Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Template-assisted photodimerization of *N*-unprotected uracil derivatives - Selective formation of the *cis-syn* photodimer

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General methods

^{1}H NMR spectra were recorded on an *Avance* 400 MHz instrument in DMSO-$d_6$or $CDCl_3$. Chemical shifts (δ) are given in parts per million (ppm). ^{1}H NMR spectra were referenced to the residual proton signal ofDMSO-d_6 at δ = 2.50 ppm (quint) or of $CDCl_3$ at δ = 7.26 ppm (s). ^{13}C NMR spectra were fully ^{1}H-decoupled by broadband decoupling and referenced to the DMSO-d_6 signal at δ = 39.43 ppm (sept) or the $CDCl_3$ signal at δ = 77.0 ppm (t). The following abbreviations were used to describe splitting patterns: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sept = septet, m = multiplet. Coupling constants *J* are given in Hz.

High resolution mass spectra were recorded with an APEX II Bruker Daltonic instrument using the ESI method in negative ion mode.

Thin-layer chromatography was carried out on precoated silica gel 60 plates marked with a fluorescence indicator (Macherey-Nagel, ALUGRAM Xtra SIL G/UV_{254}) and visualization was achieved by UV light (254 nm) or by dyeing with Pauly's Reagent.

Flash column chromatography was carried out on silica gel (0.040 – 0.063 mm).

Unless otherwise stated, all reactions were carried out under air.

IR spectra were measured neat, and were recorded with a JASCO FT/IR-4100 instrument. A selection of most intense signals is given in wavenumbers (cm^{-1}).

Ion-exchange chromatography was carried out on either strongly acidic Amberlyst 15(H) resin or on strongly basic Amberlyst A-26(OH) resin (Alfa Aesar).

X-ray single crystallographic analysis was conducted on a Bruker APEXII Duo Diffractometer using Cu K alpha radiation.

UV-Vis spectra were recorded on a Lambda 9 Spectrophotometer (Perkin Elmer).

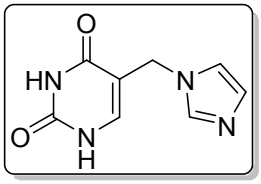
Elementary analysis was carried out on CHNS-Analyzer varioMICRO V1.9.2 (Elementar Analysesysteme GmbH).

UV-irradiation experiments were carried out using a UV-6W handlamp at 254 or 366 nm (500 µW or 850 µW at 15 cm distance) by Dr. Wieland GmbH & Co. KG.

Solvents and reagents

Solvents for flash chromatography (dichloromethane, methanol) were distilled before use. Unless stated, all other commercially available substances and reagents were used as received from their suppliers (Acros, AlfaAesar, Sigma Aldrich, VWR) without further purification. 5-(Hydroxymethyl)uracil**5** was synthesized as described earlier.[1] 5-(Chloromethyl)uracil **6** was prepared based on literature procedure.[2] Diketopiperazines **2a,b** and **4** were synthesized according to an established procedure described earlier.[3] The spectroscopic data were in agreement with the given literature. Solvent Systems for TLC: (A) *n*-BuOH/ MeOH / H_2O / NEt_3 60:20:20:1(v/v).

Synthesis of 5-((1-*H*-Imidazol-1-yl)methyl)uracil (IMU) 1



To a solution of 5-(chloromethyl)uracil **6** (1.00 g, 6.23 mmol, 1 eq) in dry DMF (8 mL) at rt, Imidazole (848 mg, 12.46 mmol, 2.00 eq) dissolved in 7 mL dry DMF was added and the reaction mixture was heated to 50 °C. After 3 hours the reaction was cooled to room temperature, the insoluble dimeric salt was filtered off and the reaction mixture was concentrated under reduced pressure. The residue was purified by flash chromatography (DCM : MeOH7:1 + 1% NH_3) to give the title compound as a colorless solid 335 mg (1.74 mmol, 28%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.39 (br s, 1H), 11.29 (s, 1H), 8.97 (s, 1H), 7.91 (d, J = 4.8 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.56 – 7.44 (m, 1H), 5.00 (s, 2H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.9, 151.2, 143.6, 135.6, 121.5, 120.8, 105.7, 44.5;

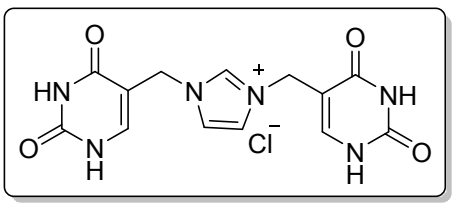
IR ν_{max}/cm^{-1} 3732, 3726, 3629, 3024, 2825, 2359, 2342, 1653, 1448, 669;

HRMS (ESI) m/z calcd. for $C_8H_9N_4O_2$ $[M+H]^+$: 193.0720, found: 193.0720;

EA calcd. for $C_8H_9N_4O_2$: C: 50.00, H: 4.20, N: 29.15; found: C: 49.79, H: 3.95, N: 29.11.

R_F (EtOAc/MeOH, 1:1 + 1% NH_3) = 0.60.

Synthesis of 1,3-bis((uracil-5-yl)methyl)-1*H*-imidazol-3-ium chloride 1b



To a solution of 5-(Chloromethyl)uracil **6** (1.00 g, 6.23 mmol, 1 eq) in dry DMF (8 mL) at rt, Imidazole (848 mg, 12.46 mmol, 2.00 eq) dissolved in 7 mL dry DMF was added and the reaction mixture was heated to 50 °C. After 3 hours the reaction was cooled to room temperature and the precipitated white

solid was filtered off. The reaction product was recrystallized from water to give the title compound as a colourless solid 624 mg (1.77 mmol, 57%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.36 (s, 2H), 11.33 (s, 2H), 9.22 (s, 1H), 7.89 (s, 2H), 7.71 (s, 2H), 4.99 (s, 4H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 163.8, 151.1, 143.9, 136.1, 122.2, 105.1, 45.3;

IR ν_{max}/cm^{-1} 3438, 3150, 3024, 2962, 2893, 2826, 2358, 2342, 1706, 1654;

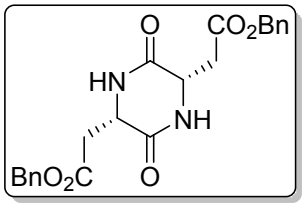
HRMS (ESI) m/z calcd. for $C_{13}H_{13}N_6O_4$ $[M]^+$: 317.0993, found: 317.0996.

R_F (A) = 0.11.

General procedure for the hydrolysis of the dimeric salt 1bto IMU 1

1,3-bis((uracil-5-yl)methyl)-1H-imidazol-3-ium chloride**1b**(350 mg, 1.00 mmol, 1 eq) was dissolved in 20% aq. NaOH and stirred at 50 °C for 15 h. The reaction mixture was adjusted to pH 7 by addition of 3 M aq. HCl and concentrated under reduced pressure. The residue was purified by flash chromatography (DCM : MeOH4:1 + 1% NH_3) to give 5-((1-*H*-Imidazol-1-yl)methyl)uracil**1** as a white solid 153 mg (0.80 mmol, 80%).

Synthesis of *cyclo*(L-Asp-L-Asp) 2a



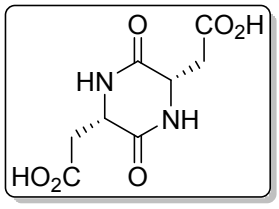
Cyclo[L-Asp(OBn)-L-Asp(OBn)] **4** was synthesized according to the literature procedure using Boc-L-Asp(OBn)-OH (10.0 g, 30.94 mmol, 2 eq) and Boc-L-Asp(OBn)-OH (14.7 g, 45.72mmol, 3 eq). The crude product was triturated in water, recrystallized from methanol/ H_2O 80:20 (v/v) and dried under reduced pressure. *Cyclo*[L-Asp(OBn)-L-Asp(OBn)] **4** was received as a colourless solid (2.96 g, 7.22 mmol, 56%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.29 (m, 10H), 6.80 (s, 2H), 5.15 (d, J = 1.6 Hz, 4H), 4.38 (m, 2H), 3.11 (dd, J = 17.8, 3.0 Hz, 2H), 2.91 (dd, J = 17.8, 9.1 Hz, 2H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 166.0, 135.2, 128.8, 128.7, 128.5, 67.4, 51.7, 38.1;

IR ν_{max}/cm^{-1} 3186, 3060, 3034, 2938, 1735, 1683, 1652, 1453, 1388, 1158;

HRMS (ESI) m/z berechnet für $C_{22}H_{22}N_2O_6Na$ $[M+Na]^+$: 433.1370, gemessen: 433.1374.



Cyclo[L-Asp(OBn)-L-Asp(OBn)] **4** (2.00 g, 4.87mmol) was suspended in 50 mL methanol and Pd/C (10%, 150 mg) was added. The reaction mixture was stirred for 3 hours under hydrogen atmosphere, filtered through Celite® and the solvent was removed under reduced pressure. The raw product was further recrystallized from methanol to give the *cyclo*(L-Asp-L-Asp) **2a** as a colourless solid (1.02 g, 4.43 mmol, 91%).

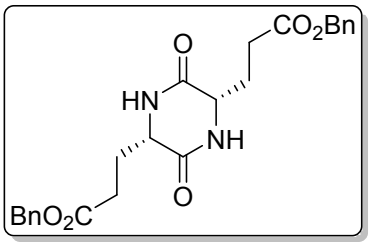
^{1}H NMR (400 MHz, DMSO-d_6) δ 12.35 (s, 2H), 8.08 (s, 2H), 4.24 (t, J = 5.6 Hz, 2H), 2.73 – 2.57 (m, 4H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 171.6, 167.6, 51.0, 36.2;

IR ν_{max}/cm^{-1} 2359, 2339, 1716, 1683, 1652, 1456, 1418, 1327, 1278, 1243;

HRMS (ESI) m/z calcd. for $C_8H_{10}N_2O_6Na$ $[M+Na]^+$: 253.0431, found: 253.0431.

Synthesis of *cyclo*[L-Glu-L-Glu] 2b



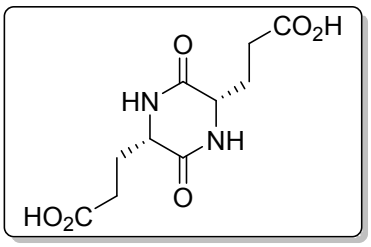
Cyclo[L-Glu(OBn)-L-Glu(OBn)] **4b** was synthesized according to the literature procedure using Boc-L-Glu(OBn)-OH (12.5 g, 37.0mmol, 2 eq) and Boc-L-Glu(OBn)-OH (19.1 g, 56.0mmol, 3 eq). The crude product was triturated in water and dried under reduced pressure. *Cyclo*[L-Glu(OBn)-L-Glu(OBn)] **4b** was received as a colourless solid (1.3 g, 3.00mmol, 19%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.22 (d, J = 1.9 Hz, 2H), 7.44 – 7.26 (m, 10H), 5.08 (s, 4H), 3.96 – 3.86 (m, 2H), 2.48 – 2.39 (m, 4H), 2.05 – 1.87 (m, 4H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 172.2, 167.8, 136.2, 128.4, 128.0, 127.9, 65.5, 53.1, 29.2, 27.9;

IR ν_{max}/cm^{-1} 3182, 3037, 2962, 2892, 2356, 2342, 1733, 1670, 1162, 697;

HRMS (ESI) m/z calcd. for $C_{24}H_{26}N_2O_6Na$ $[M+Na]^+$: 461.1683, found: 461.1681.



Cyclo[L-Glu(OBn)-L-Glu(OBn)] **4b** (1.00 g, 2.30mmol) was suspended in 70 mL methanol and Pd/C (10%, 61 mg) was added. The reaction mixture was stirred for 3 hours under hydrogen atmosphere, filtered through Celite® and the solvent was removed under reduced pressure. The raw product was further recrystallized from methanol to give the *cyclo*(L-Glu-L-Glu) **2b** (190 mg, 0.70 mmol, 31%).as a colourless solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.98 (s, 2H), 8.19 (s, 2H), 3.87 (t, J = 5.4 Hz, 2H), 2.40 – 2.19 (m, 4H), 2.06 – 1.74 (m, 4H);

^{13}C NMR (100 MHz, DMSO-d_6) δ 174.1, 167.9, 53.3, 29.5, 28.3;

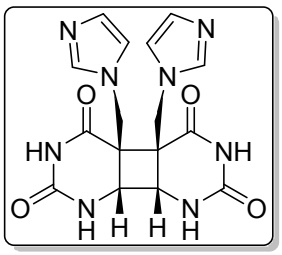
IR ν_{max}/cm^{-1} 3735, 3628, 3618, 3207, 2939, 2359, 2341, 1699, 1635, 1254;

HRMS (ESI) m/z calcd.for $C_{10}H_{14}N_2O_6Na$ $[M+Na]^+$: 281.0744, found: 281.0744.

General procedure for the photodimerzation of IMU 1

A solution of IMU **1** (50 mg, 0.26 mmol) and indicated amounts of template in 20 mL H_2O / Acetone 60/40 (v/v) was degassed by a stream of nitrogen for 30 minutes in a quartz glass tube. The solution was exposed to UV irradiation at 366 nm for 3 days. The reaction mixture was concentrated under reduced pressure. In case of **2a,b** as a template, the residue was dissolved in ca. 3 mL water and applied to an Amberlyst 15(H) cation-exchange column. The column was washed with water to neutral, eluting the template **2a,b**. Subsequently, the photodimers **3a,b** were eluted with NH_4OH (1 M aq). The photodimer containing fractions were pooled and concentrated under reduced pressure. The residue was purified by flash chromatography (DCM :MeOH 2 : 1 → 1 : 1 + 1% NH_3) to give the photodimers **3a,b** as a colourless solid (22 mg, 0.057 mmol, 22%).

