Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Installing Tungsten Fischer Carbene Complexes into a Calixarene Framework

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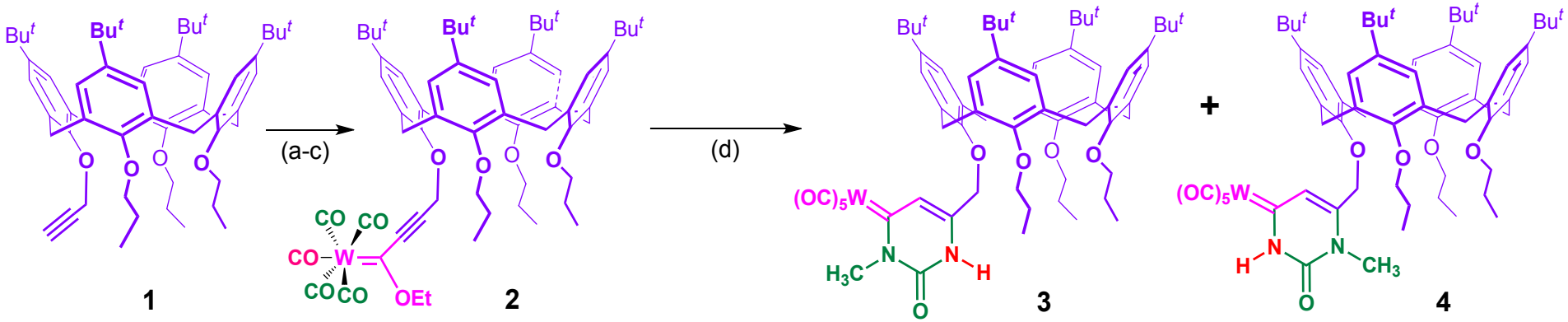
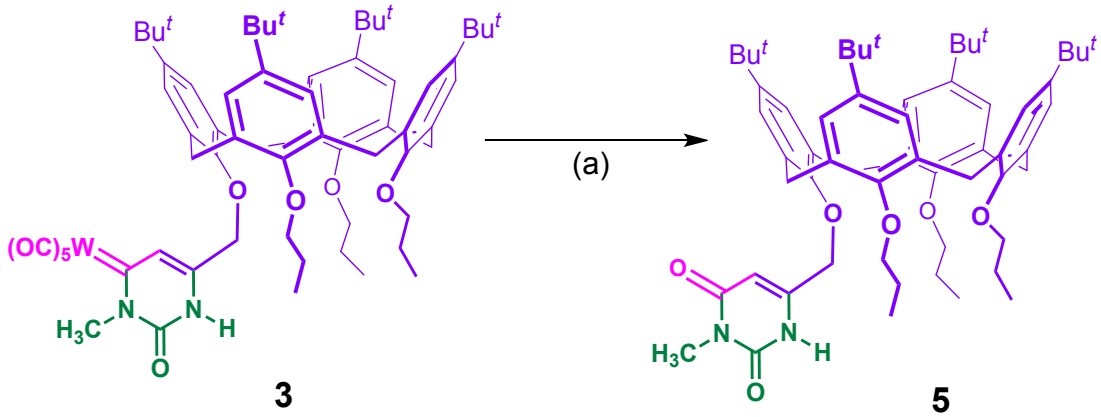
GENERAL COMMENTS

All reactions were carried out under a dry N_2 atmosphere using freshly distilled solvents, unless otherwise noted. Tetrahydrofuran (THF) was distilled from sodium/benzophenone complex. Glassware was flame-dried (0.05 Torr) prior to use. Starting materials and reagents purchased from commercial suppliers were generally used without purification. Derivative **1**[15] was synthesized according to literature procedures and was lyophilized under vacuum prior to use. Reaction temperatures were measured externally; reactions were monitored by thin layer chromatography on Merck silica gel plates (0.25 mm) and visualized by UV light and spraying with phosphomolybdic acid or $Ce(SO_4)_2$ solutions and drying. Flash chromatography was performed on silica gel (particle size: 40-63 μm). Yields refer to chromatographically and spectroscopically (^1H and ^{13}C NMR) pure materials. 1D and 2D NMR spectra were recorded on a Bruker Avance-600 spectrometer [600 (^1H) and 150 MHz (^{13}C)], Bruker Avance-400 spectrometer [400 (^1H) and 100 MHz (^{13}C)] and Bruker Avance-300 spectrometer [300 (^1H) and 75 MHz (^{13}C)]; chemical shifts are reported relative to the residual solvent peak ($CHCl_3$: δ 7.26, $CDCl_3$: δ 77.23). Standard pulse programs, provided by the manufacturer, were used for HMBC, HSQC and COSY experiments.

Synthesis of derivatives 2-5.

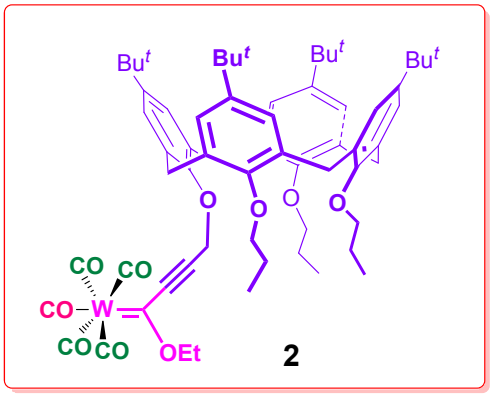


Scheme S1. Synthesis of derivatives **3** and **4**. Reagents and conditions: (a) n-BuLi, THF, from - 78 °C to -30 °C, 1h; (b) $W(CO)_6$, from -30 °C to -10 °C, 2h; (c) Et_3OBF_4, from -10 °C to -5 °C, 15 min; (d) methylurea, dry THF, 60 °C, 4 h.



Scheme S2. Synthesis of derivative **5**. Reagents and conditions: (a) ^{t}BuOOH, dry DCE, Ace Tube, 70°C, 2h.

Derivative 2



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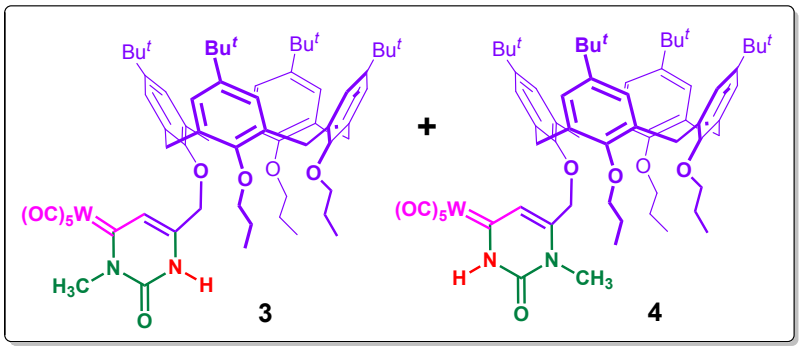
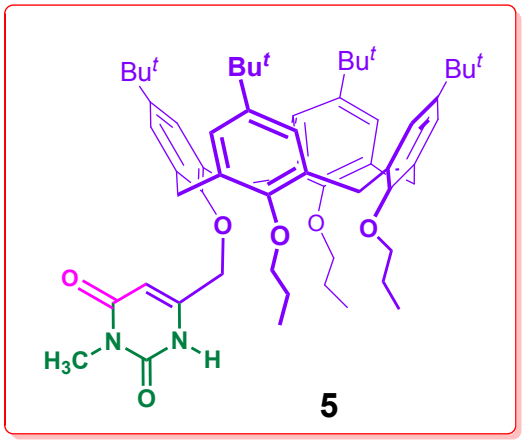
A stirred solution of derivative **1** (0.200 g, 0.250 mmol) in 2.00 mL of dry THF, under nitrogen, was cooled to -78 °C and then BuLi (2.5 M in hexane, 0.300 mmol) was added. The reaction mixture was stirred at -30 °C for 1 h. At this tempetarure $W(CO)_6$ (0.088 g, 0.250 mmol) was added and the mixture was warmed to -10 °C and left under stirring for 2 h.Then the alkylating agent $Et_3O^+BF_4^-$ (0.072 g, 0.380 mmol) was added and the mixture was warmed to -5 °C and at left at this temperature for 15 minutes. The mixture was quenched with satured $NaHCO_3$, and extracted with ethyl acetate. The organic layers collected were washed with brine, dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was subjected to flash chromatography on silica gel (from 100% to 70% of hexane in CH_2Cl_2), to give derivative **2** as a brown solid (0.060 g, 20%). **^{1}H NMR** (300 MHz, $CDCl_3$, 298 K): δ 0.84 (s, *t*-Bu, 18H), 1.02 (t, $OCH_2CH_2CH_3$, J = 7.4 Hz, 3H), 1.11 (t, $OCH_2CH_2CH_3$, J = 7.4 Hz, 6H), 1.35 (overlapped, OCH_2CH_3 + *t*-Bu, 12H), 1.37 (s, *t*-Bu, 9H), 1.93 (m, $OCH_2CH_2CH_3$, 4H), 2.13 (m, $OCH_2CH_2CH_3$, 2H), 3.17 and 4.28 (AX, $ArCH_2Ar$, J = 13.2 Hz, 2H each), 3.19 and 4.47 (AX, $ArCH_2Ar$, J = 12.6 Hz, 2H each), 3.62-3.77 (m, $OCH_2CH_2CH_3$, 4H), 3.99-4.02 (overlapped, $OCH_2CH_2CH_3$ + OCH_2CH_3, 4H), 5.89 (s, $OCH_2C{\equiv}C$-, 2H), 6.37 (AB, ArH, J = 2.4 Hz, 2H), 6.56 (AB, ArH, J = 2.4 Hz, 2H), 7.09 (s, ArH, 2H), 7.17 (s, ArH, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$, 298 K): δ 10.1, 11.0, 15.0, 23.8, 23.9, 29.8, 30.9, 31.3, 31.8, 31.9, 32.5, 33.8, 34.2, 34.4, 59.0, 77.8, 124.3, 124.7, 125.4, 125.8, 131.8, 131.9, 135.7, 137.1, 144.5, 145.1, 146.3, 151.7, 153.1, 154.7, 197.3, 197.7, 198.1, 205.9, 286.5. Mp: 230-231°C, Anal. Calcd for $C_{64}H_{80}O_{10}W$: C, 64.42; H, 6.76. Found: C, 64.53; H, 6.85.

