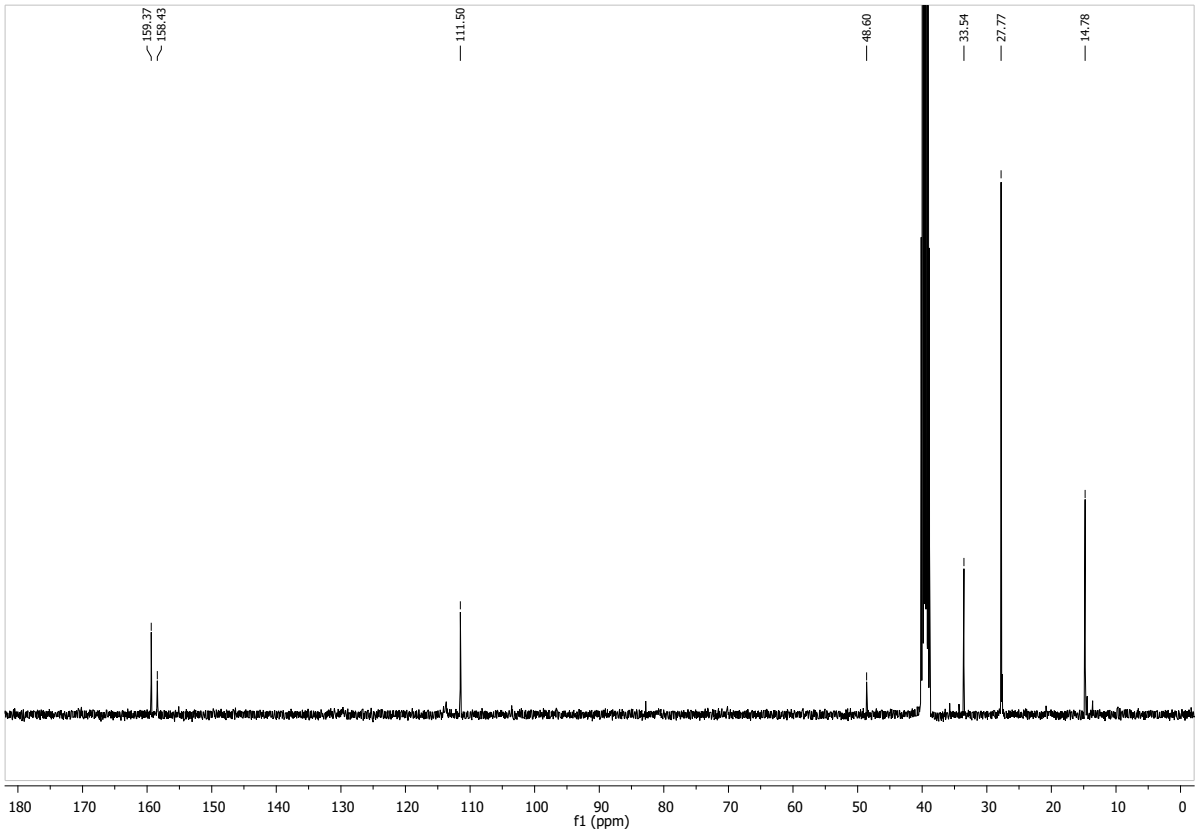


Figure S15: ^{13}C-NMR spectrum of **L** (101 MHz, DMSO-d_6).