Synthesis of *cis*-4a,4b-bis((1*H*-imidazol-1-yl)methyl)hexahydro-cyclobuta[1,2-e:4,3-e']dipyrimidine-2,4,5,7(1*H*,3*H*,6*H*,8*H*)-tetraone 3a



A solution of IMU **1** (50 mg, 0.26 mmol) in 20 mL H_2O / Acetone 60/40 (v/v) was degassed by a stream of nitrogen for 30 minutes in a quartzglass tube. The solution was exposed to UV irradiation at 366 nm for 3 days. The reaction mixture was concentrated under reduced pressure and the residue was purified by flash chromatography (DCM :MeOH 2 : 1 → 1 : 1 + 1% NH_3) to give the title compound as a colourless solid (11 mg, 0,029 mmol, 11%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.38 (br s, 2H), 7.84 (s, 2H), 7.51 (s, 2H), 6.99 (s, 2H), 6.89 (s, 2H), 4.76 (d, J = 13.8 Hz, 2H), 4.67 (d, J = 13.8 Hz, 2H), 4.24 (s, 2H);

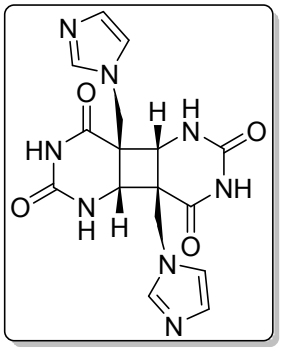
^{13}C NMR (100 MHz, DMSO-d_6) δ 168.1, 151.4, 138.0, 128.8, 119.9, 50.4, 49.2, 46.2;

IR ν_{max}/cm^{-1} 3424, 2919, 2843, 1456, 1373, 1223, 1078, 1033, 820, 667;

HRMS (ESI) m/z calcd. for $C_{16}H_{17}N_8O_4$ $[M+H]^+$: 385.1367, found: 385.1374.

R_F (EtOAc/MeOH, 1:1 + 1% NH_3) = 0.16.

Synthesis of *cis*-4a,8a-bis((1*H*-imidazol-1-yl)methyl)hexahydro-cyclobuta[1,2-e:3,4-e']dipyrimidine-2,4,6,8(1*H*,3*H*,5*H*,7*H*)-tetraone 3b



A solution of IMU **1** (50 mg, 0.26 mmol) in 20 mL H_2O / Acetone 60/40 (v/v) was degassed by a stream of nitrogen for 30 minutes in a quartz glass tube. The solution was exposed to UV irradiation at 366 nm for 3 days. The reaction mixture was concentrated under

reduced pressure and the residue was purified by flash chromatography (DCM :MeOH 2 : 1 → 1 : 1 + 1% NH_3) to give the title compound as a colourless solid (11 mg, 0,029 mmol, 11%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.26 (s, 2H), 7.90 (d, J = 4.6 Hz, 2H), 7.46 (s, 2H), 6.95 (s, 2H), 6.87 (s, 2H), 4.49 (d, J = 14.0 Hz, 2H), 4.43 (d, J = 14.0 Hz, 2H), 4.06 (d, J = 4.6 Hz, 2H);

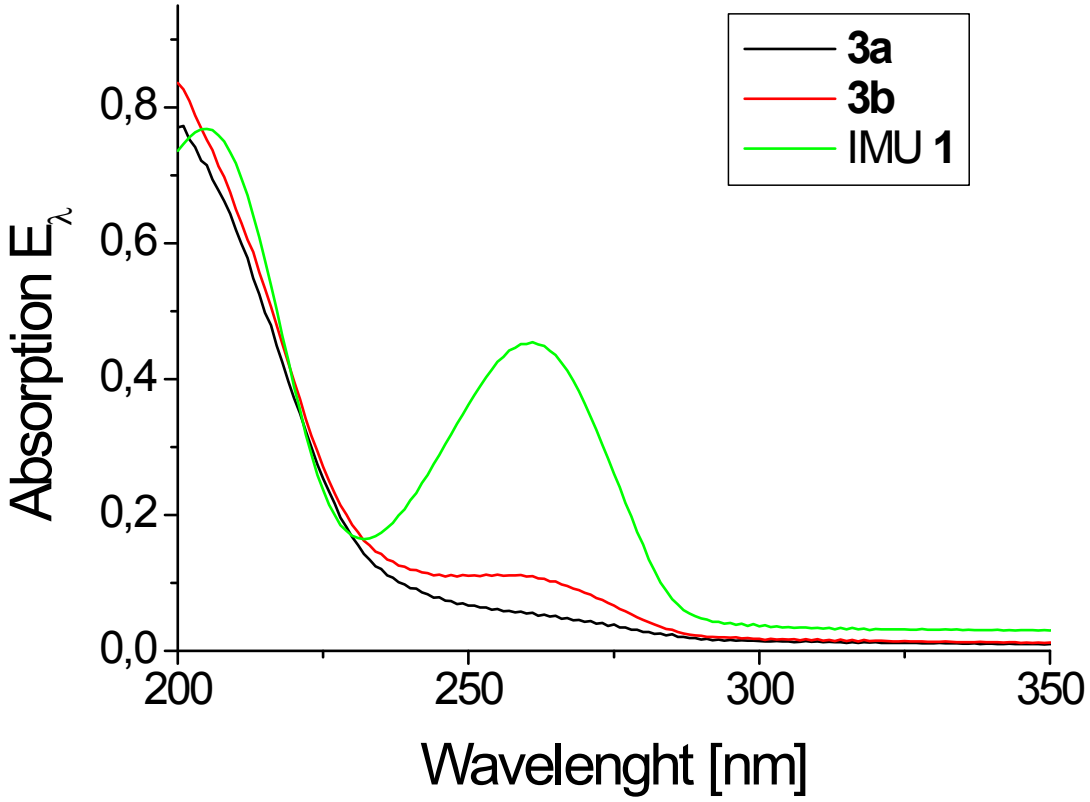
^{13}C NMR (100 MHz, DMSO-d_6) δ 168.5, 151.2, 137.5, 128.9, 119.3, 51.6, 51.1, 50.7;

IR ν_{max}/cm^{-1} 3445, 2920, 1771, 1684, 1507, 1456, 1077, 1033, 1023, 720;

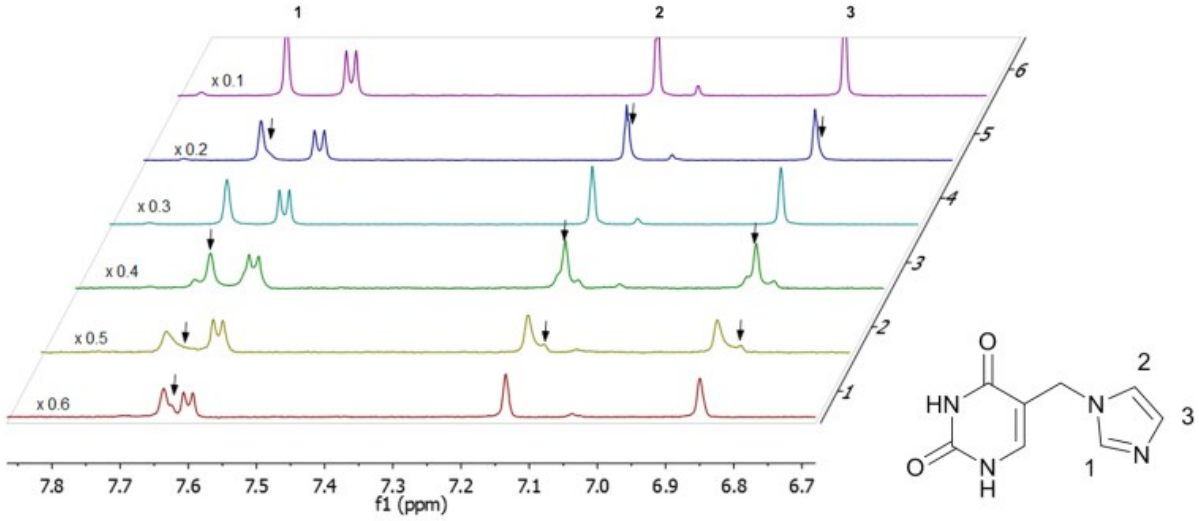
HRMS (ESI) m/z calcd.for $C_{16}H_{17}N_8O_4$ $[M+H]^+$: 385.13673, found: 385.13715.

R_F (EtOAc/MeOH, 1:1 + 1% NH_3) = 0.23.

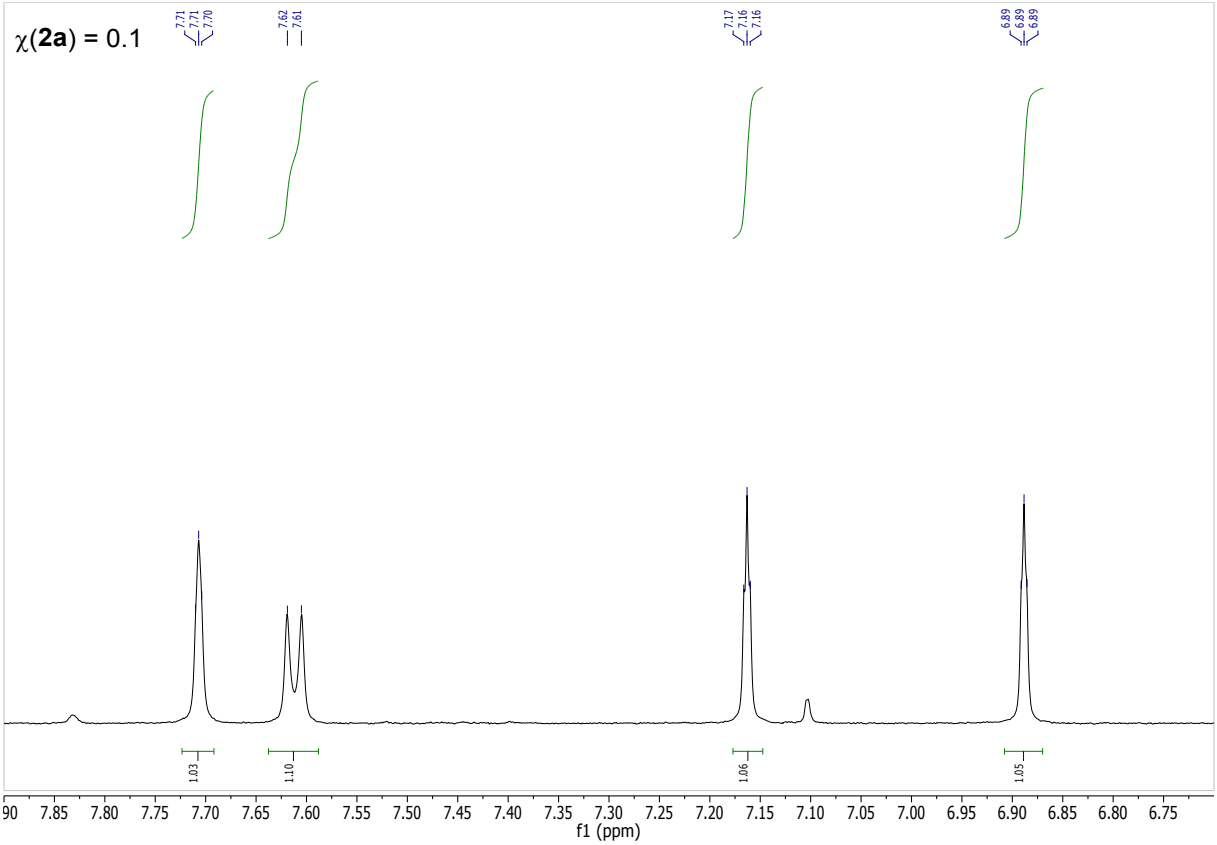
UV-Vis Spectra (0.04 mM in water)