Derivatives 3 and 4



A solution of derivative **4** (0.097 g, 0.081 mmol) and methyl urea (0.012 g, 0.016 mmol) in 0.10 mL of dry THF, under nitrogen, was placed in a ACE tube equipped with a magnetic bar and warmed to 60 °C. The reaction was monitored until the starting material disappears. The mixture was concentrated under *vacuo* and the crude product was subjected to flash chromatography on silica gel (100% to 60% of hexane in CH_2Cl_2), to give derivatives **3** (0.050 g, 50%) and **4** (0.006 g, 6%). Derivative **3**. **^{1}H NMR** (600 MHz, $CDCl_3$, 298 K): δ 0.76 (t, $OCH_2CH_2CH_3$, J = 7.2 Hz, 3H), 0.86 (s, *t*-Bu, 18H), 0.94 (t, $OCH_2CH_2CH_3$, J = 7.8 Hz, 6H), 1.38 (s, *t*-Bu, 18H), 1.74-1.83 (overlapped, $OCH_2CH_2CH_3$, 6H), 3.18 and 4.46 (AX, $ArCH_2Ar$, J = 12.6 Hz, 2H each), 3.32 and 4.30 (AX, $ArCH_2Ar$, J = 13.2 Hz, 2H each), 3.77 (m, $OCH_2CH_2CH_3$, 2H), 3.87 (m, $OCH_2CH_2CH_3$, 2H), 4.05 (m, $OCH_2CH_2CH_3$, 2H), 4.22 (s, NCH_3, 3H), 5.02 (s, OCH_2, 2H), 6.50 (AB, ArH, J = 1.8 Hz, 2H), 6.56 (AB, ArH, J = 1.2 Hz, 2H), 7.15 (s, ArH, 2H), 7.20 (s, ArH, 2H), 7.36 (s, OCH_2C=C*H*-, 1H), 11.99 (s, NH, 1H). **^{13}C NMR** (150 MHz, $CDCl_3$, 298 K): δ 9.8, 10.3, 22.3, 22.9, 29.8, 31.2, 31.5, 31.6, 31.7, 31.9, 33.8, 34.2, 34.3, 47.1, 70.5, 76.2, 77.4, 78.0, 121.4, 124.4, 125.4, 125.6, 126.5, 131.3, 132.5, 134.7, 135.7, 144.3, 144.9, 145.2, 147.1, 148.8, 151.3, 152.8,153.6, 198.8, 203.6, 240.4. Mp: 225-226 °C. Anal. Calcd for $C_{64}H_{80}N_2O_{10}W$: C, 62.69; H, 6.99. Found: C, 62.79; H, 6.89. Derivative **4**. **^{1}H NMR** (600 MHz, $CDCl_3$, 298 K): δ 0.92 (overlapped, $OCH_2CH_2CH_3$, J = 7.2 Hz, 6H), 1.05-1.09 (overlapped, *t*-Bu, 36H + $OCH_2CH_2CH_3$, 3H), 1.90 (m, $OCH_2CH_2CH_3$, 6H), 3.14 and 4.14 (AX, $ArCH_2Ar$, J = 12.6 Hz, 2H each), 3.15 and 4.40 (AX, $ArCH_2Ar$, J = 13.2 Hz, 2H each), 3.71-3.88 (overlapped, $OCH_2CH_2CH_3$, 6H + NCH_3, 3H), 4.81 (s, OCH_2, 2H), 6.72 (s, OCH_2C=C*H*, 1H), 6.73-6.81 (overlapped, ArH, 8H), 10.37 (s, NH, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$, 298 K): δ 9.9, 10.4, 14.3, 22.9, 23.5, 27.6, 29.4, 29.9, 31.3, 31.9, 34.0, 37.3, 37.8, 45.7, 72.4, 77.3, 124.8, 125.2, 125.6, 126.1, 126.8, 133.0, 133.5, 134.0, 145.1, 148.8, 151.8, 153.8, 198.2, 203.6, 236.4. Mp: 222-223°C. Anal. Calcd for $C_{64}H_{80}N_2O_{10}W$: C, 62.69; H, 6.99. Found: C, 62.80; H, 6.88.

Derivative 5



In a screw capped ACE tube containing a solution of derivative **3** (0.025, 0.020 mmol) in 1.00 mL of dry 1,2-dichloroethane a solution of TBHP (5.5 M in *n*-decane, 0.200 mmol) was added. The mixture was warmed at 70 °C until completion of oxidation. Decoloration of solution and formation of a white precipitate was indicative of transformation. Sodium sulfite (10%) aqueous solution (6.00 mL) was added and the resulting mixture was stirred for 3 h then extracted with dichloromethane. The organic layers were dried over sodium sulfate, filtered and concentrated under reduced pressure. The crude product was subjected to flash chromatography on silica gel (100% to 60% of hexane in ethyl acetate) to give derivative **5** as a pale oil **(**0.017 g, 93%). **^{1}H NMR** (600 MHz, $CDCl_3$, 298 K): δ 0.76 (t, $OCH_2CH_2CH_3$, J = 7.2 Hz, 3H), 0.82 (s, *t*-Bu, 18H), 0.92 (t, $OCH_2CH_2CH_3$, J = 7.8 Hz, 6H), 1.33 (s, *t*-Bu, 18H), 1.75 (m, $OCH_2CH_2CH_3$, 4H), 1.84 (m, $OCH_2CH_2CH_3$, 2H), 3.13 and 4.43 (AX, $ArCH_2Ar$, J = 12.6 Hz, 2H each), 3.24 and 4.28 (AX, $ArCH_2Ar$, J = 12.6 Hz, 2H each), 3.40 (s, NCH_3, 3H), 3.75 (m, $OCH_2CH_2CH_3$, 2H), 3.89 (m, $OCH_2CH_2CH_3$, 2H), 4.02 (m, $OCH_2CH_2CH_3$, 2H), 4.82(s, OCH_2Hetcycl, 2H), 5.63 (s, OCH_2CH Hetcycl, 1H), 6.44 (AB, ArH, J = 1.8 Hz, 2H), 6.50 (s, ArH,2H), 7.10 (s, ArH, 2H), 7.14 (s, ArH, 2H), 10.77 (s, NH, 1H).**^{13}C NMR** (150 MHz, $CDCl_3$, 298 K): δ 9.9, 10.4, 14.3, 22.5, 23.0, 27.4, 29.9, 31.3, 31.6, 31.8, 32.0, 33.9, 34.2, 34.4, 53.6, 71.9, 76.3, 78.0, 98.1, 124.4, 125.4, 125.8, 126.6, 131.5, 132.6, 135.2, 136.0, 144.7, 145.2, 146.7, 150.4, 151.8, 152.2, 153.0, 154.0, 164.2. Anal. Calcd for $C_{59}H_{80}N_2O_6$: C, 77.59; H, 8.83. Found: C, 77.69; H, 8.74.

^{1}H and ^{13}C NMR spectra of derivative 2

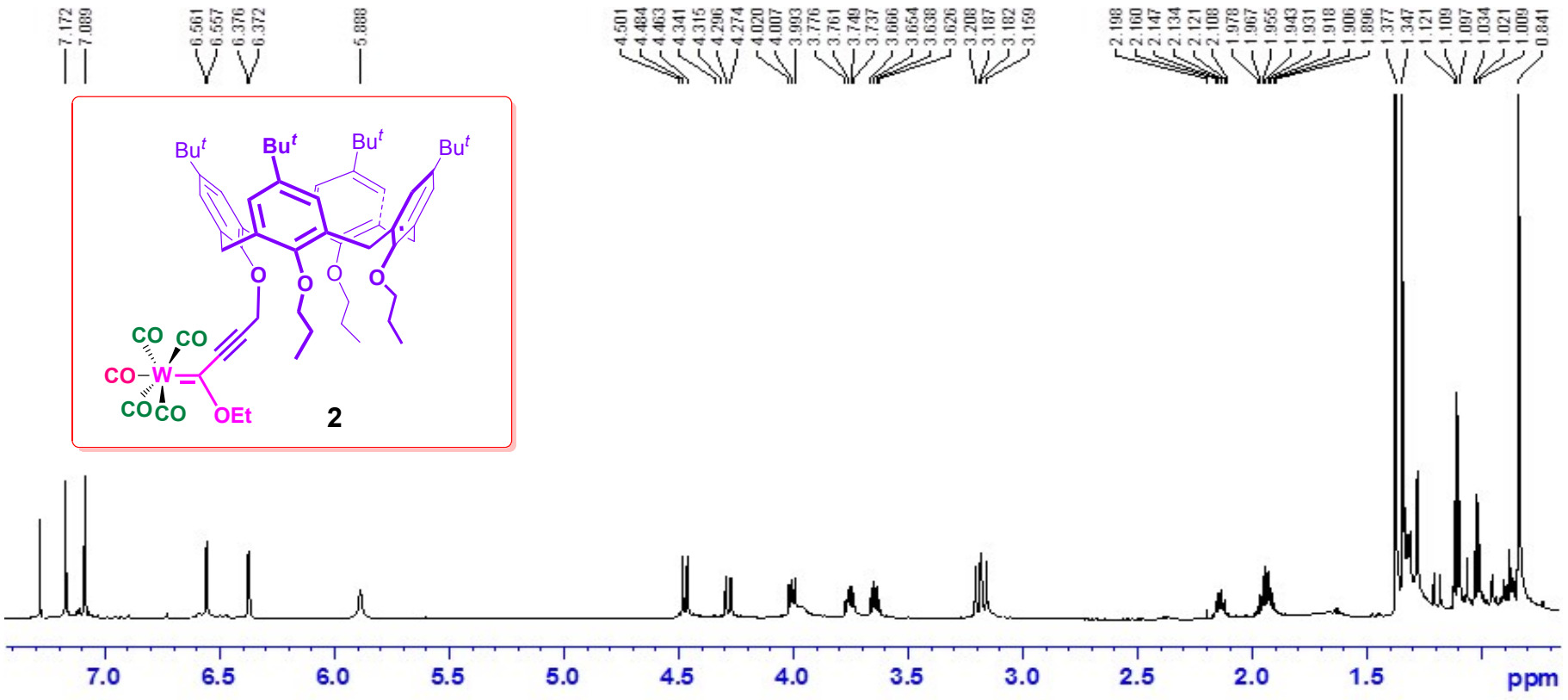


Figure S1. ^{1}H NMR of derivative **2** (300 MHz, $CDCl_3$, 298 K).

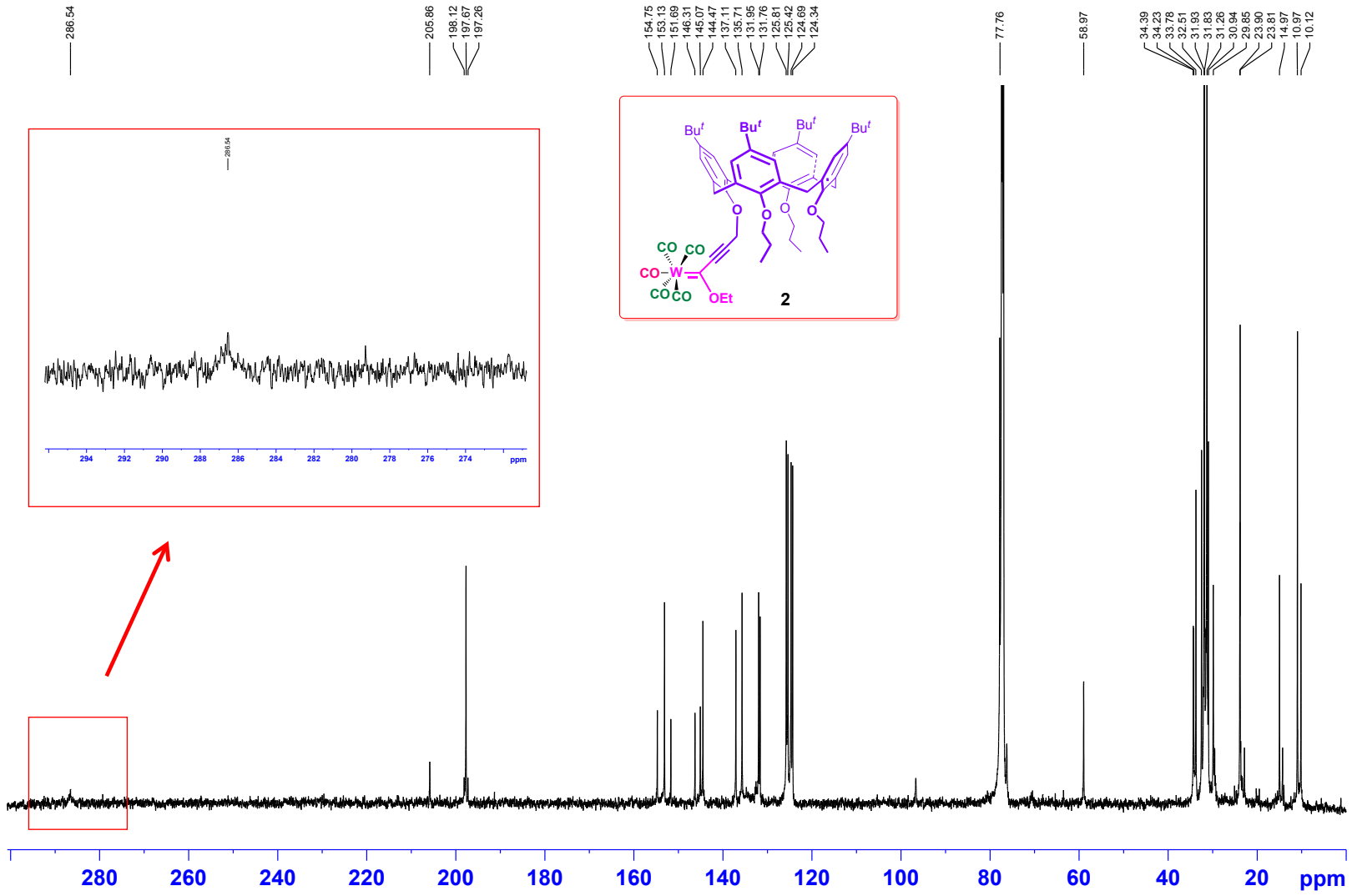


Figure S2. ^{13}C NMR of derivative **2** (150 MHz, $CDCl_3$, 298 K).