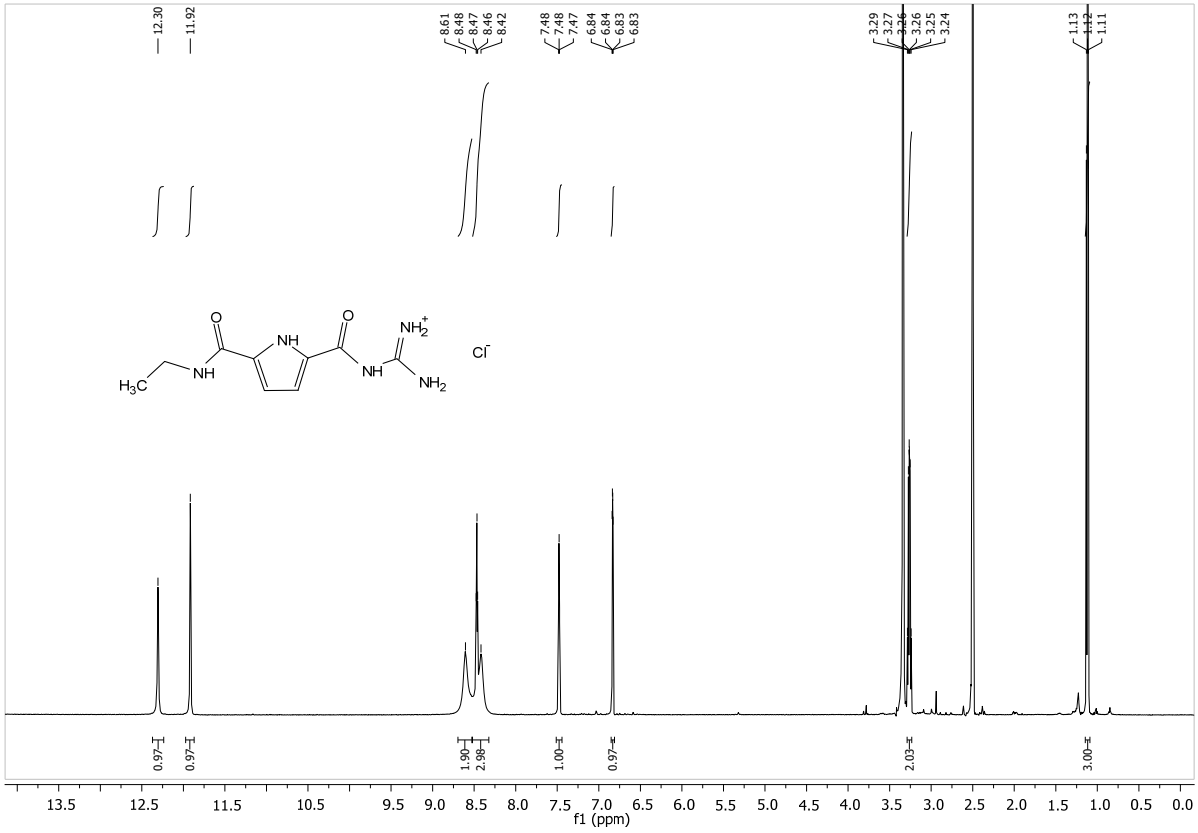


Figure S16: ^{1}H-NMR spectrum of **1** (600 MHz, DMSO-d_6).