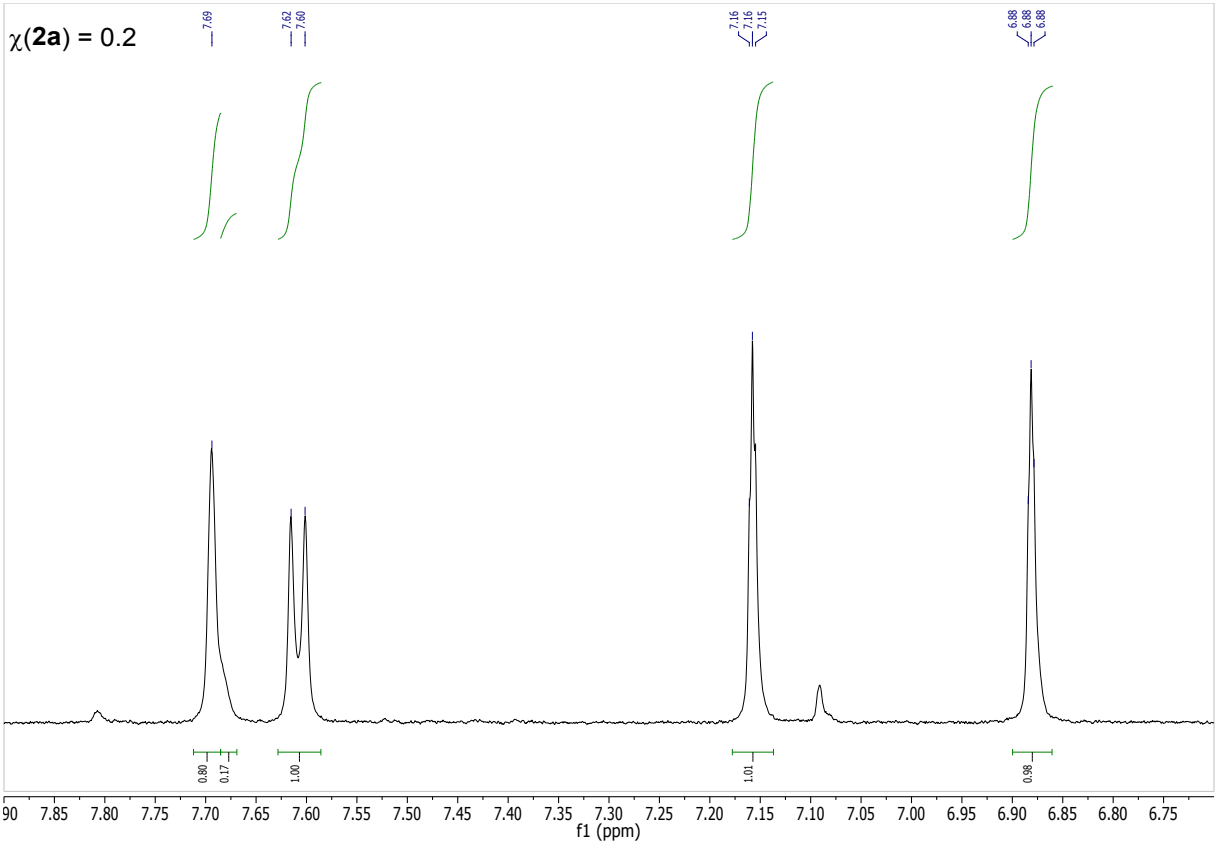


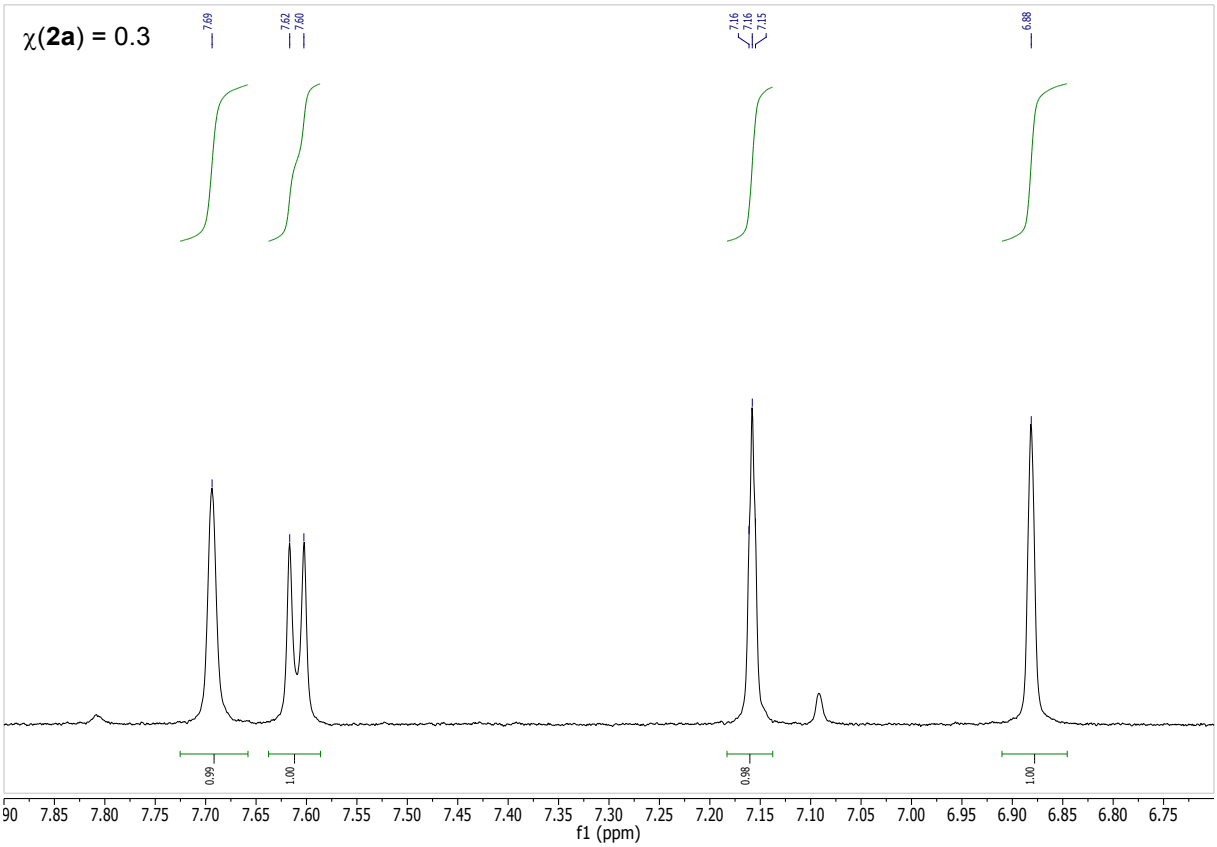
NMR-titration experiment *via* the method of continuous variation of mole fraction χ (2a) in a solution of 1 in DMSO-d_6.

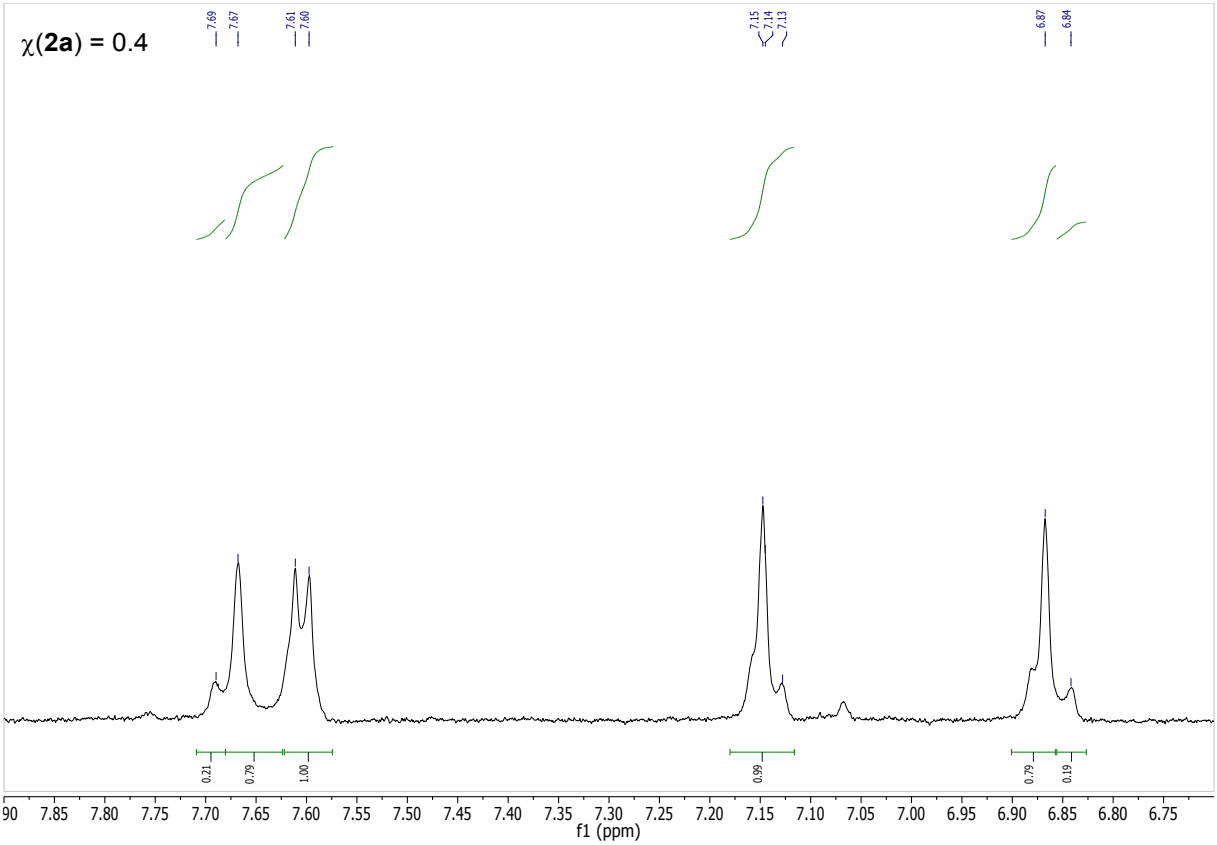


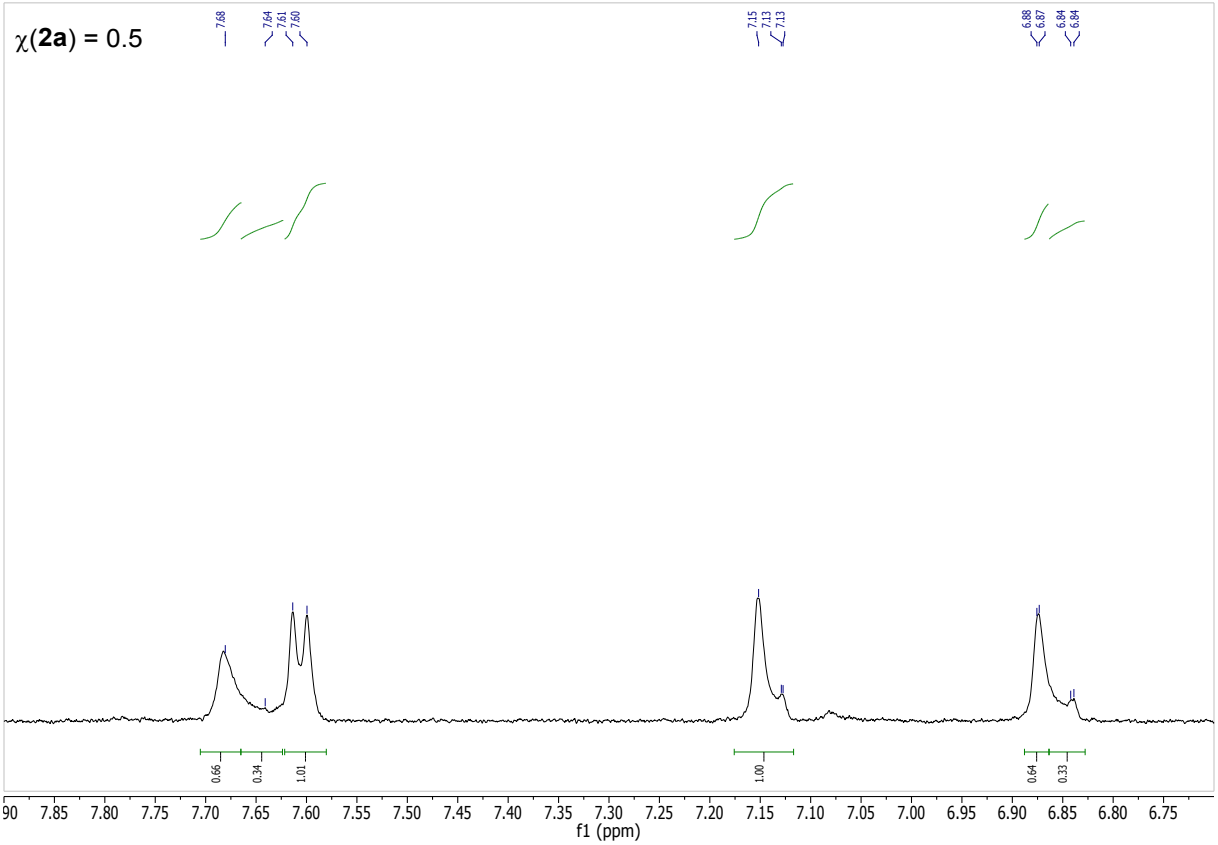
Single Spectra



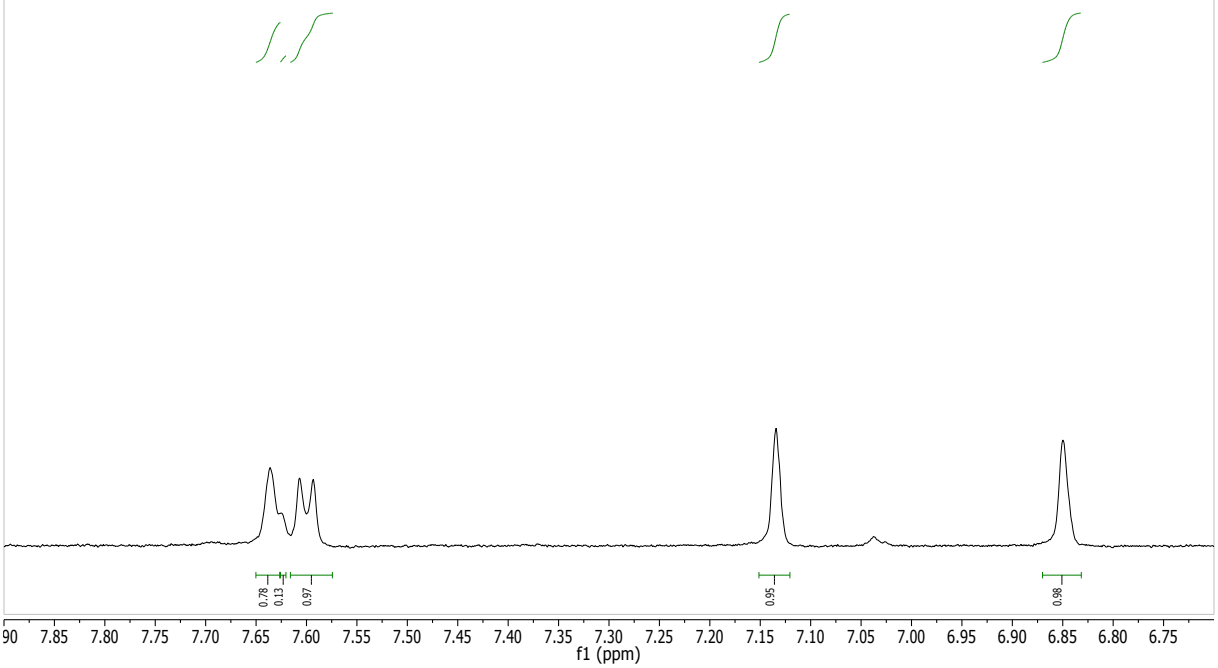

χ(2a) = 0.2
7.69
7.62
7.60
7.16
7.16
7.15
6.88
6.88
6.88
0.80
0.17
1.00
1.01
0.98
f1 (ppm)