2D NMR Spectra of derivative 2

2D COSY-45 Spectrum of derivative **2**

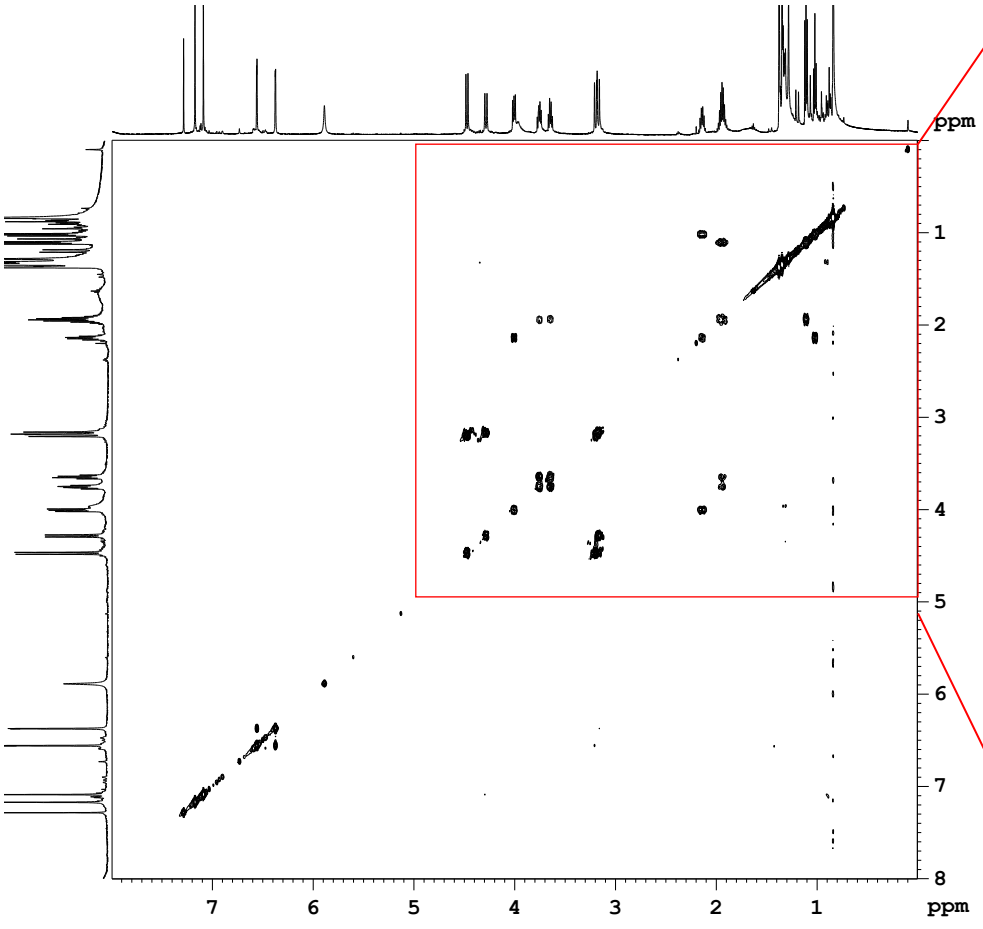


Figure S3. 2D COSY-45 spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **2**.

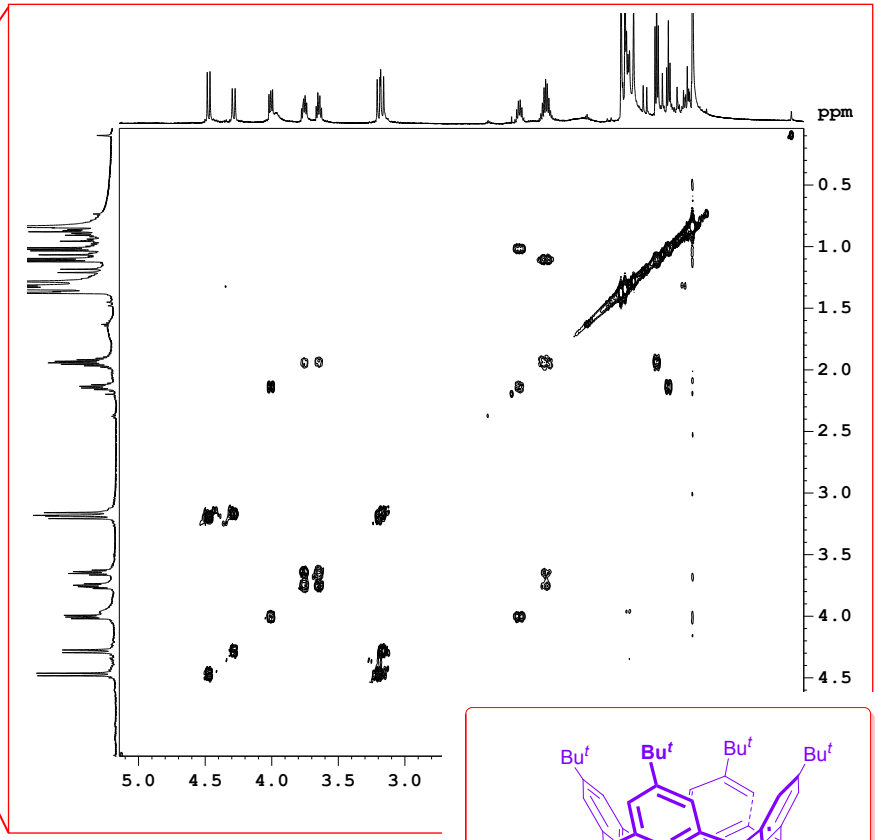
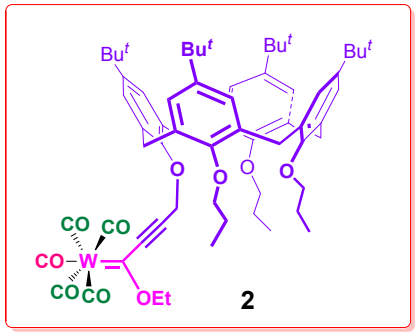


Figure S4. Portion of 2D COSY-45 spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **2**.