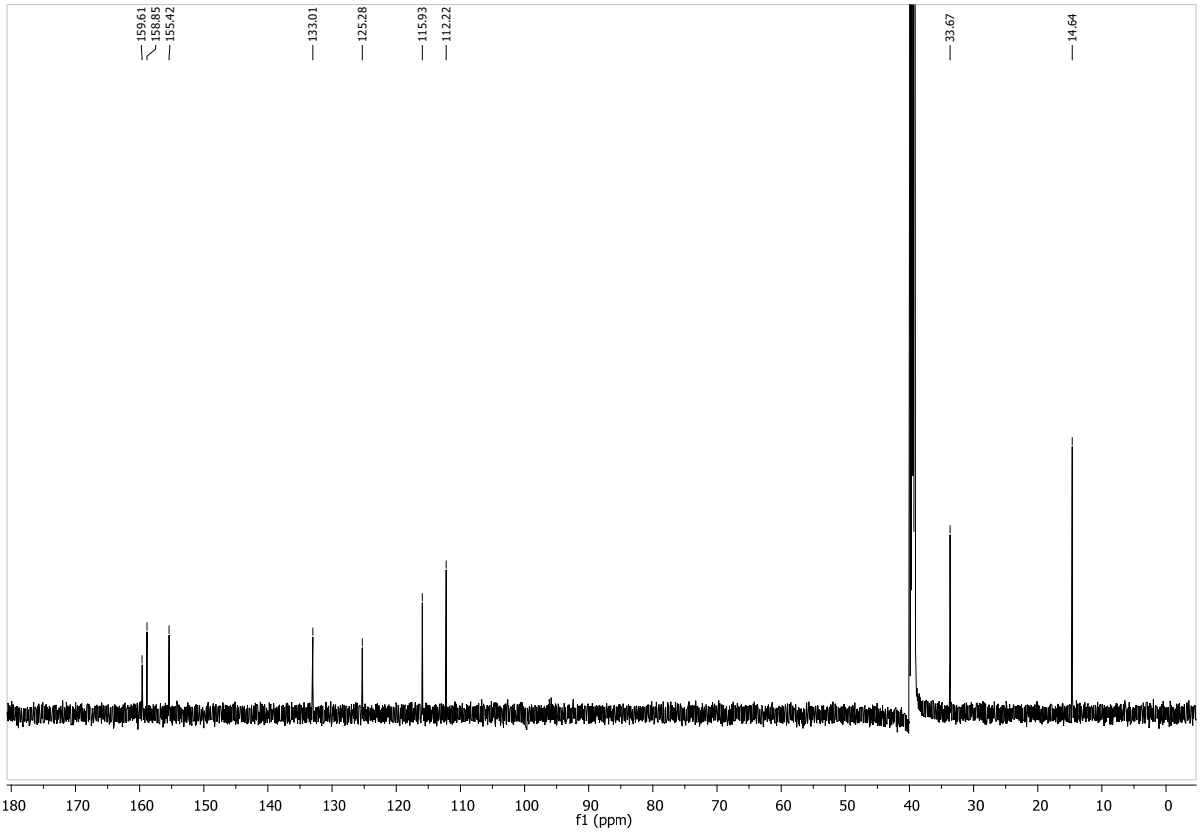


Figure S17: ^{13}C-NMR spectrum of **1** (151 MHz, DMSO-d_6).

Mass Spectra

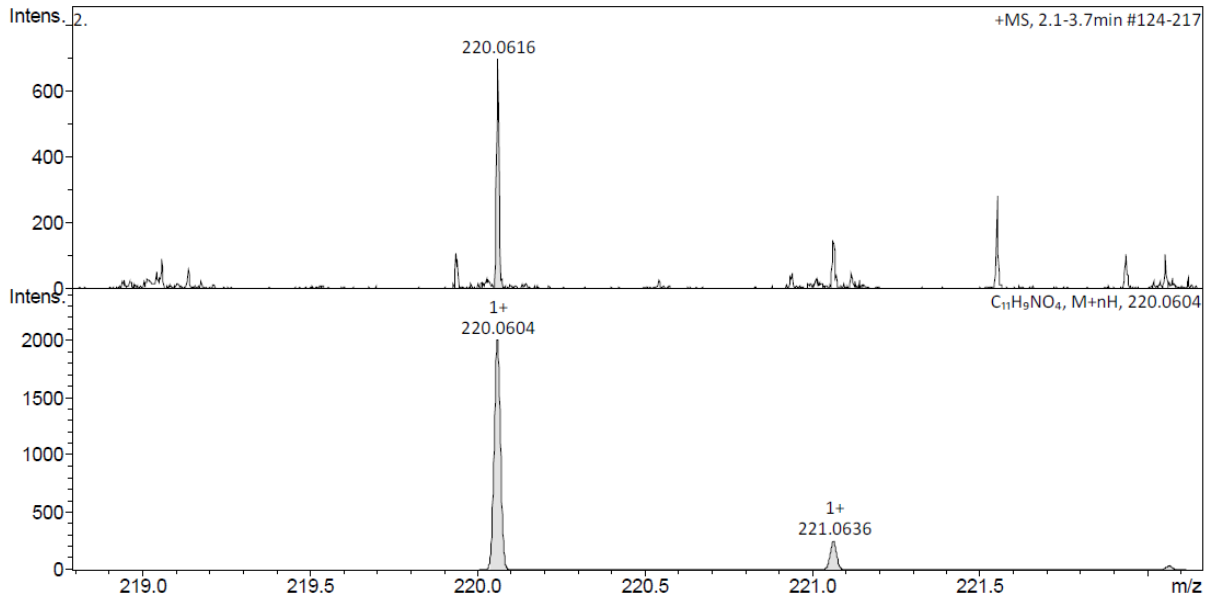


Figure S18: HR-ESI mass spectrum of **C** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **C.**