χ(2a) = 0.3
7.69
7.62
7.60
7.16
7.16
7.15
6.88
0.99
1.00
0.98
1.00
f1 (ppm)


χ(2a) = 0.4
7.69
7.67
7.61
7.60
7.15
7.14
7.13
6.87
6.84
0.21
0.79
1.00
0.99
0.79
0.19
f1 (ppm)


χ(2a) = 0.5
7.68
7.64
7.61
7.60
7.15
7.13
7.13
6.88
6.87
6.84
6.84
0.66
0.34
1.01
1.00
0.64
0.33
f1 (ppm)

χ(**2a**) = 0.6


0.78
0.13
0.97
0.95
0.98
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 6.80 6.75
f1 (ppm)

χ(**2a**) = 0.7

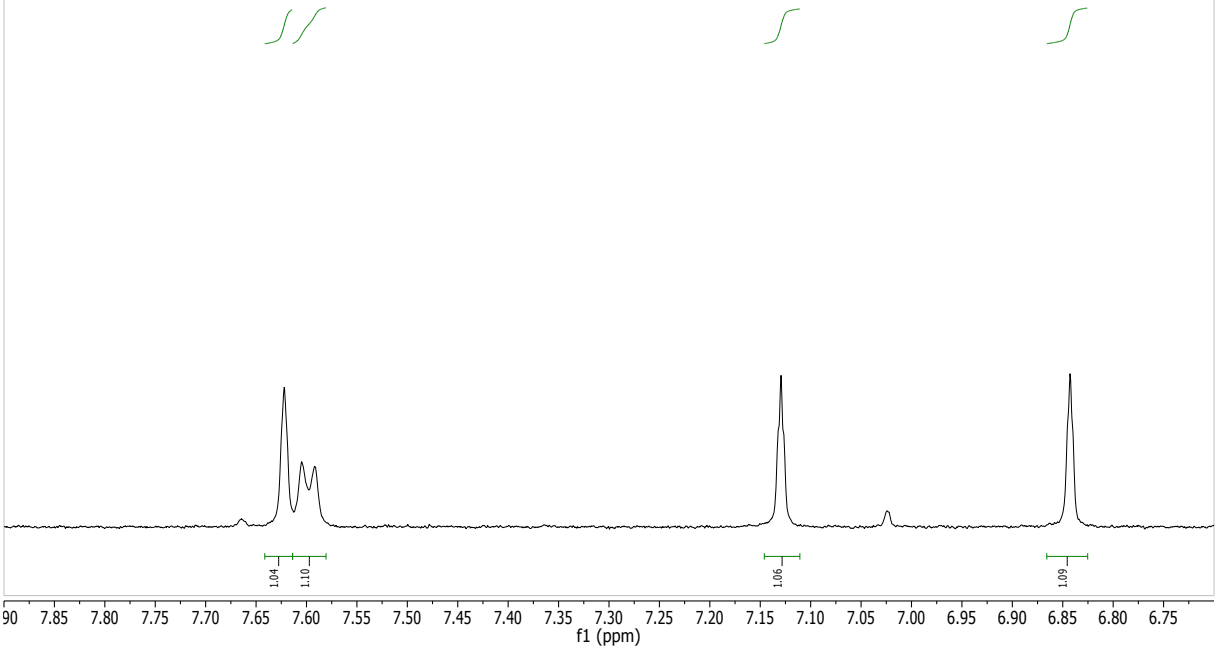
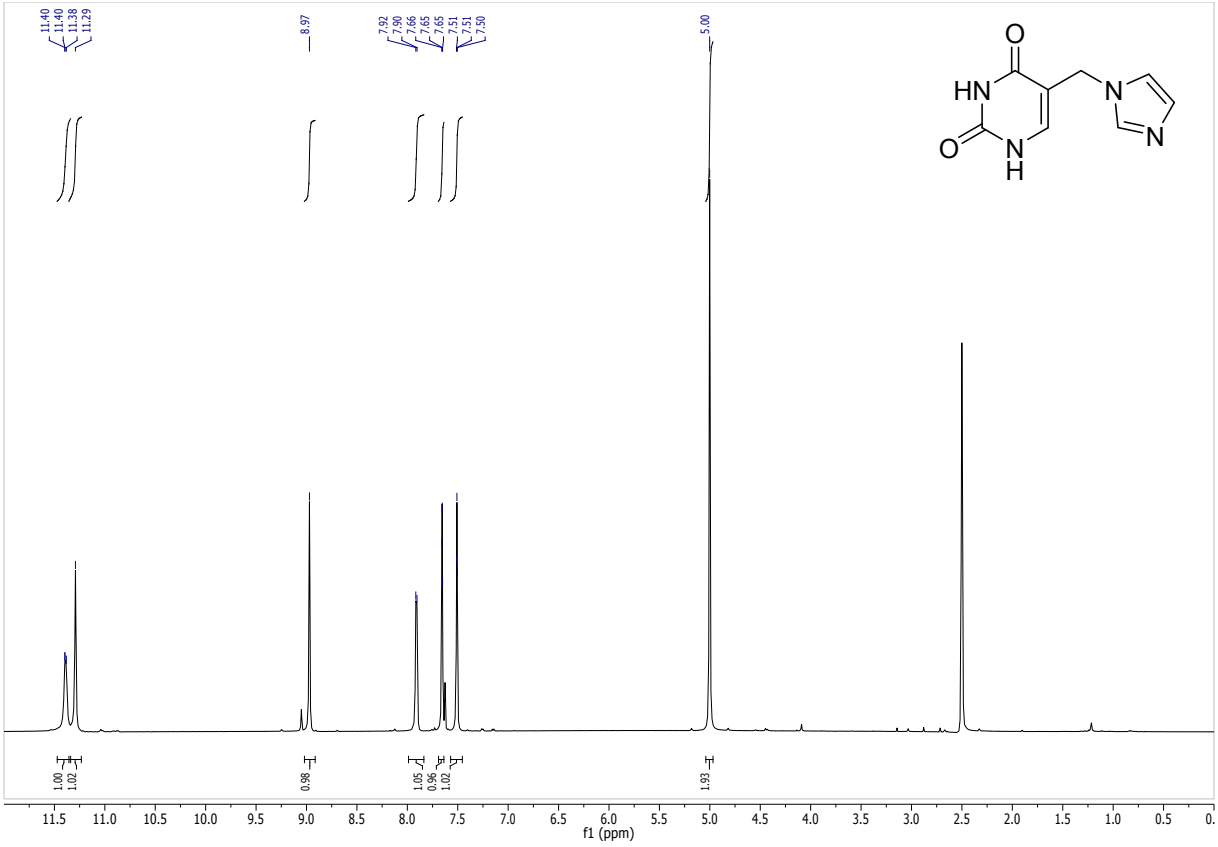

1.04
1.10
1.06
1.09
90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75
f1 (ppm)

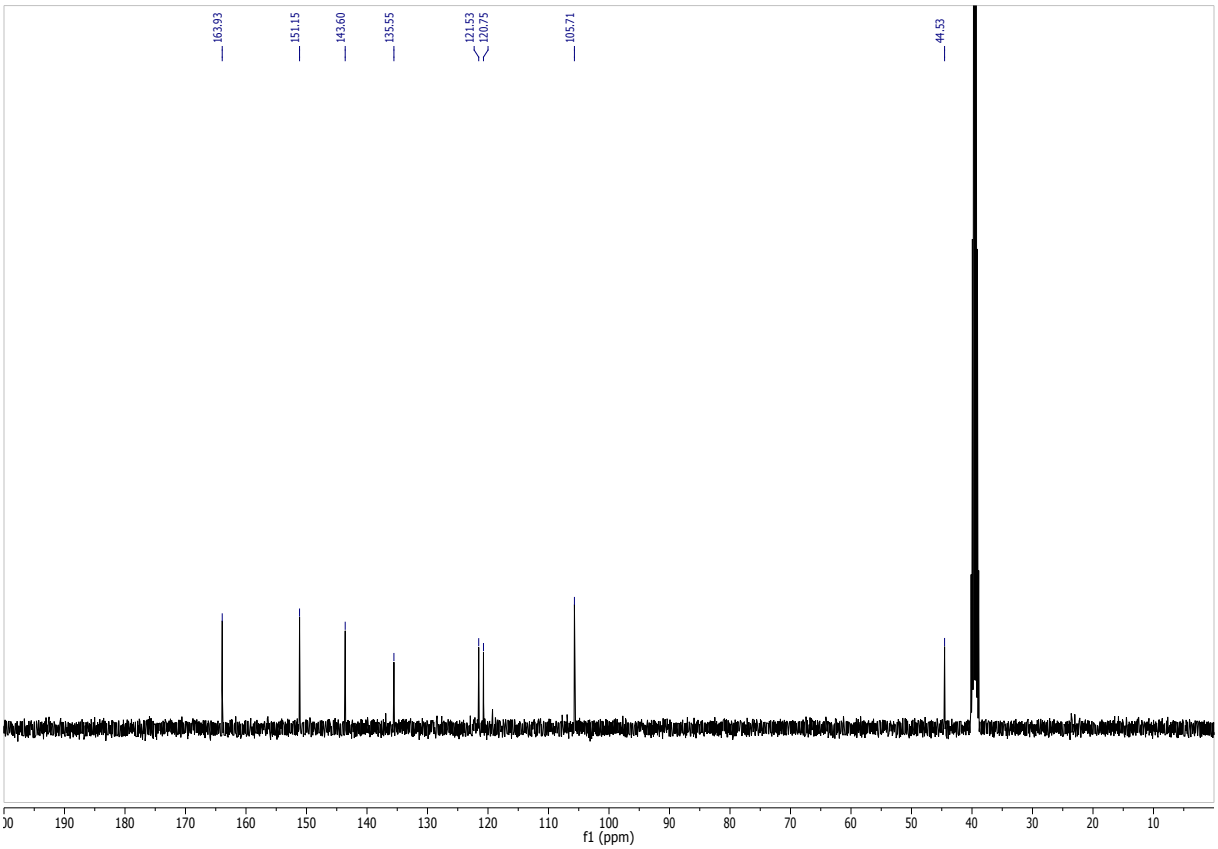
Table 2 – Calculation of molarity bound **2a** during NMR titration.

χ(**2a**)	integral bound **1**	molarity **2a**	molarity bound **2a**
0.1	0	1 mM	0 mM
0.2	0.8	2 mM	1.6 mM
0.3	0.99	3 mM	2.97 mM
0.4	0.79	4 mM	3.16 mM
0.5	0.34	5 mM	1.7 mM
0.6	0.13	6 mM	0.78 mM
0.7	0	7 mM	0 mM