2D HSQC Spectrum of derivative **2**

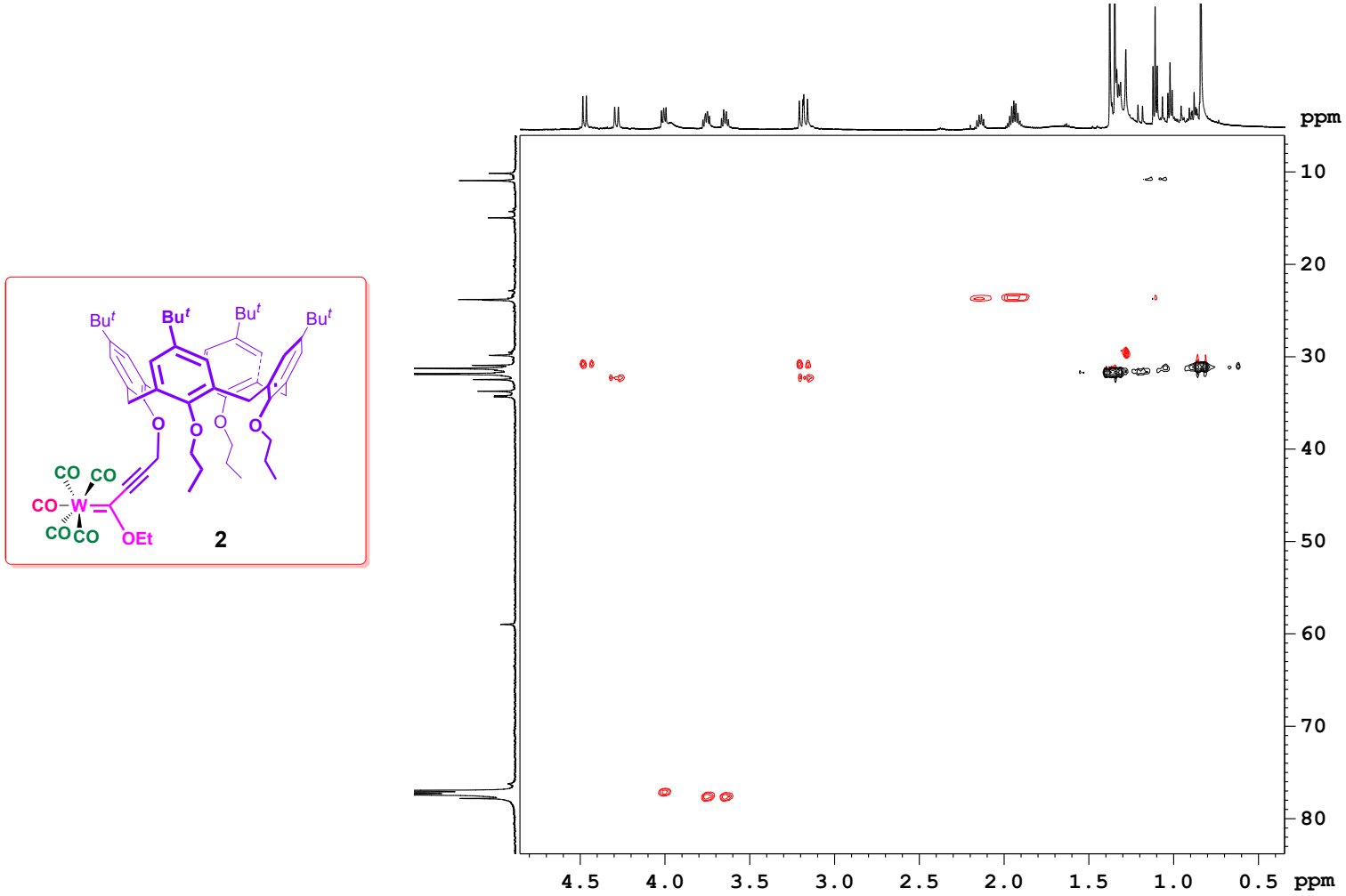


Figure S5. 2D HSQC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **2**

^{1}H and ^{13}C NMR Spectra of derivative 3

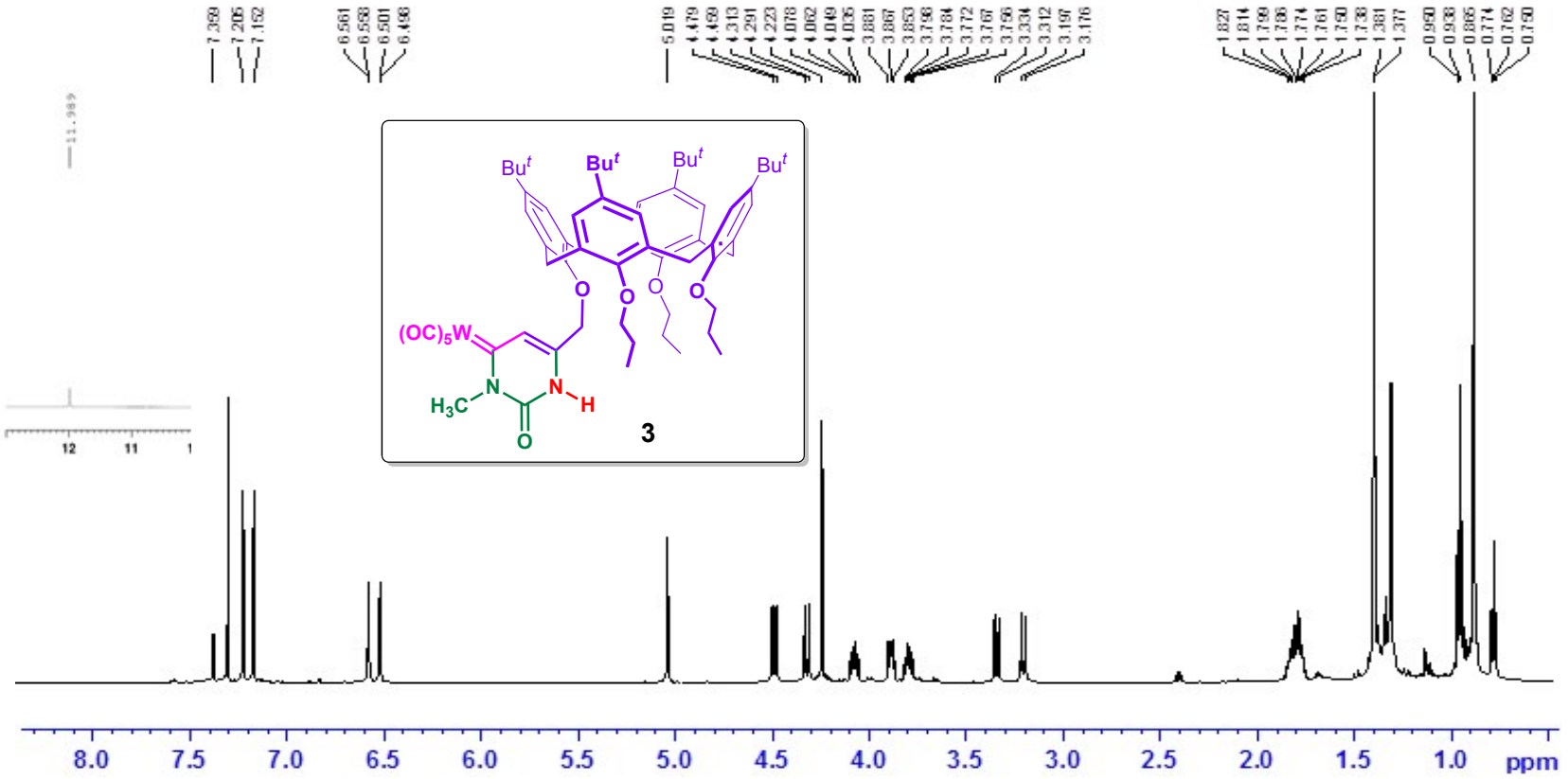


Figure S6. ^{1}H NMR of derivative **3** (600 MHz, $CDCl_3$, 298 K).

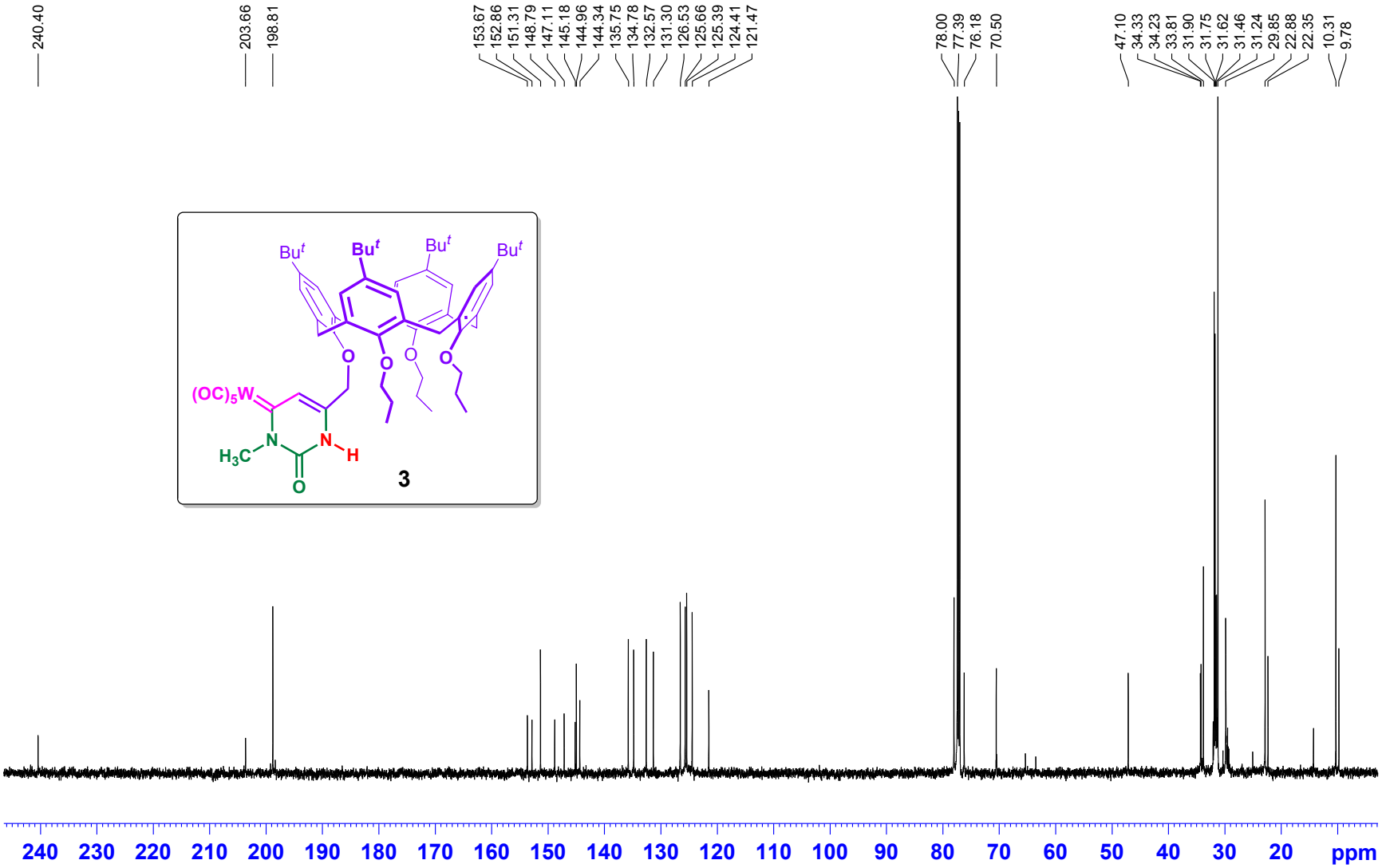
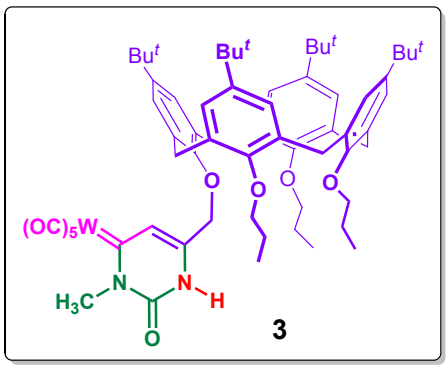


Figure S7. ^{13}C NMR of derivative **3** (150 MHz, $CDCl_3$, 298 K).

2D NMR Spectra of derivative 3

2D COSY Spectrum of derivative **3**



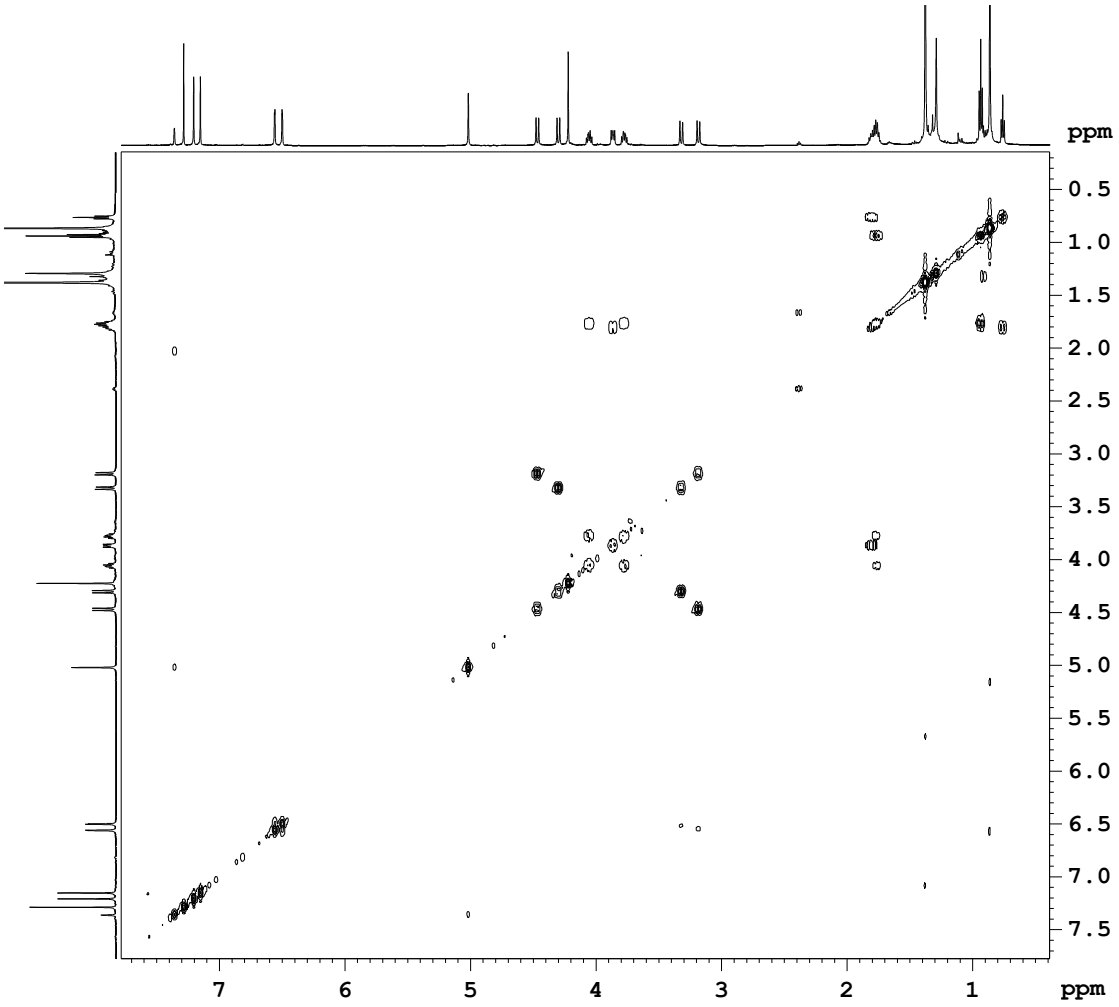


Figure S8. Portion of 2D COSY spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **3**.