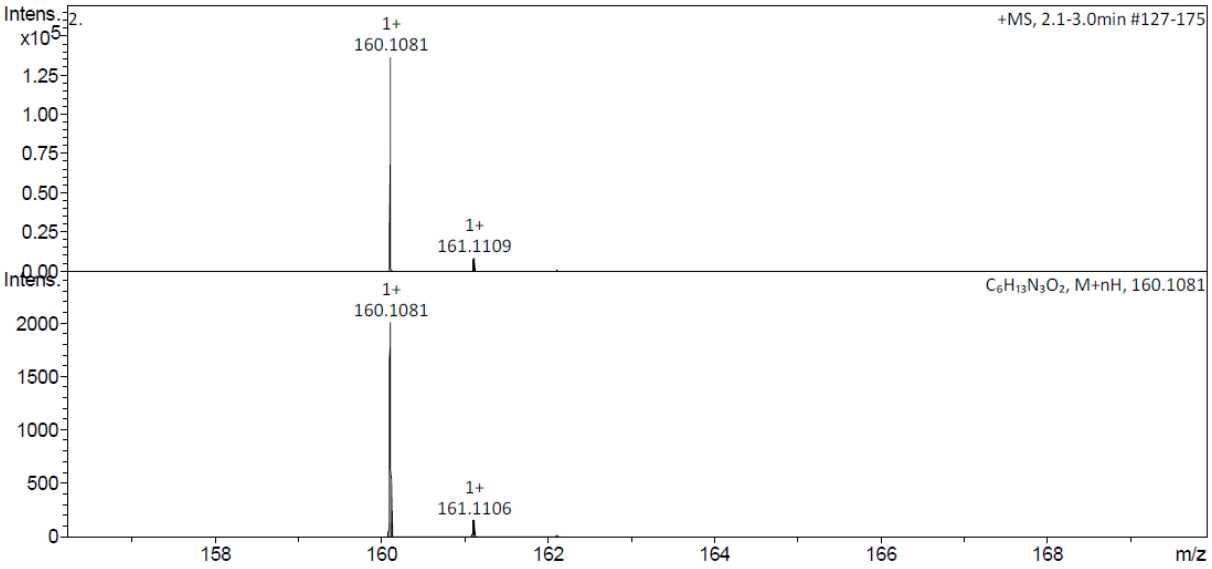


Figure S19: HR-ESI mass spectrum of **F** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **F.**

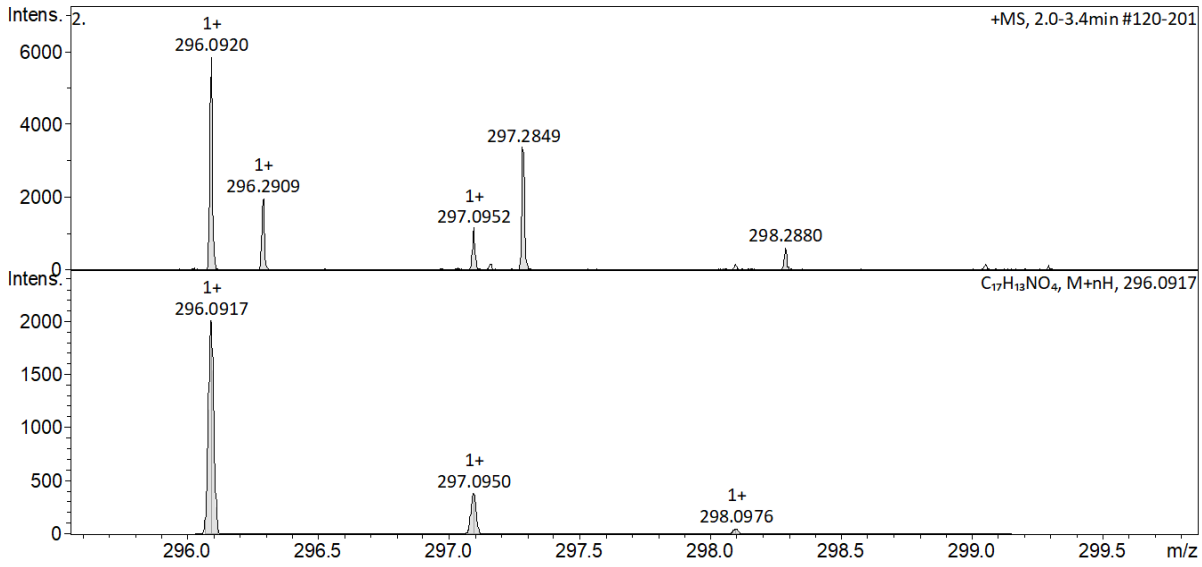


Figure S20: HR-ESI mass spectrum of **G** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **G.**