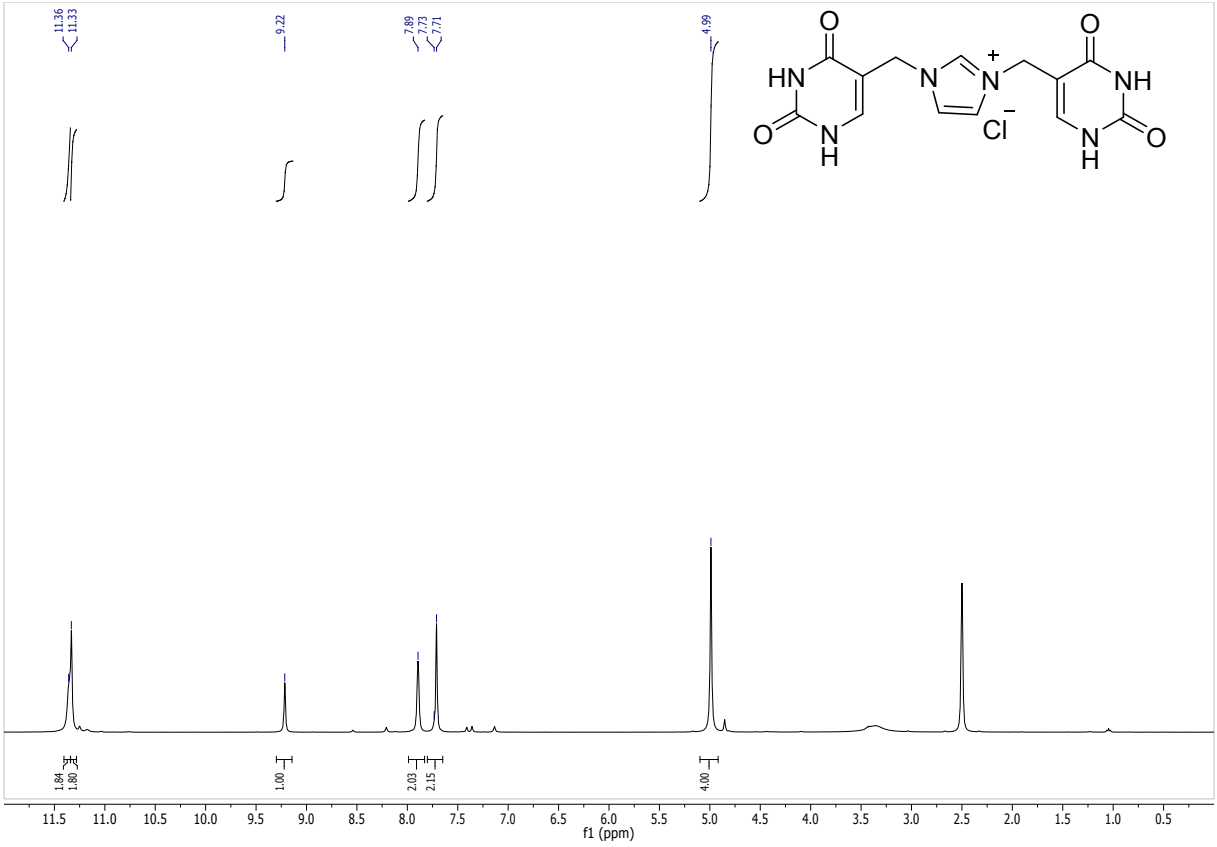
1H and 13C Spectra

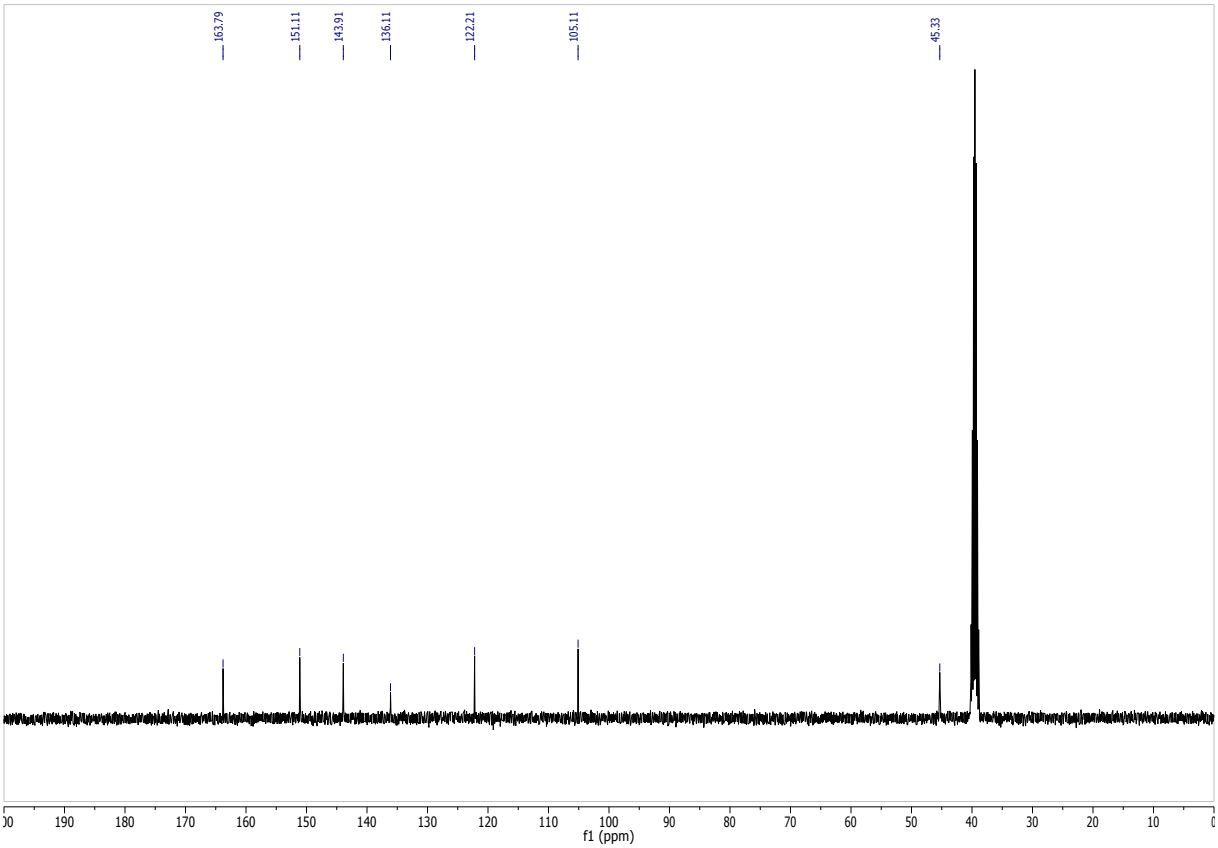
5-((1-*H*-Imidazol-1-yl)methyl)uracil (IMU) 1



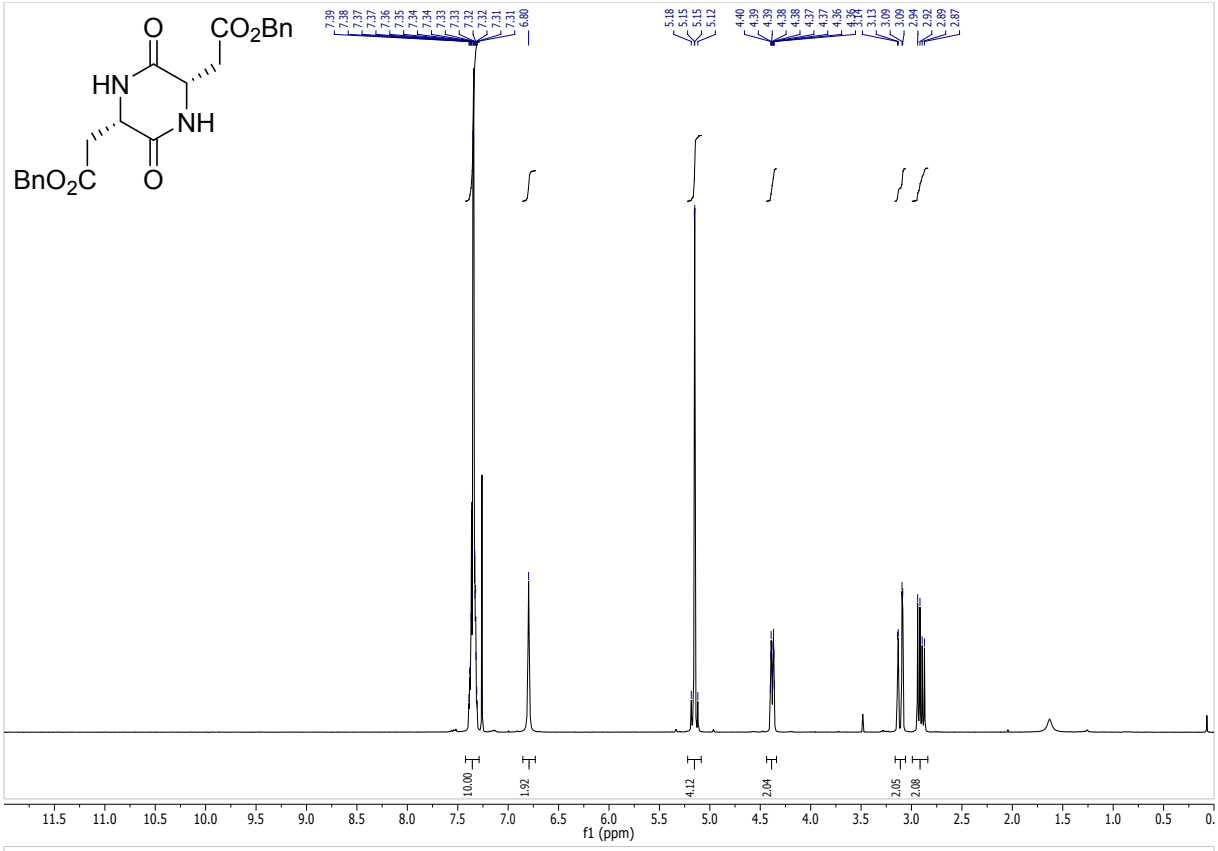


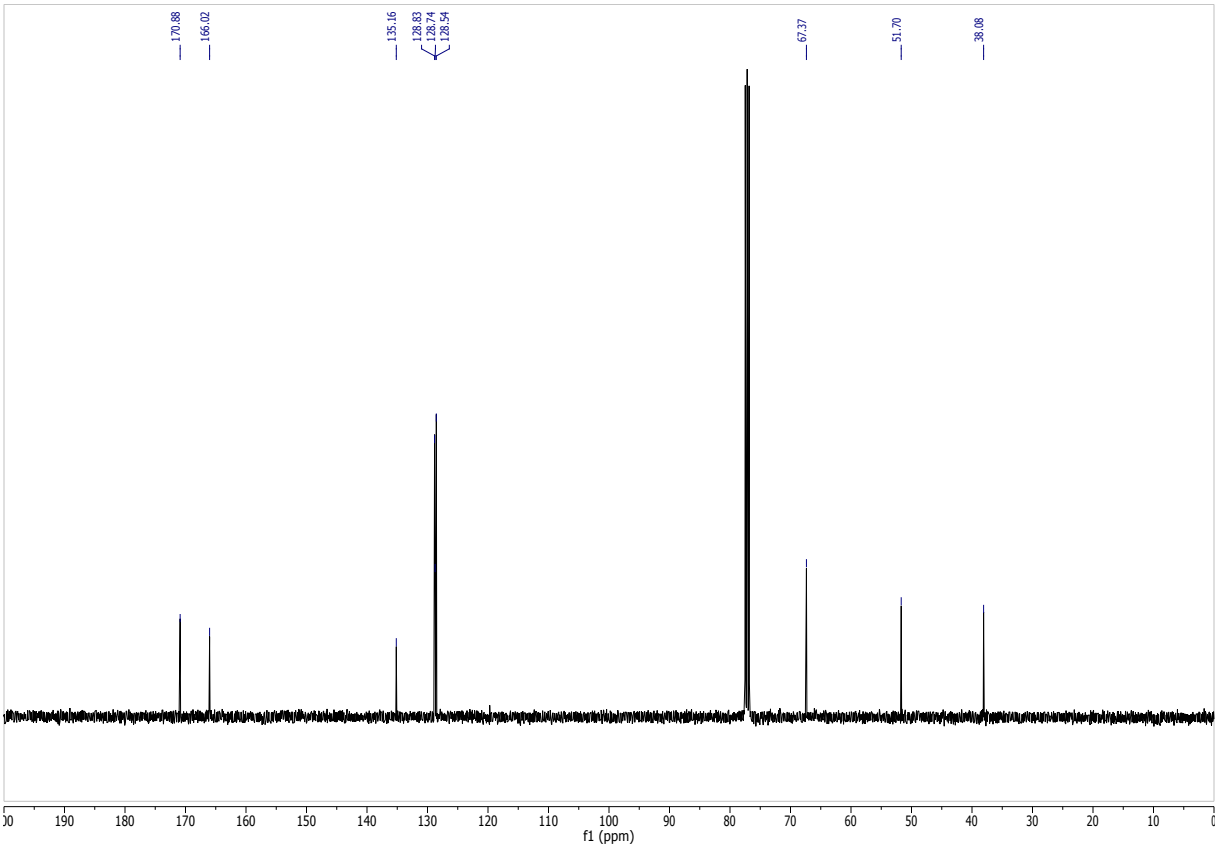
1,3-bis((uracil-5-yl)methyl)-1*H*-imidazol-3-ium chloride 1b