HSQC Spectrum of derivative **3**

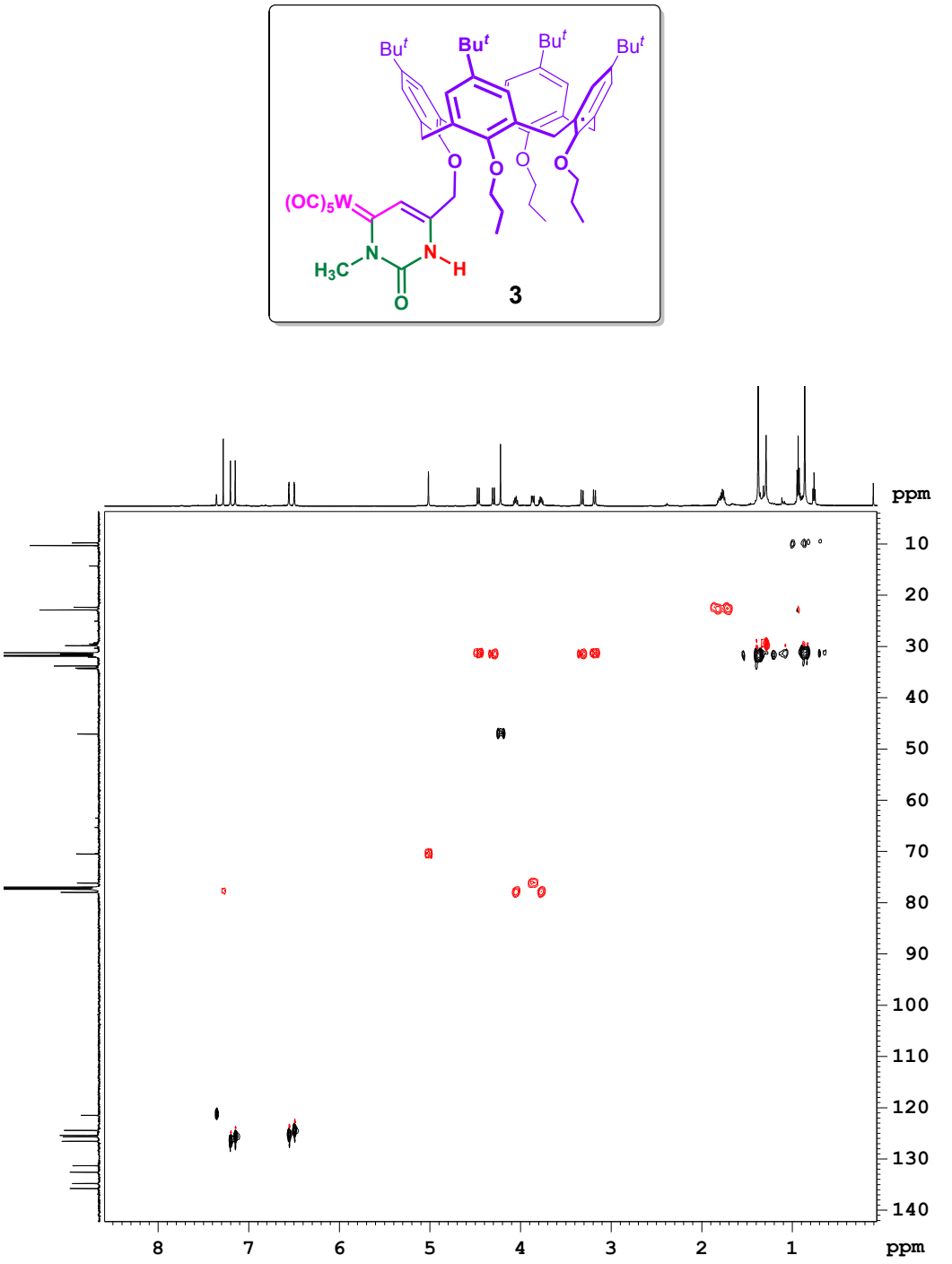
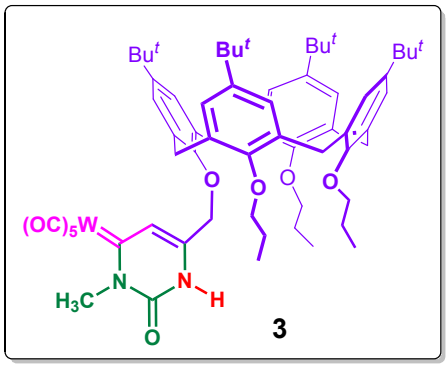


Figure S9. Portion of HSQC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **3**.

HMBC Spectrum of derivative **3**



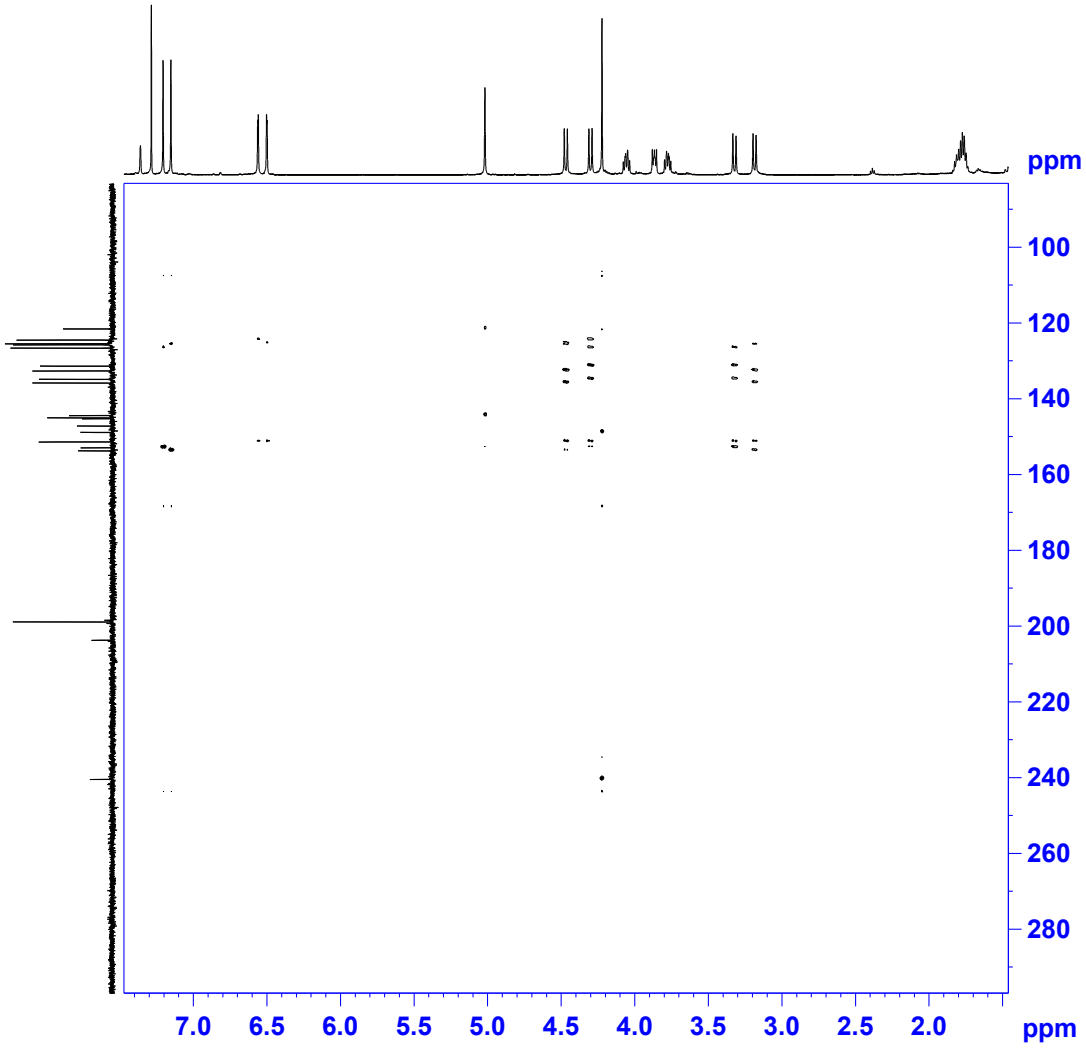


Figure S10. Portion of HMBC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **3**.

^{1}H and ^{13}C NMR Spectra of derivative 4

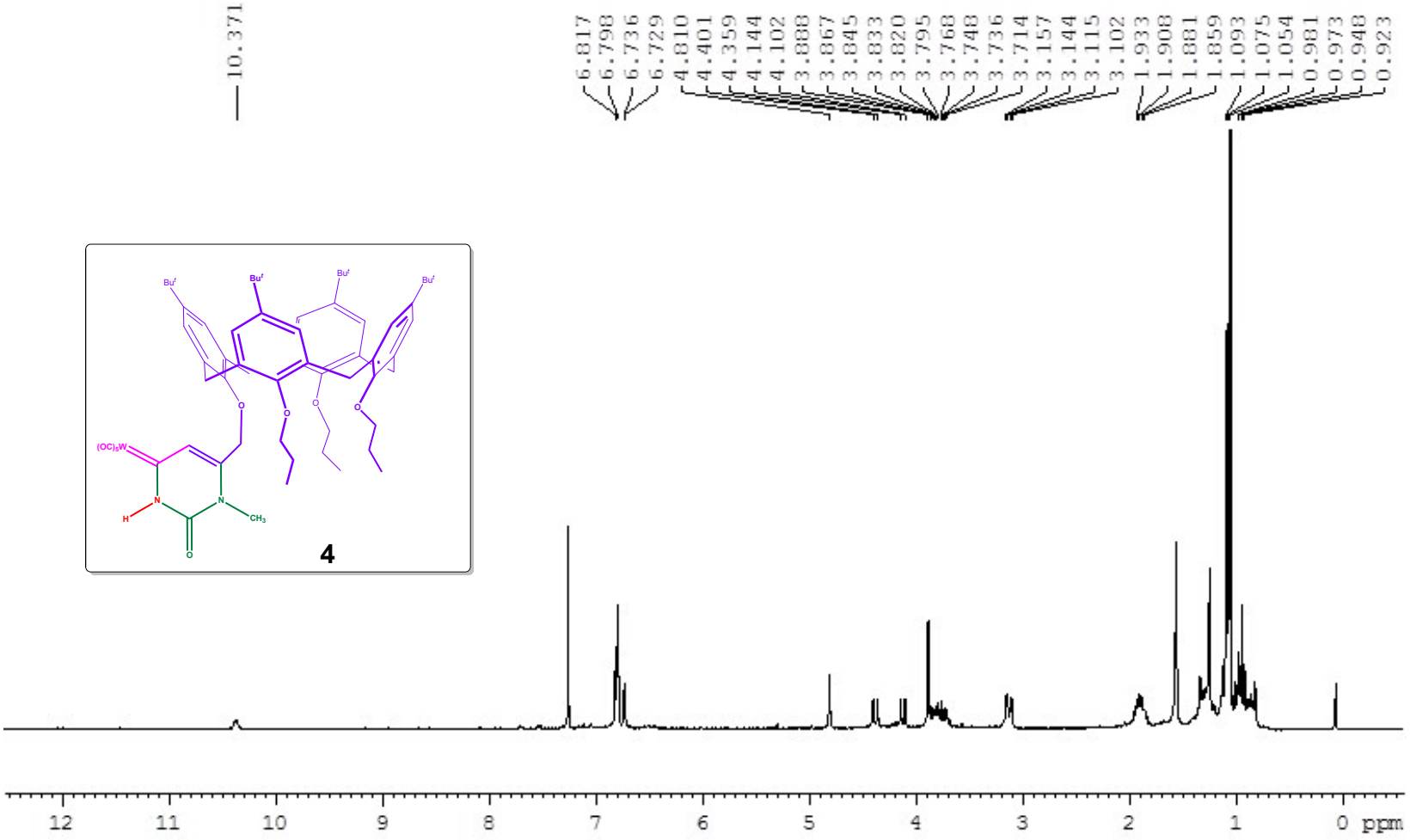


Figure S11. ^{1}H NMR of derivative **4** (300 MHz, $CDCl_3$, 298 K).