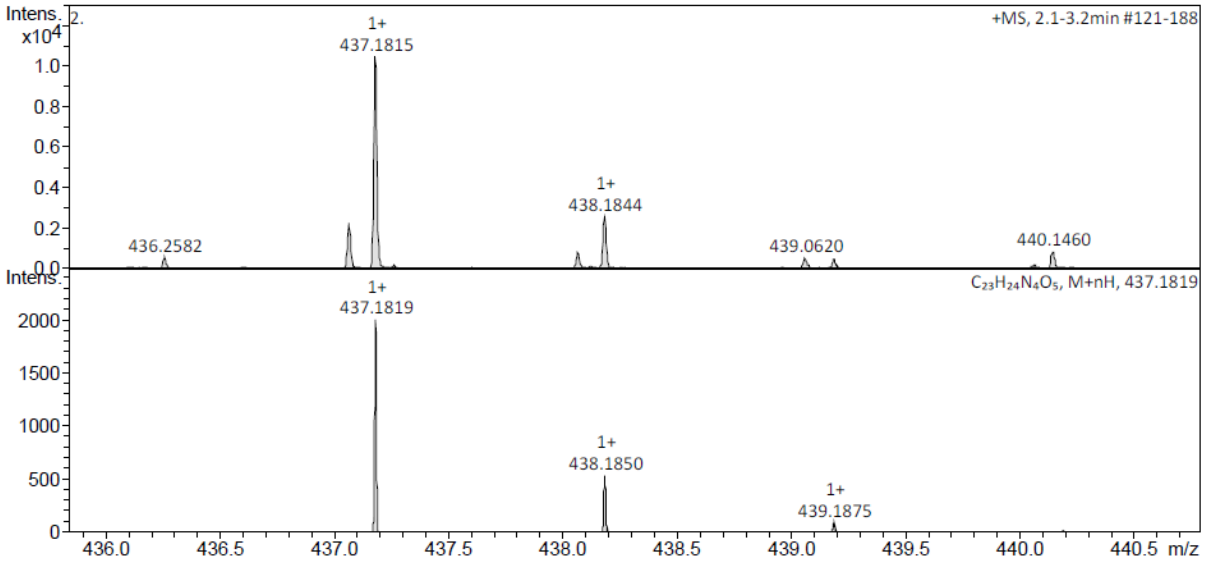


Figure S21: HR-ESI mass spectrum of **H** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **H.**

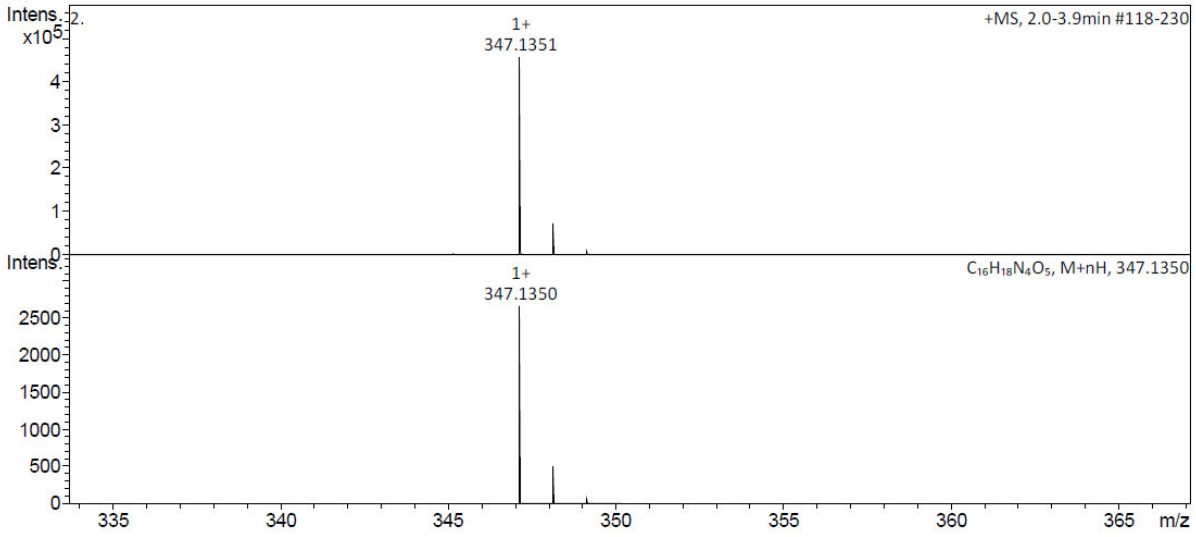


Figure S22: HR-ESI mass spectrum of **I** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **I.**