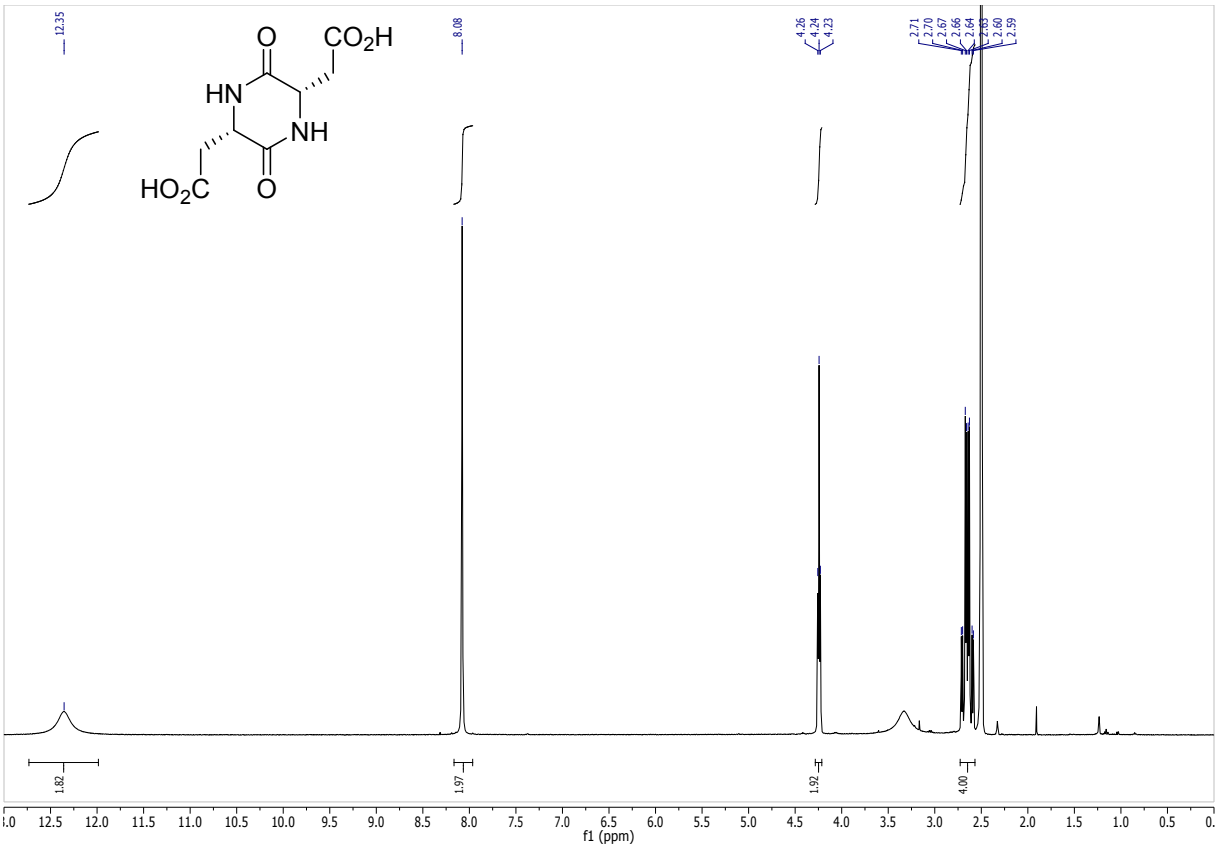


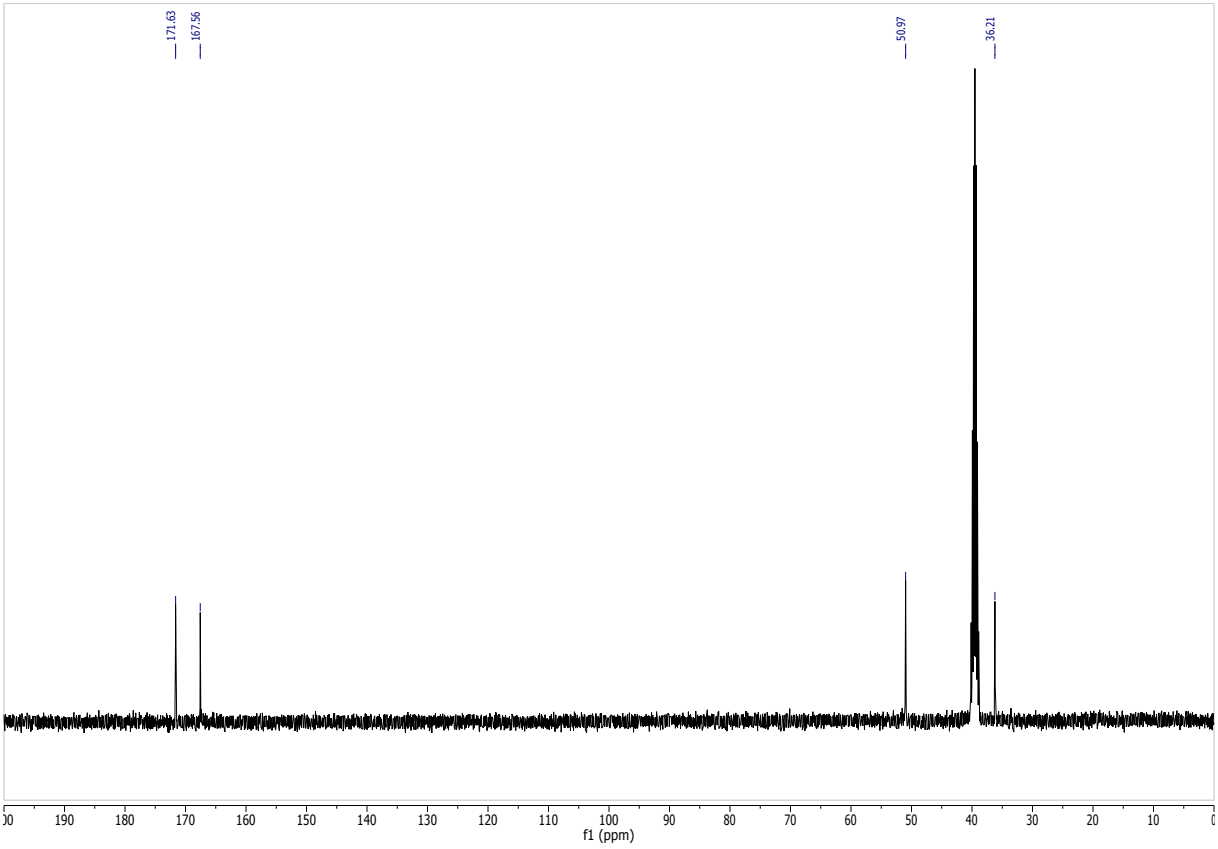
Cyclo[L-Asp(OBn)-L-Asp(OBn)] 4



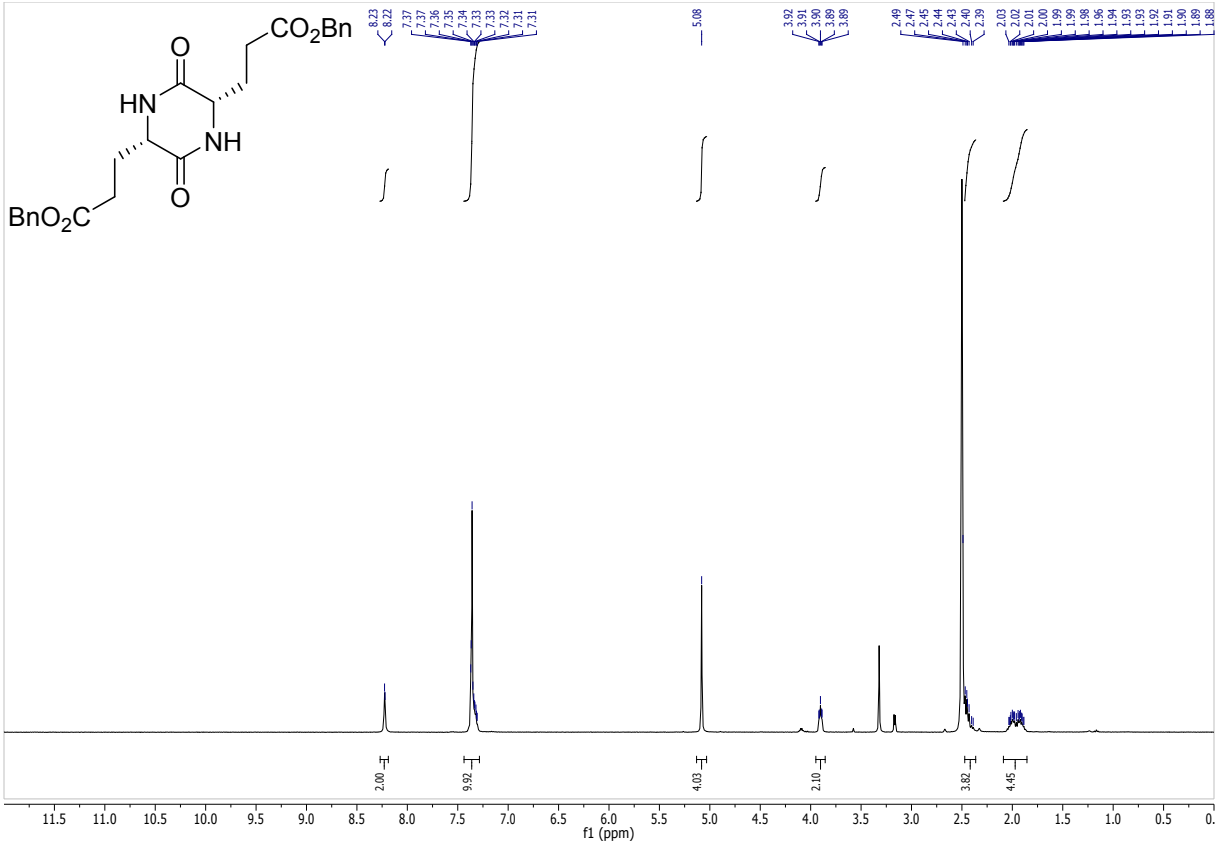


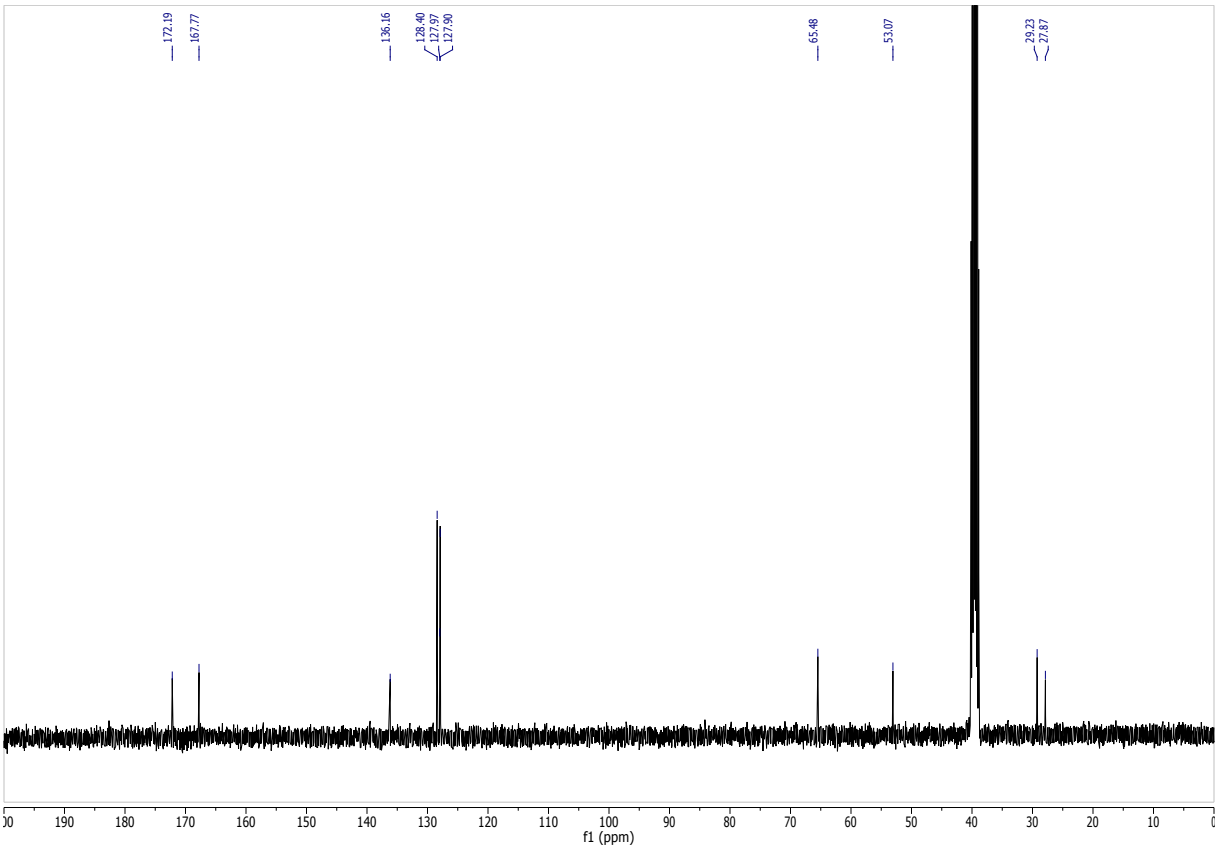
Cyclo(L-Asp-L-Asp) 2a



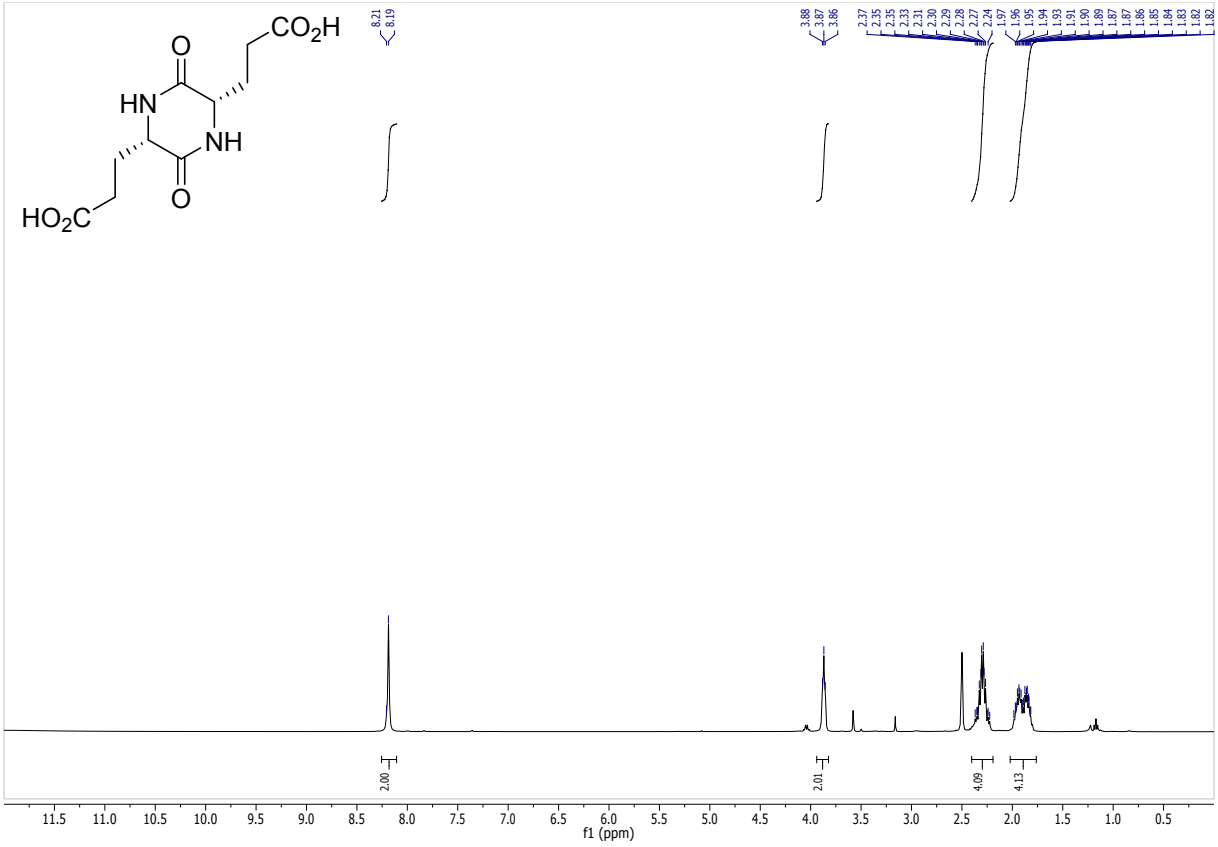


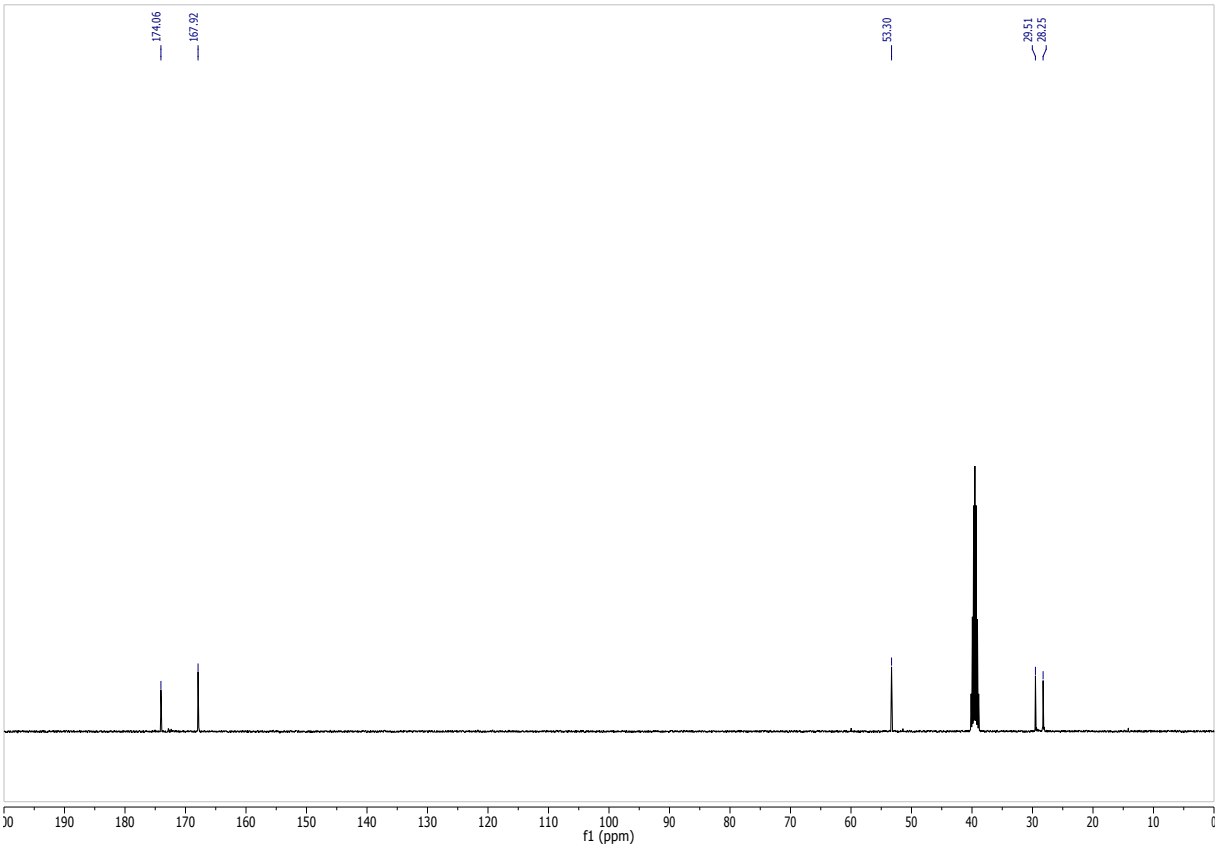
Cyclo[L-Glu(OBn)-L-Glu(OBn)] 4b



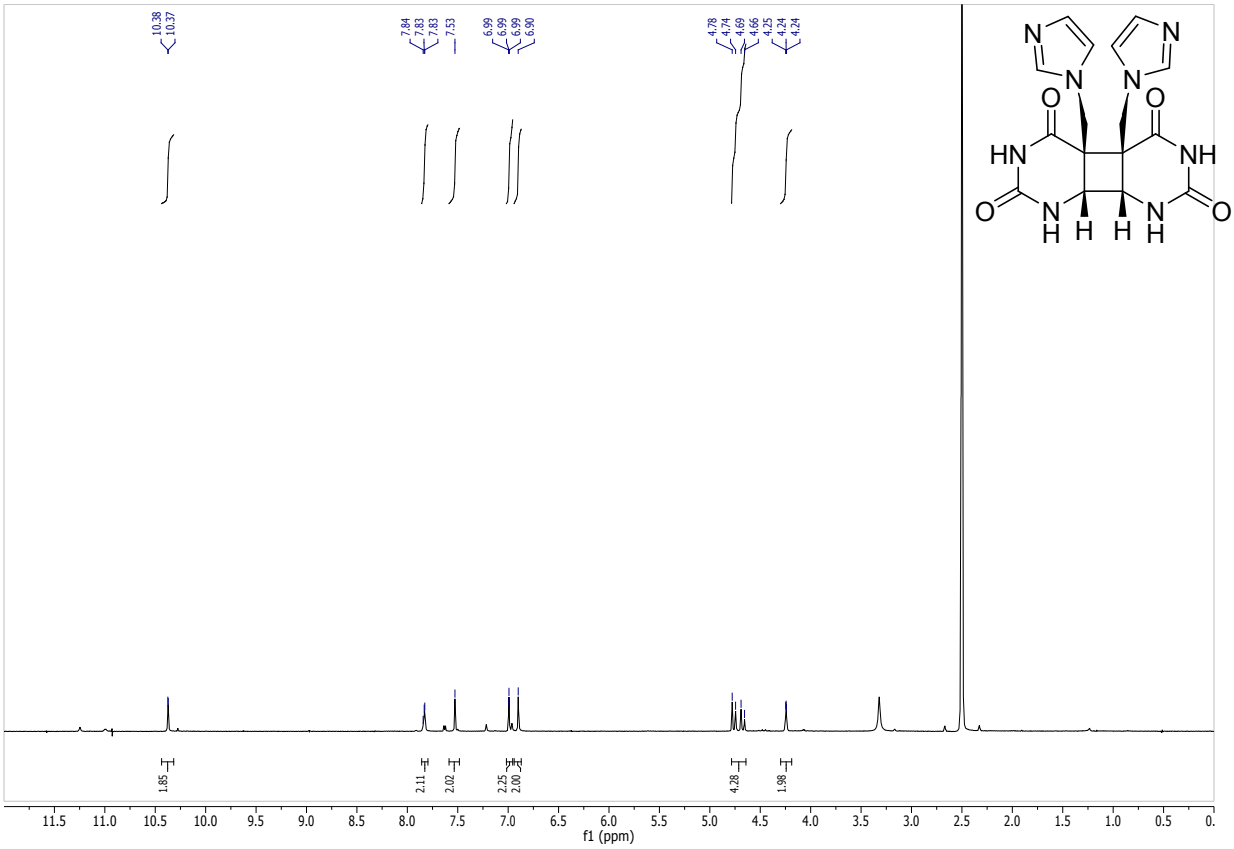


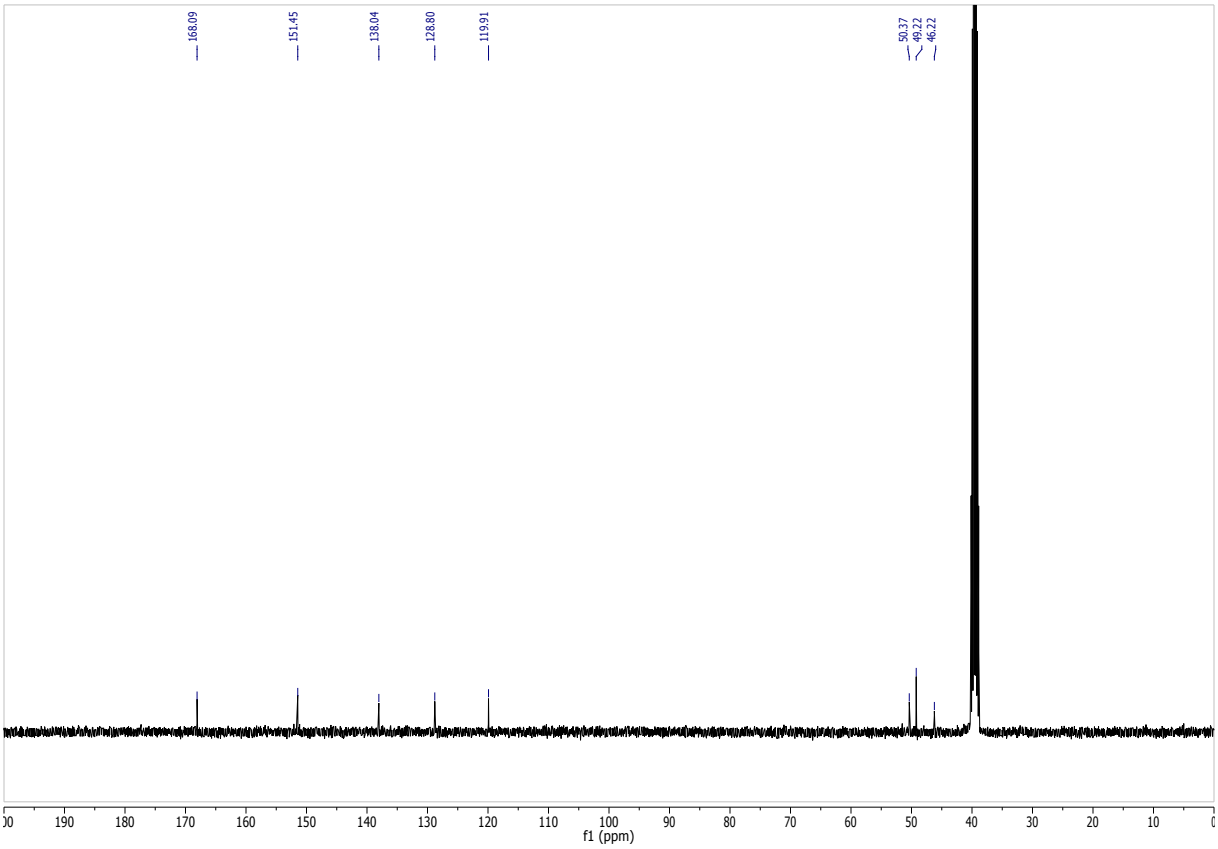
Cyclo(L-Glu-L-Glu) 2b



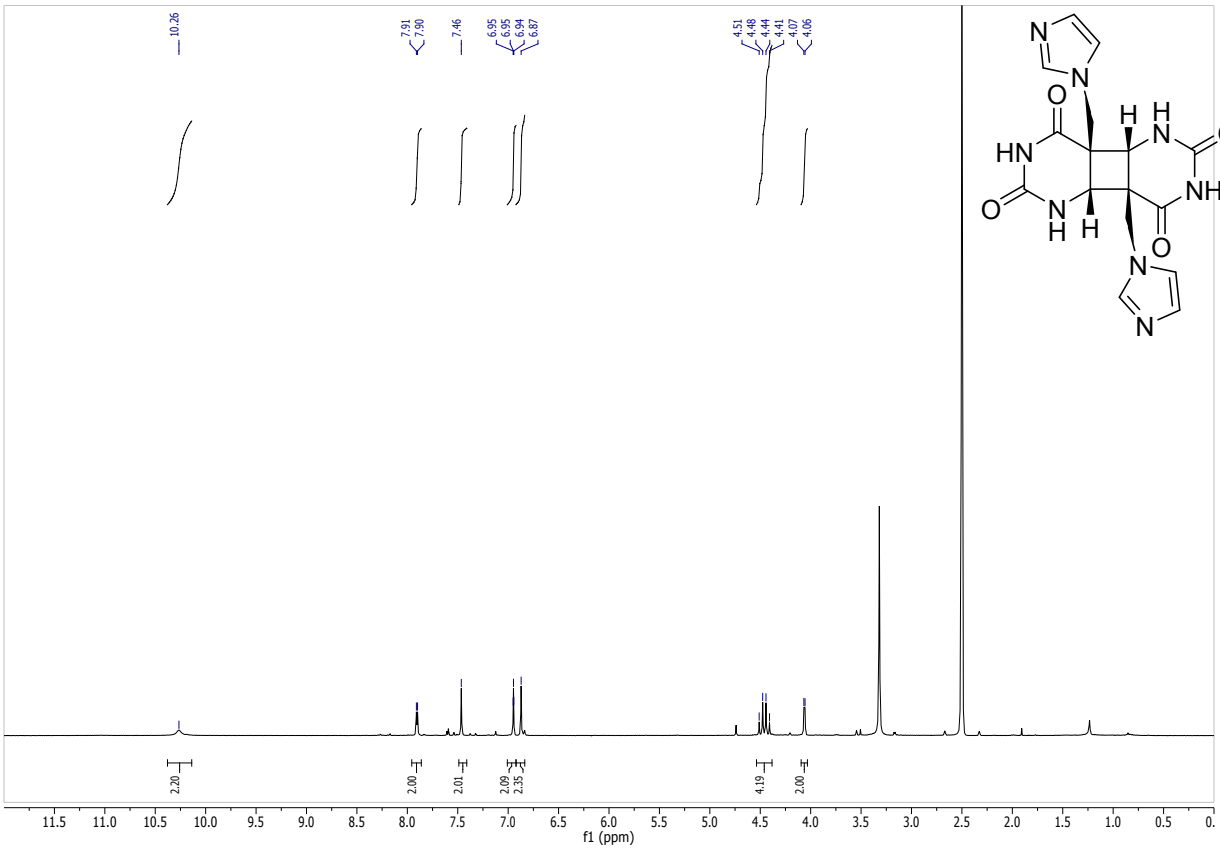


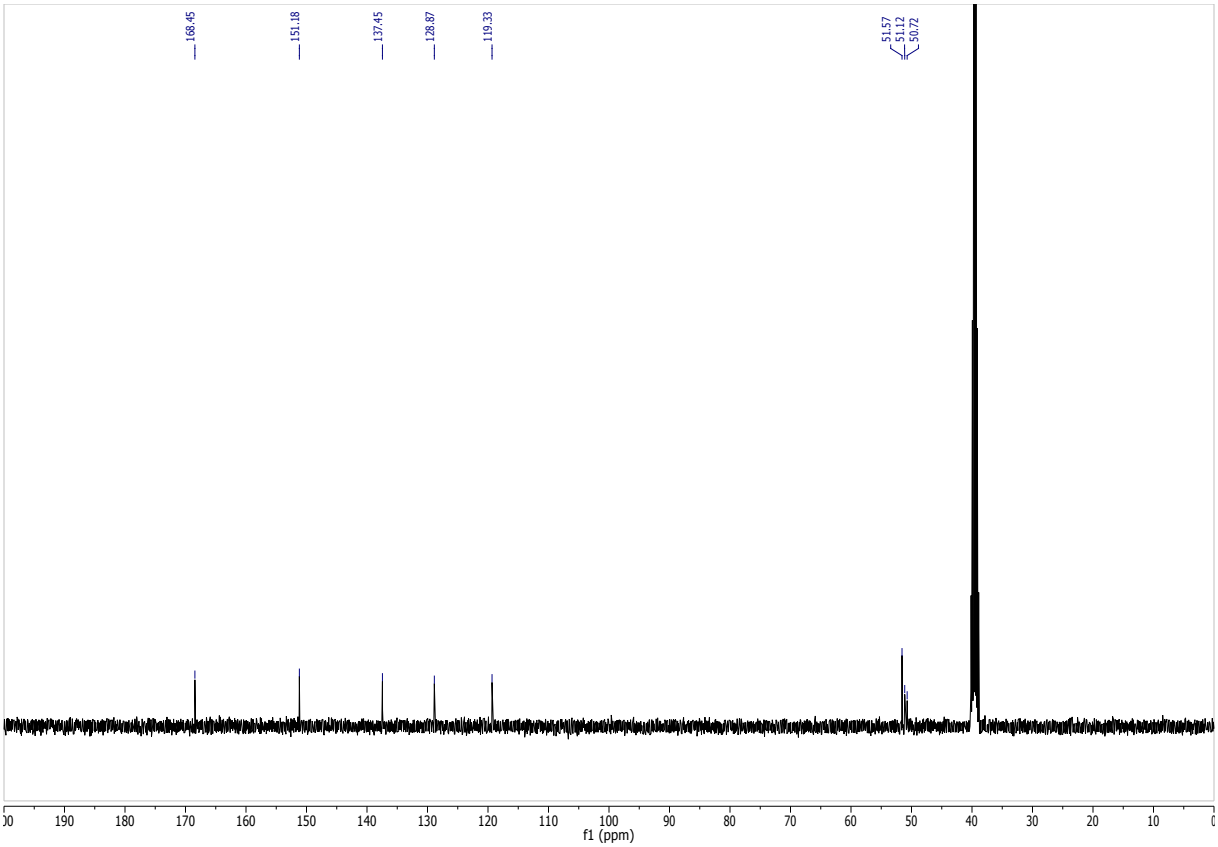
***cis*-4a,4b-bis((1*H*-imidazol-1-yl)methyl)hexahydro-cyclobuta[1,2-e:4,3-e']dipyrimidine-2,4,5,7(1*H*,3*H*,6*H*,8*H*)-tetraone 3a**





***cis*-4a,8a-bis((1*H*-imidazol-1-yl)methyl)hexahydro-cyclobuta[1,2-e:3,4-e']dipyrimidine-2,4,6,8(1*H*,3*H*,5*H*,7*H*)-tetraone 3b**





Crystallographic data

Suiteablecrystalls for X-ray analysis were obtained (a) for **3a** by slow evaporation in a mixture of DCM/MeOH 1:1 (v/v) (b) for **3b** by slow evaporation in a mixture of EtOH/H_2O 1:1 (v/v).

***cis-syn*Photodimer 3a**

Table 3 – Crystal data and structure refinement for photodimer**3a.**

Empiricalformula	$C_{17}H_{20}N_8O_5$
M_r	416.41 g(mol)$^{-1}$
T	100(2) K
λ	0.71073 Å
Crystal system	monoclinic
Space group	P 21/c
	a = 14.3939(17) Å α = 90°
	b = 9.0882(11) Å β = 107.205(4)°
	c = 14.4576(17) Å γ = 90°
V	1806.6(4) Å^3
Z	4
D_c	1.531 mg(m)$^{-3}$
μ	0.117 mm^{-1}
F(000)	872
Crystal size	0.247 x 0.121 x 0.110 mm
Θ range	1.481° bis 27.090°
Limitingindices	$-18 \leq h \leq 18$, $0 \leq k \leq 11$, $0 \leq l \leq 18$
Reflects. collect.	25242
IndepdntReflects	3979 R(int) = 0.0424
Completeness	99.8%
Absorp. Corr.	numerical
Restraints/parameters	0 / 280
GooF on F^2	1.068
R Index ($I > 2\sigma(I)$)	R1 = 0.0488, wR2 = 0.1046
R Index (all data)	R1 = 0.0766, wR2 = 0.1141