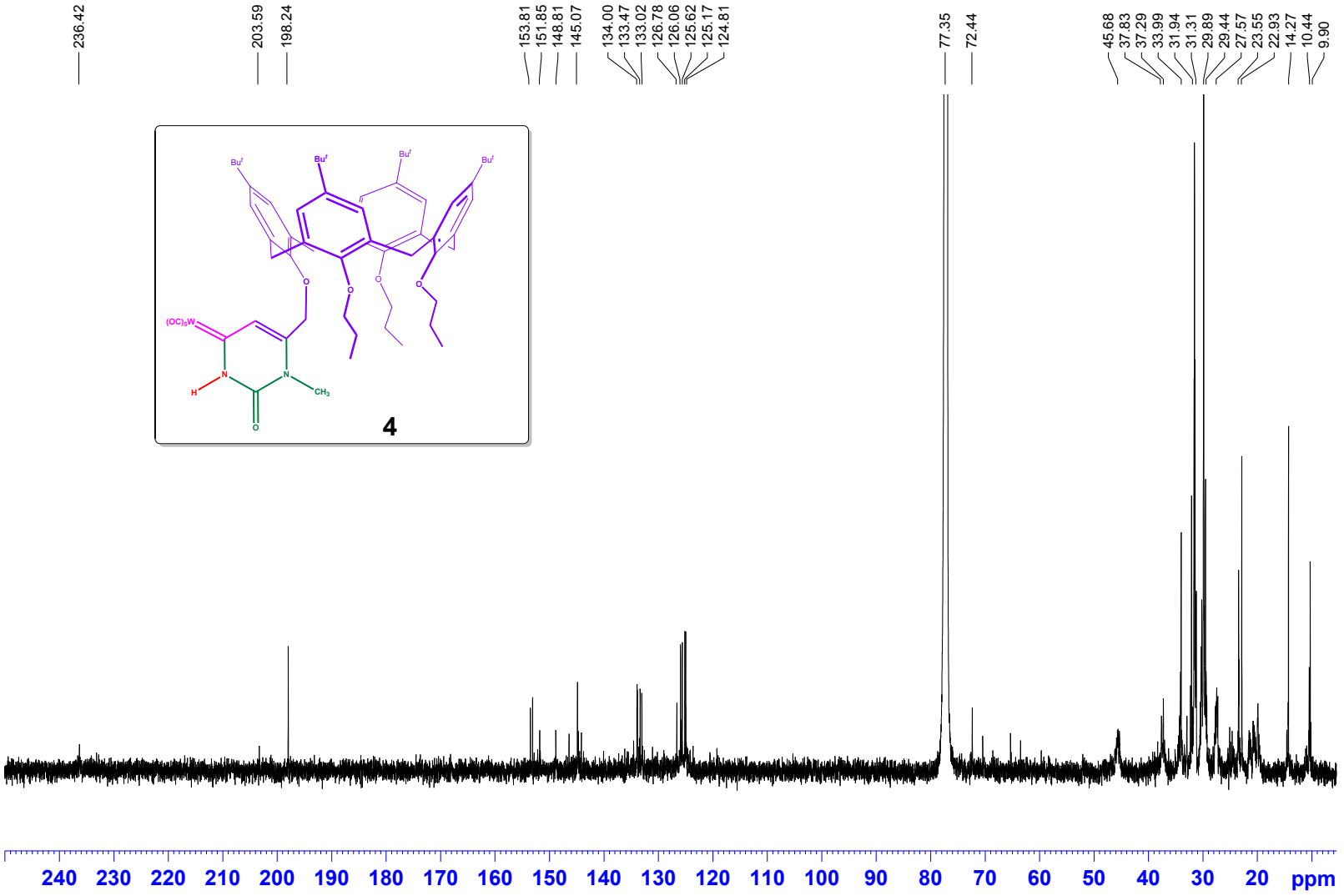


Figure S12. ^{13}C NMR spectrum of derivative **4** (600 MHz, $CDCl_3$, 298 K).

2D NMR Spectra of derivative 4

2D COSY Spectrum of derivative **4**

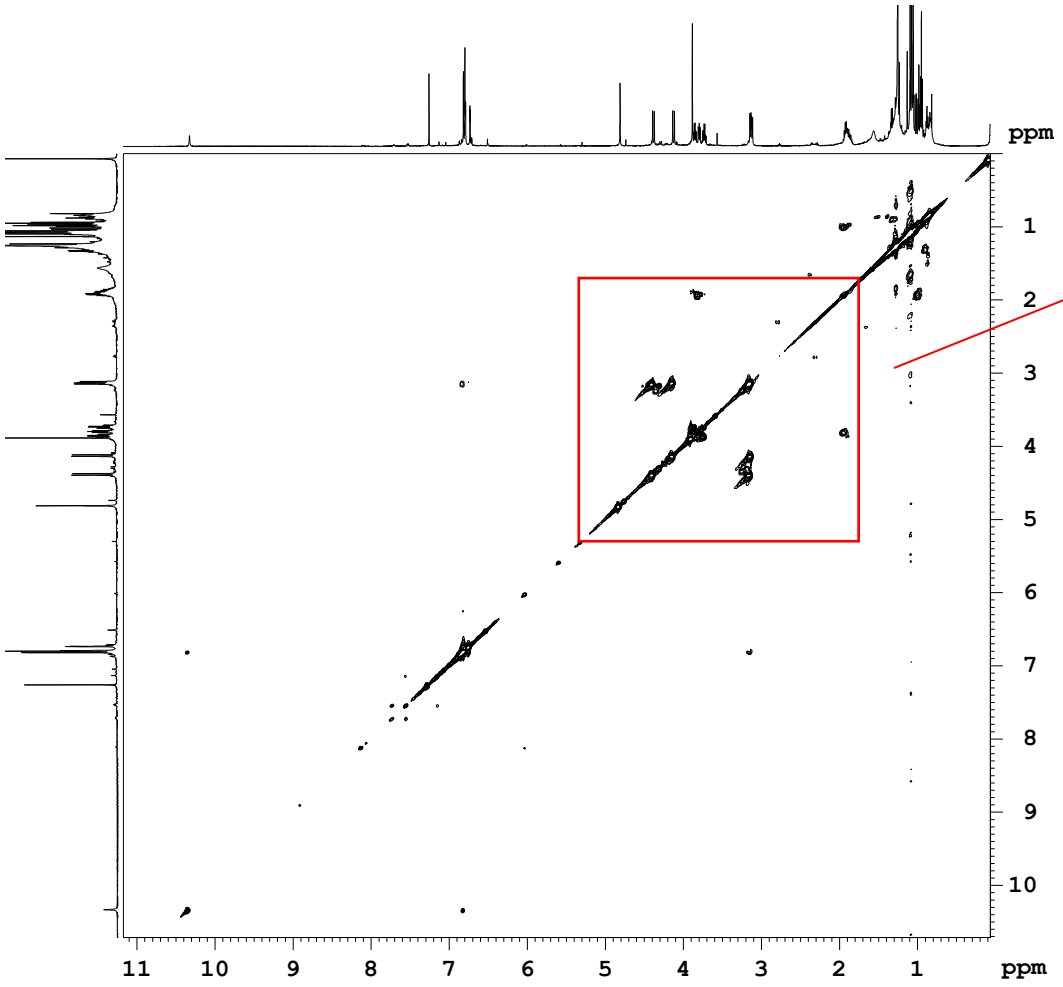


Figure S13. 2D COSY spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **4**.

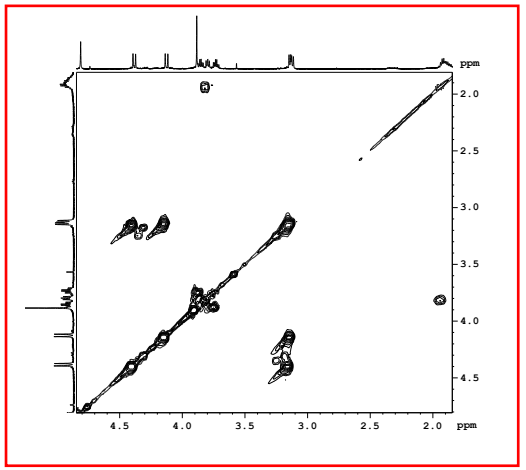
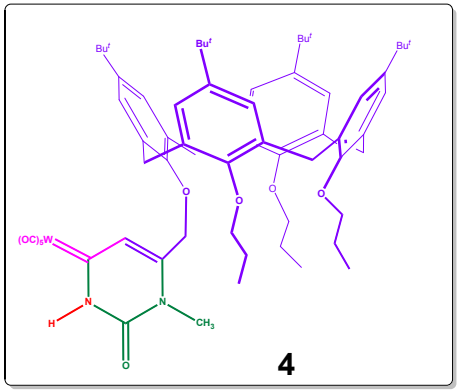


Figure S14. Portion of 2D COSY spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **4**.