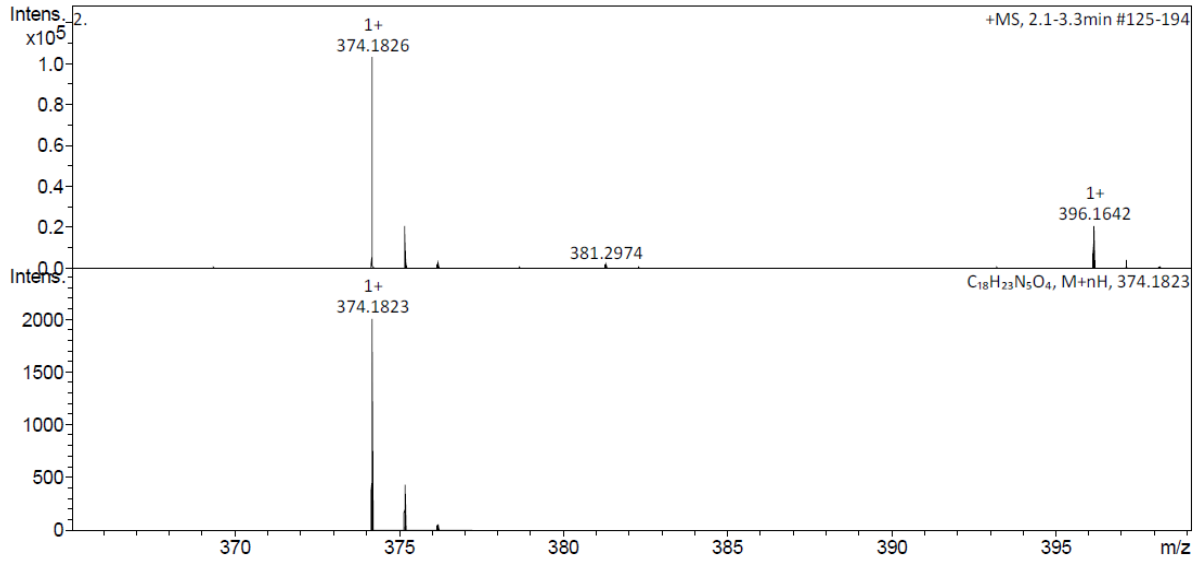


Figure S23: HR-ESI mass spectrum of **J** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **J.**

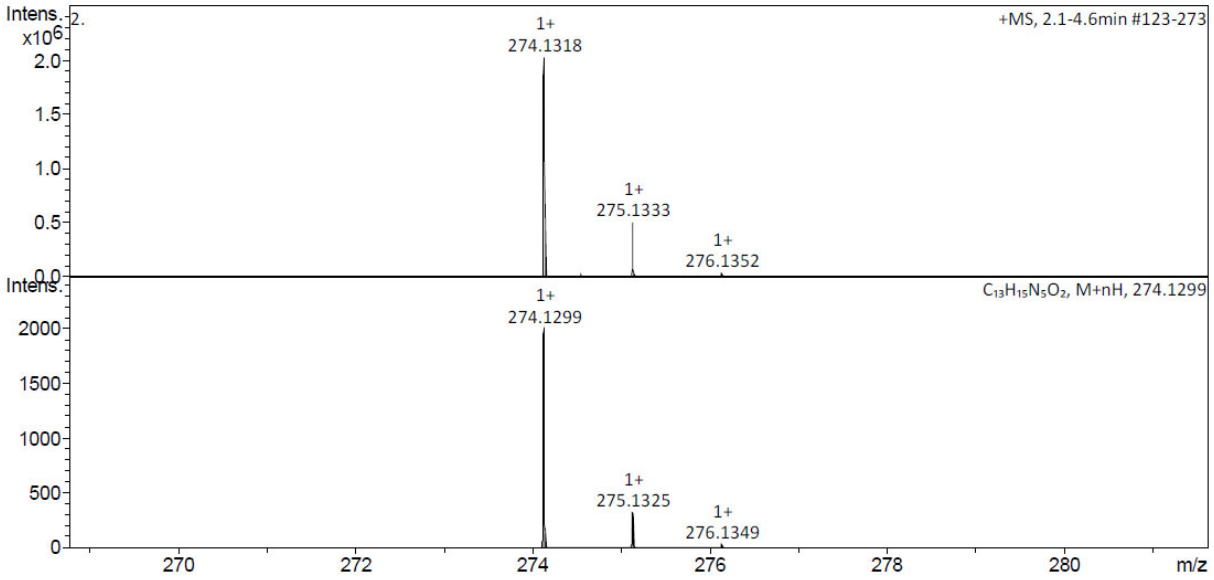


Figure S24: HR-ESI mass spectrum of **2** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **2.**