$\Delta\rho_{max,min}$	Max = 0.350 eÅ^{-3} / Min = -0.273 eÅ^{-3}

Table 4 - Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) forphotodimer**3a.**

	x	y	z	U(eq) $Å^2$
C(1)	8200(1)	1511(2)	9032(1)	10(1)
C(2)	7207(1)	2210(2)	8479(1)	12(1)
C(3)	6730(1)	1381(2)	9164(1)	12(1)
C(4)	7640(1)	398(2)	9521(1)	11(1)
C(5)	7508(1)	-1119(2)	9052(1)	12(1)
C(6)	5727(1)	-743(2)	8462(1)	13(1)
C(7)	8790(1)	832(2)	8424(1)	11(1)
C(8)	7441(1)	1339(2)	6939(1)	13(1)
C(9)	8857(1)	2608(2)	9742(1)	12(1)
C(10)	10177(1)	3650(2)	9138(1)	16(1)
C(11)	9473(2)	5482(2)	8294(2)	19(1)
C(12)	8809(2)	4884(2)	8676(2)	17(1)
C(13)	8057(1)	161(2)	10620(1)	13(1)
C(14)	6547(1)	-418(2)	11080(2)	17(1)
C(15)	6694(2)	-2733(2)	11341(2)	19(1)
C(16)	7512(2)	-2298(2)	11137(2)	19(1)
C(17)	5638(2)	5144(3)	9060(2)	26(1)
N(1)	6573(1)	-1559(2)	8595(1)	14(1)
N(2)	5814(1)	652(2)	8752(1)	13(1)
N(3)	8362(1)	835(2)	7438(1)	13(1)
N(4)	6859(1)	1755(2)	7478(1)	15(1)
N(5)	9265(1)	3695(2)	9218(1)	12(1)
N(6)	10336(1)	4714(2)	8581(1)	17(1)
N(7)	7414(1)	-804(2)	10967(1)	13(1)
N(8)	6087(1)	-1554(2)	11305(1)	19(1)
O(1)	8190(1)	-1941(2)	9118(1)	17(1)
O(2)	4927(1)	-1347(2)	8083(1)	17(1)
O(3)	9604(1)	361(2)	8783(1)	16(1)
O(4)	7171(1)	1342(2)	6050(1)	19(1)
O(5)	6203(1)	5426(2)	8412(1)	21(1)

***cis-anti*Photodimer3b**

Table 5 - Crystal data and structure refinement for photodimer**3b.**

Empiricalformula	$C_{16}H_{22}N_8O_7$
M_r	438.41 g(mol)$^{-1}$
T	100(2) K
λ	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
	a = 7.9316(10) Å α = 81.196(5)°
	b = 8.1627(10) Å β = 78.977(6)°
	c = 16.285(2) Å γ = 74.965(5)°
V	993.5(2) Å^3
Z	2
D_c	1.466 mg(m)$^{-3}$
μ	0.117 mm^{-1}
F(000)	460
Crystal size	0.320 x 0.040 x 0.010 mm
Θ range	1.282° bis 25.095°
Limitingindices	$-9 \leq h \leq 9$, $-9 \leq k \leq 9$, $-19 \leq l \leq 19$
Reflects. collect.	15153