HSQC Spectrum of derivative **4**

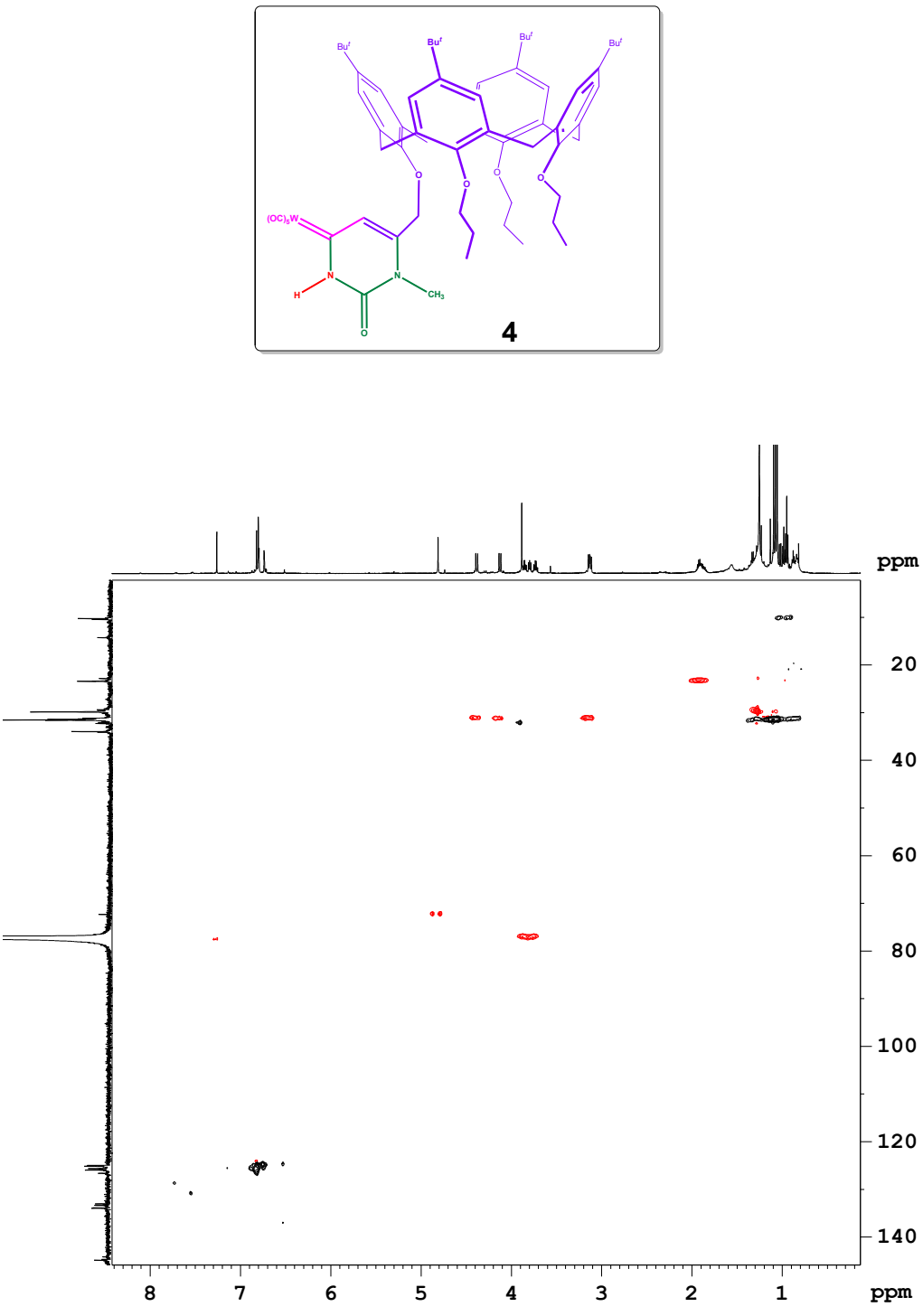


Figure S15. HSQC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **4**

HMBC Spectrum of derivative **4**

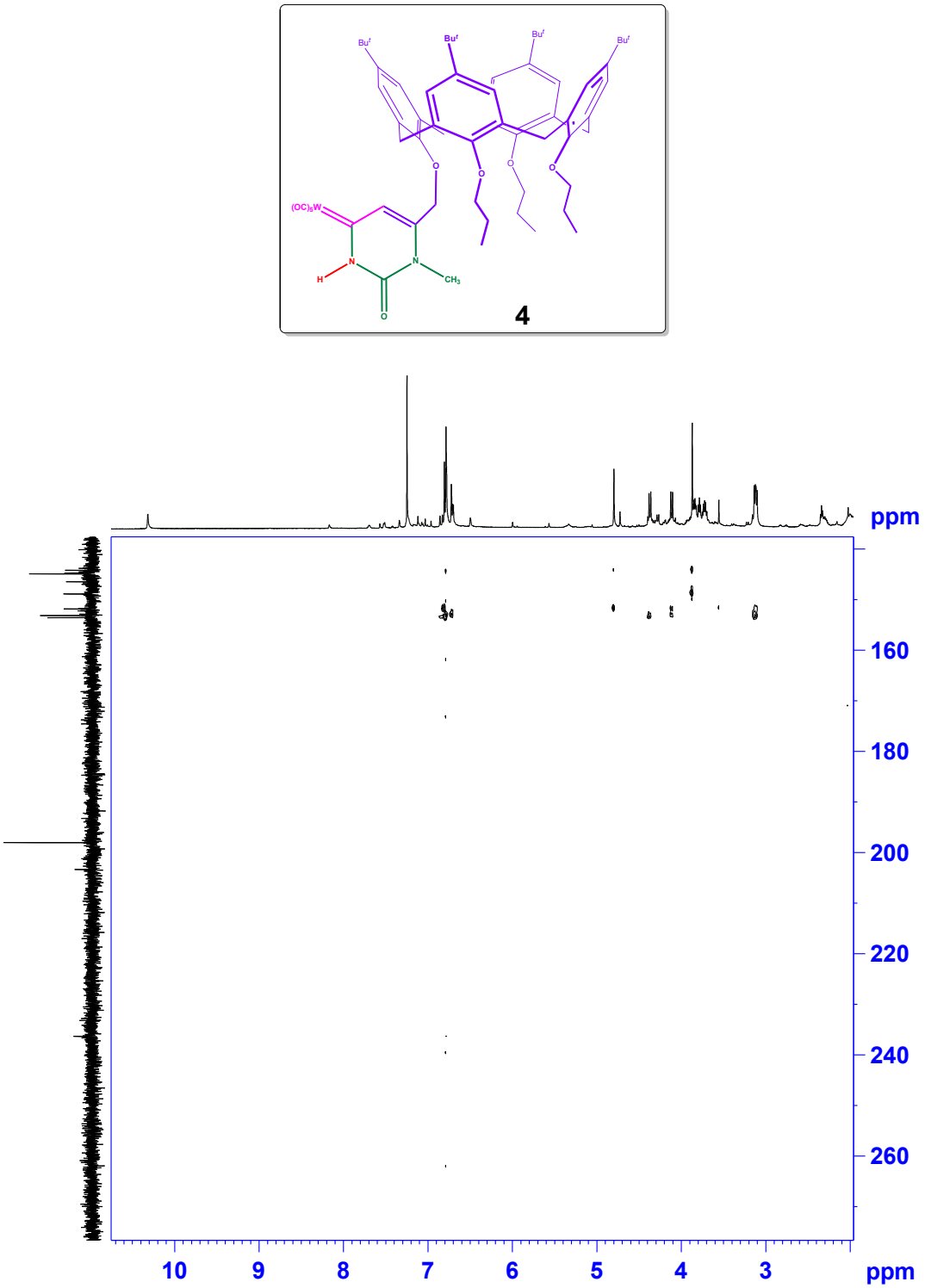


Figure S16. Portion of HMBC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **4**.

^{1}H and ^{13}C NMR spectra of derivative 5

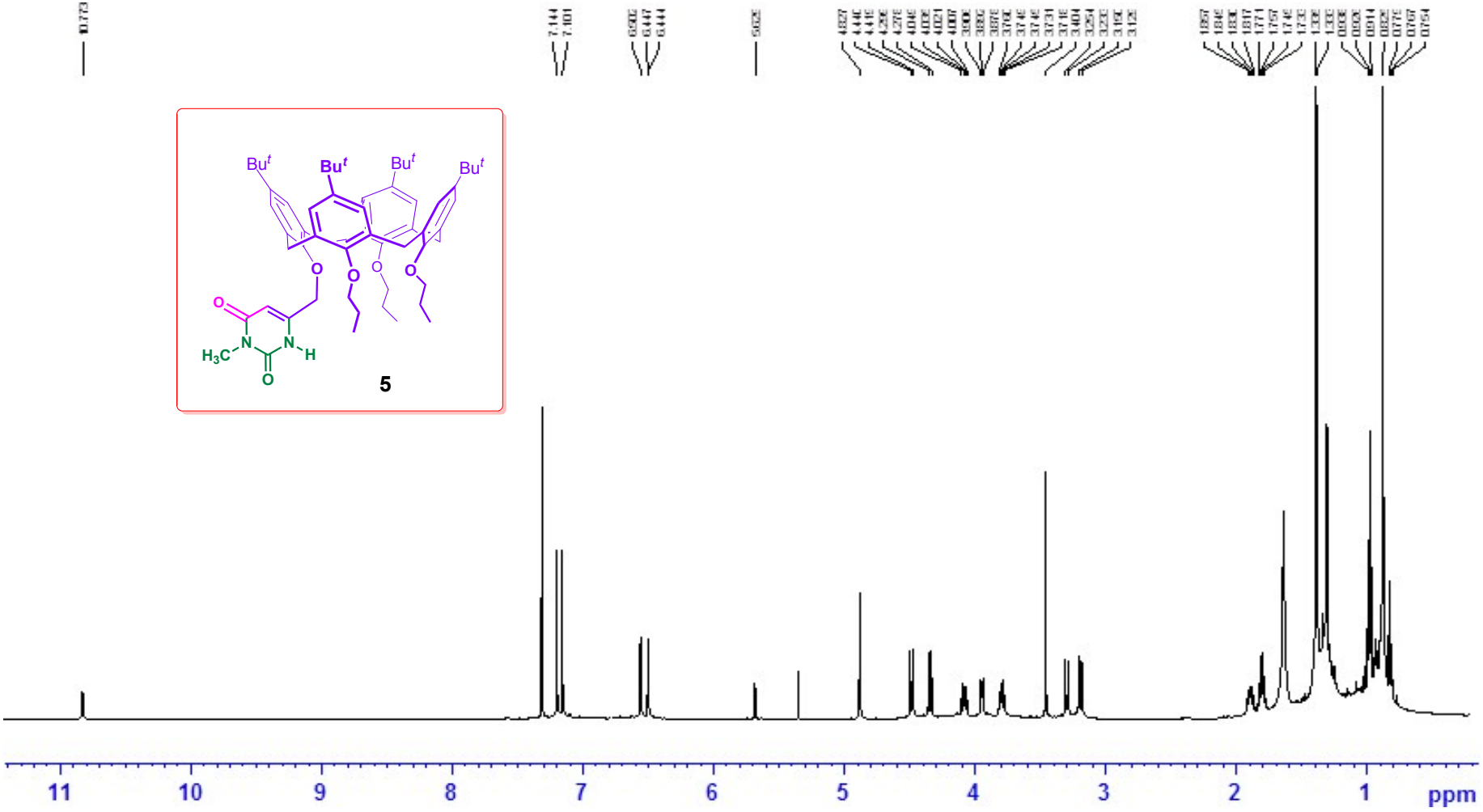


Figure S17. ^{1}H NMR of derivative **5** (600 MHz, $CDCl_3$, 298 K).