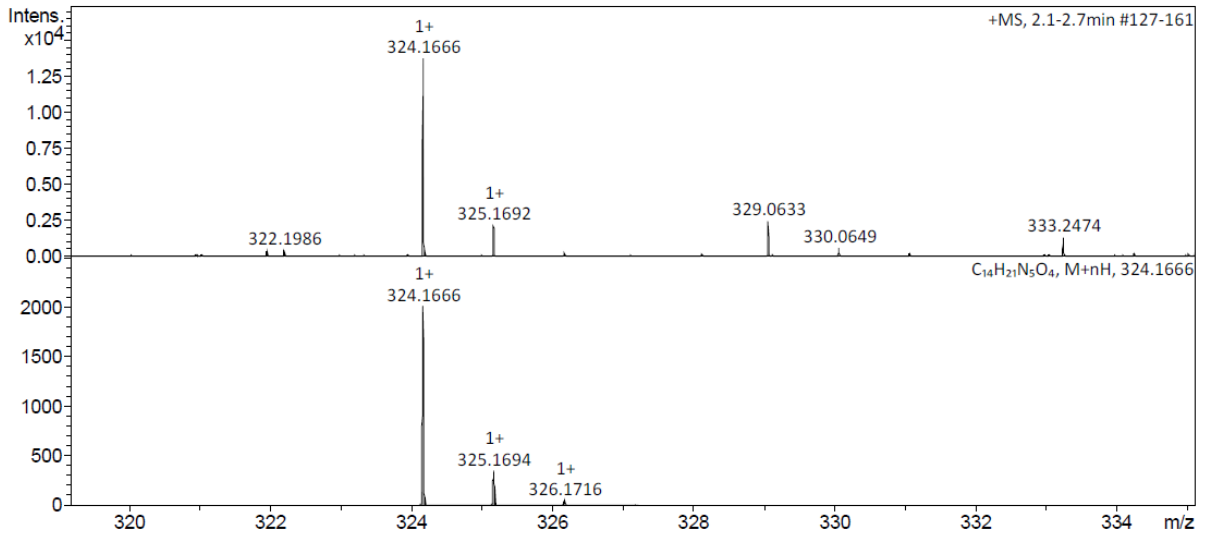


Figure S25: HR-ESI mass spectrum of **L** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **L.**

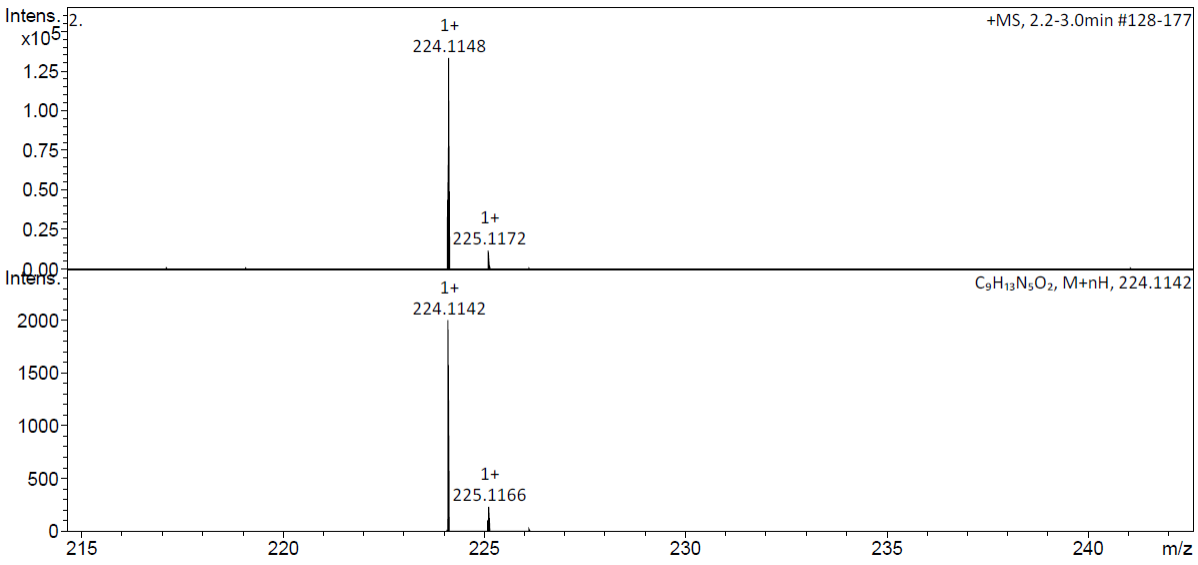


Figure S26: HR-ESI mass spectrum of **1** (positive ion mode, MeOH) and predicted mass spectrum of peaks which belongs to **1.**