IndepdntReflects	3523 R(int) = 0.0540
Completeness	99.2%
Absorp. Corr.	numerical
Restraints/parameters	2 / 322
GooF on F^2	1.015
R Index ($I > 2\sigma(I)$)	R1 = 0.0429, wR2 = 0.0984
R Index (all data)	R1 = 0.0627, wR2 = 0.1100
$\Delta\rho_{max,min}$	Max = 0.335 eÅ^{-3} / Min = -0.286 eÅ^{-3}

Table 1 - Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) forphotodimer**3b.**

	x	y	z	U(eq) $Å^2$
C(1)	6663(3)	637(2)	2589(1)	16(1)
C(2)	7067(3)	2152(2)	1955(1)	15(1)
C(3)	7846(3)	2684(2)	2669(1)	15(1)
C(4)	6806(3)	1598(2)	3333(1)	14(1)
C(5)	5060(3)	2658(2)	3729(1)	15(1)
C(6)	6109(3)	5309(3)	3227(1)	17(1)
C(7)	7750(3)	467(2)	4026(1)	16(1)
C(8)	9842(3)	2016(3)	4395(1)	18(1)
C(9)	9758(3)	2950(2)	5024(1)	18(1)
C(10)	7352(3)	2101(3)	5265(1)	18(1)
C(11)	5391(3)	3409(3)	1750(1)	18(1)
C(12)	3622(3)	1289(2)	2352(1)	15(1)
C(13)	8423(3)	1797(3)	1166(1)	22(1)
C(14)	7020(3)	1759(3)	-103(1)	32(1)
C(15)	6834(3)	474(4)	-493(1)	38(1)
C(16)	8148(3)	-727(3)	549(1)	30(1)
N(1)	4861(2)	4395(2)	3622(1)	17(1)
N(2)	7574(2)	4430(2)	2793(1)	18(1)
N(3)	8296(2)	1472(2)	4550(1)	15(1)
N(4)	8188(2)	3014(2)	5573(1)	20(1)
N(5)	3832(2)	2913(2)	2029(1)	17(1)
N(6)	5077(2)	150(2)	2549(1)	16(1)
N(7)	7863(2)	964(2)	570(1)	24(1)
N(8)	7552(3)	-1088(3)	-84(1)	36(1)
O(1)	3911(2)	2002(2)	4144(1)	20(1)
O(2)	5823(2)	6837(2)	3301(1)	23(1)
O(3)	5411(2)	4798(2)	1366(1)	30(1)
O(4)	2162(2)	971(2)	2426(1)	20(1)
O(10)	1139(2)	7908(2)	2675(1)	26(1)
O(11)	9372(2)	4520(2)	-1841(1)	26(1)
O(12)	8840(18)	5225(9)	-307(3)	33(2)
O(12A)	7773(17)	5706(9)	-172(3)	27(2)

Literature:

[1] A. J. Kleinsmann, N. M. Weckenmann, B. J. Nachtsheim, *Chem. Eur. J.,* **2014**, *20*, 9753 – 9761.

[2] J.Cai, X. Li, J. S. Taylor, *Org. Lett.* **2005**, *7,* 751–754.

[3] A. J. Kleinsmann, B. J. Nachtsheim, *Chem. Commun.* **2013**, *49,* 7818 – 7820.