Analytical HPLC

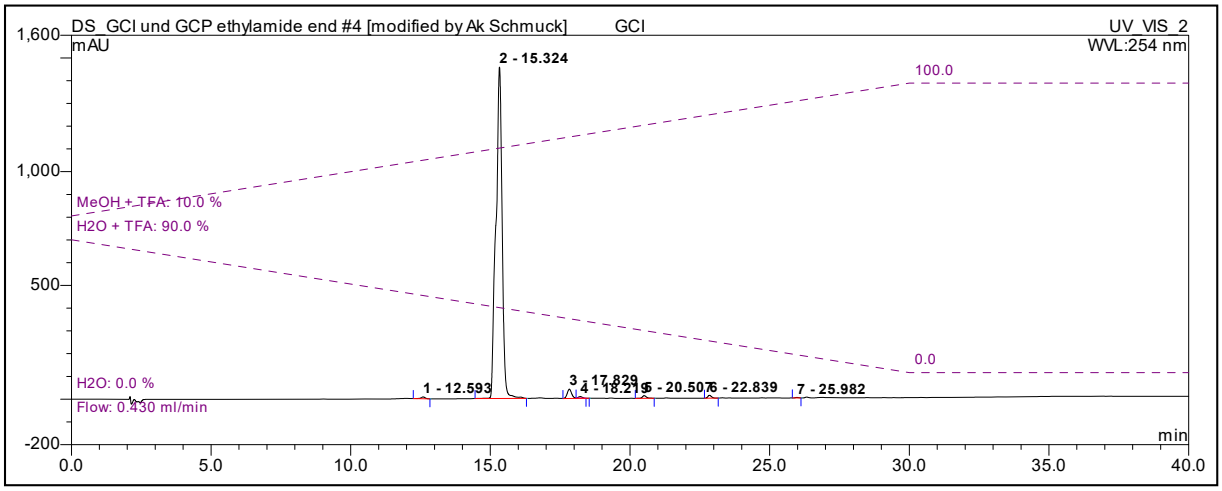


Figure S27: Analytical HPLC (RP 18 MeOH/H2O + 0.1% TFA, 10% MeOH + 0.1% TFA to 100% MeOH + 0.1% TFA, gradient) **2**.

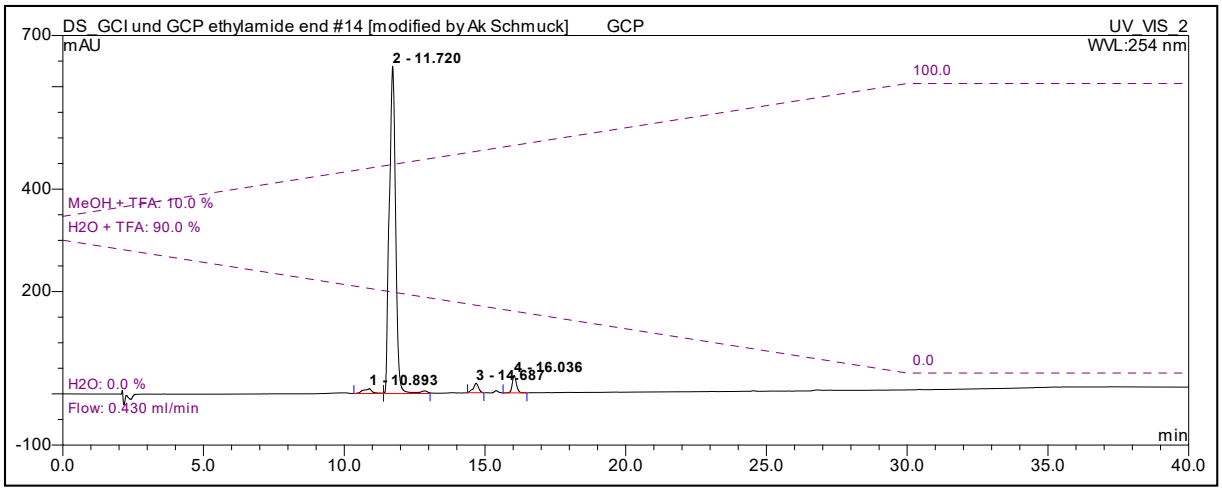


Figure S28: Analytical HPLC (RP 18 MeOH/H2O + 0.1% TFA, 10% MeOH + 0.1% TFA to 100% MeOH + 0.1% TFA, gradient) **1**.

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References

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2. C. Rether and C. Schmuck, *European Journal of Organic Chemistry*, 2011, **2011**, 1459.
3. C. Schmuck, V. Bickert, M. Merschky, L. Geiger, D. Rupprecht, J. Dudaczek, P. Wich, T. Rehm and U. Machon, *European Journal of Organic Chemistry*, 2008, **2008**, 324.