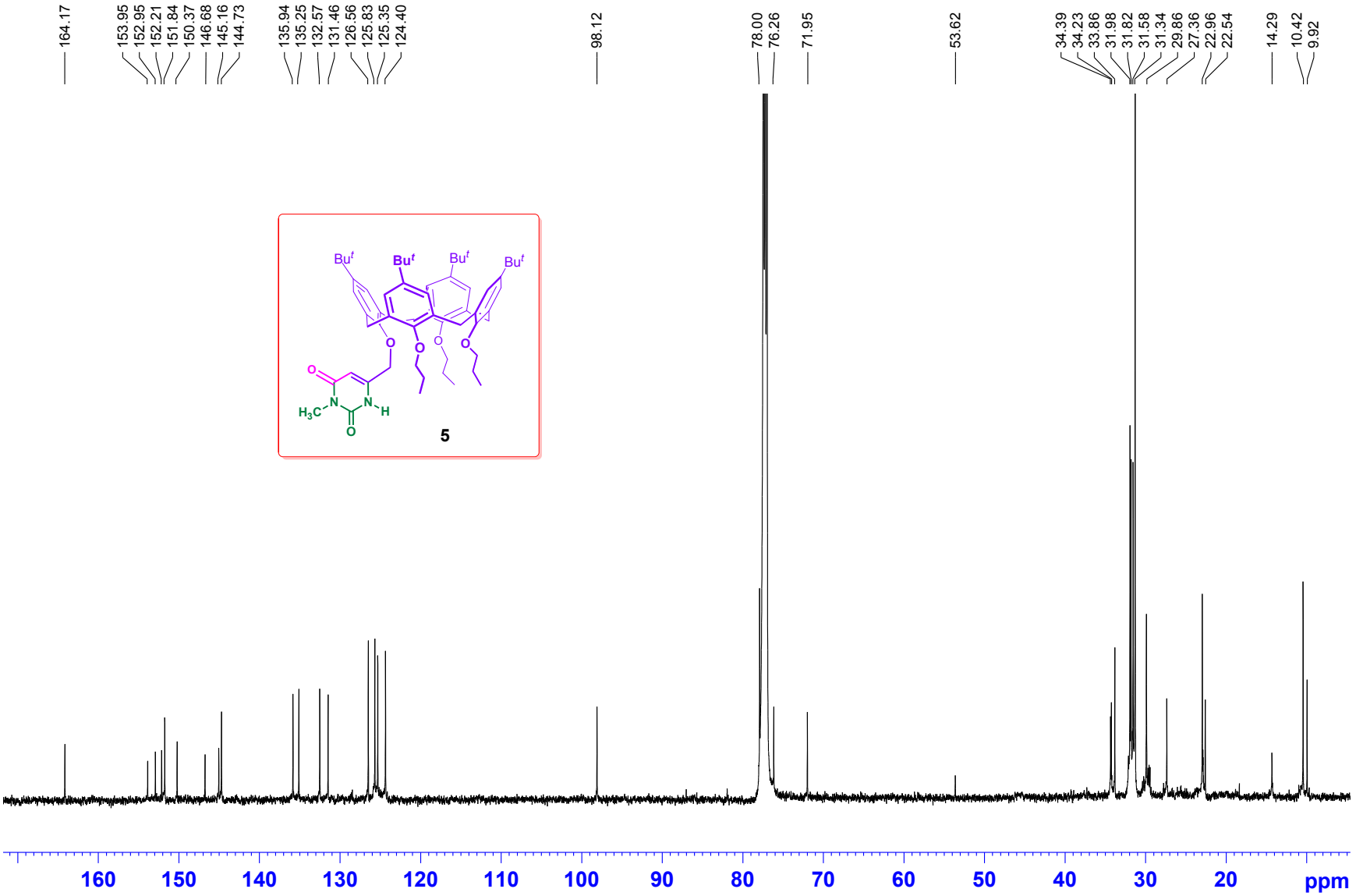
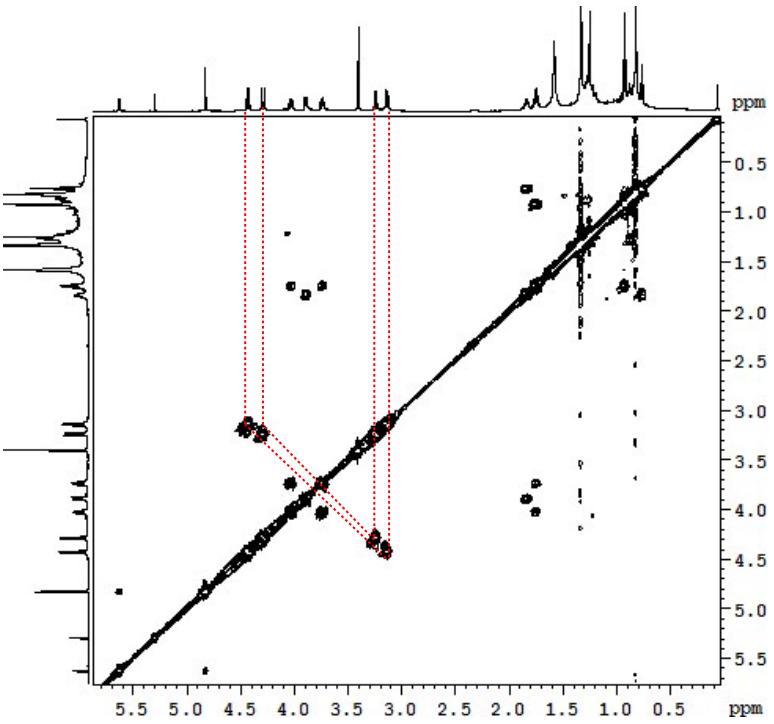


Figure S18. ^{13}C NMR of derivative **5** (150 MHz, $CDCl_3$, 298 K).

2D NMR Spectra of derivative 5

2D COSY Spectrum of derivative **5**



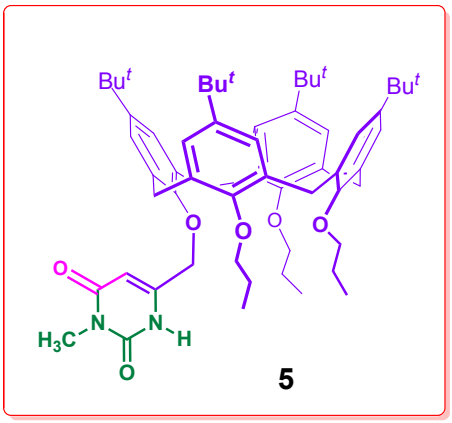


Figure S19. Portion of 2D COSY spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **5**.

HSQC Spectrum of derivative **5**

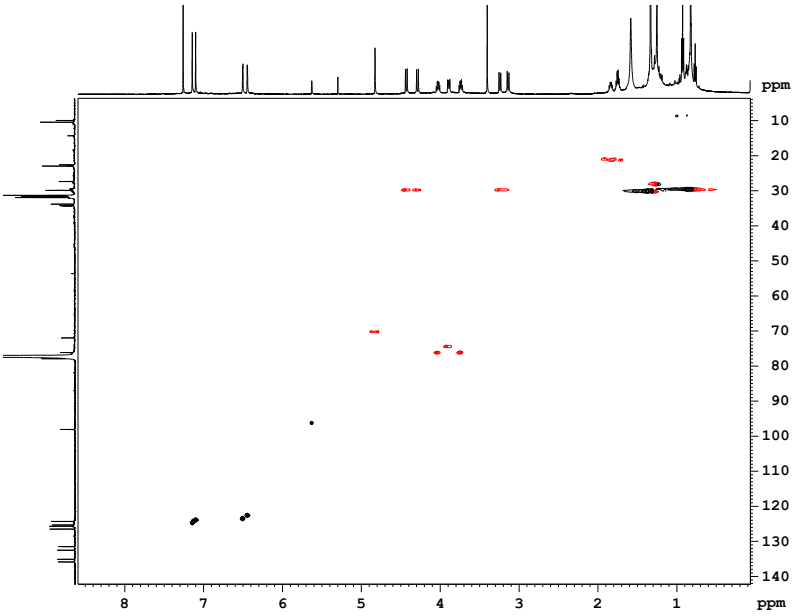
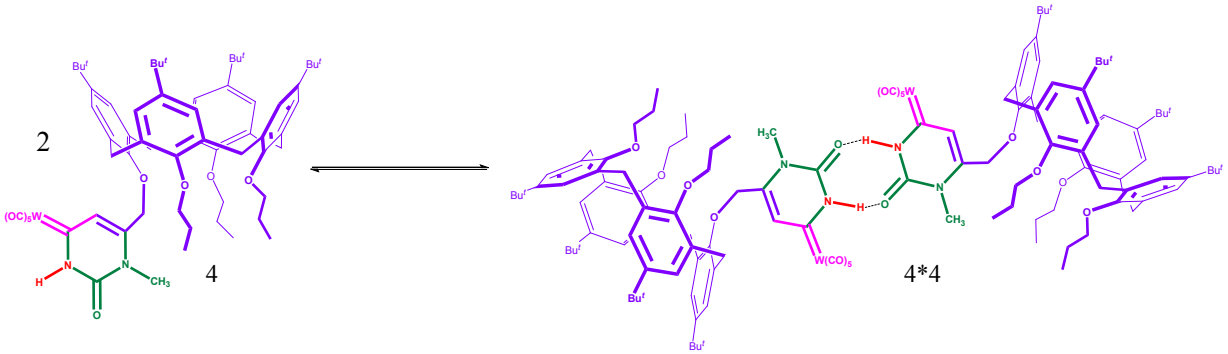


Figure S20. Portion of HSQC spectrum (600 MHz, $CDCl_3$, 298 K) of derivative **5**.

Dimerization Constant Calculation



The dimerisation of derivative **4** was studied by monitoring the NMR chemical shifts of NH upon dilution. The NMR diluition data in Figure S22 were fitted to the dimer model[1] in order to determine the possible structure of the aggregate, following the Saunders-Hyne method.

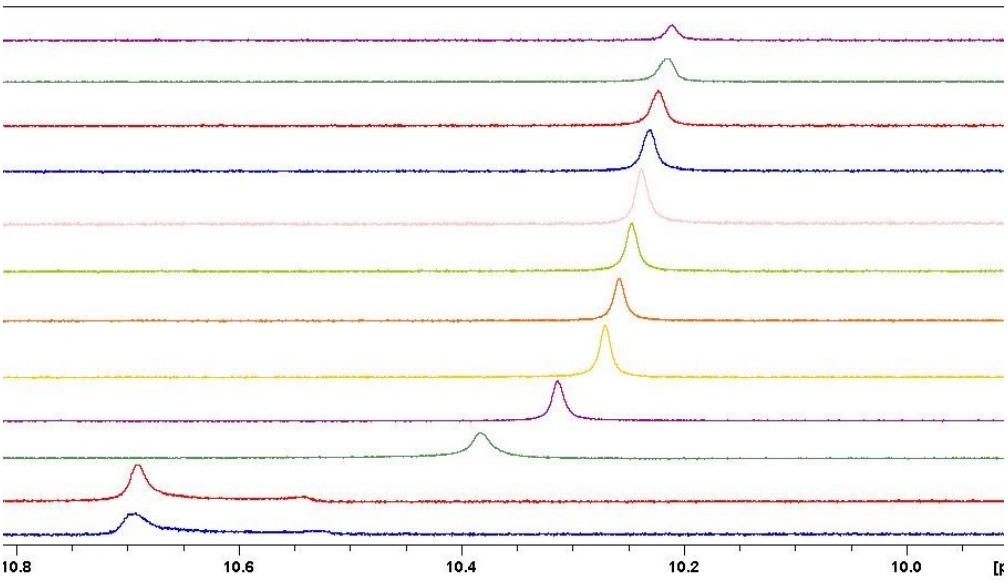


Figure S21. NMR study upon dilution (600 MHz, $CDCl_3$, 298 K) of derivative **4**

[1] Saunders, M.; Hyne, J. B. *J. Chem. Phys.* **1958**, *29*,1319-1323. Zafar, A.; Geib, S. J.,Hamuro, Y. Carr, A.; Hamilton, A. J. *Tetrahedron* **2000**, *56*, 8419-8427. Zimmerman, S.C.; Duerr, B. F. *J. Org. Chem* **1992**, *56*, 2215-2217

Biological assays

Cell lines

The cell lines CAL-27 , oral adenosquamous carcinoma cell line and PC3, human prostate cancer cell line, were grown in RPMI (Life Technologies, Paisley, UK) supplemented with 10% heat-inactivated fetal bovine serum (FBS; Life Technologies), 2 mM glutamine (Hyclone, Cramlington, UK), 50 U/ml penicillin and 50 U/ml streptomycin (Hyclone), hereafter defined as CM They were grown in adhesion culture, respectively, at a density of $3x10^5$ cells per ml and splitted twice a week.

MTT assay

PBMC was evaluated by MTT assay. Inhibition of cell metabolic activity was detected through formazan product formation, using a commercial colorimetric kit (MTT [3,4-(5-dimethylthiazol-2-yl)-5- (3-carboxymethoxyphenyl)-2-(4-sulfophenyl)-2H-tetrazolium salt]) (Cell Titer 96 Aqueous One Solution; Promega). This colorimetric standard assay measures the activity of the enzymes that reduce MTT to formazan. The assay was performed by seeding $5x10^3$ CAL-27 or PC3 cells in 100 µl of CM per well in 96 wells plate in triplicate in absence or in presence of 5, 10, 20, and 40 µM of compound **2**, **3**, **4** and **5** or SN38 (7-etil-10-idrossi-camptotecina) as positive control, for 24 hours. Twenty microliters of 'Cell Titer 96 Aqueous One Solution' reagent was added directly to culture wells at the end of the culture period and samples were incubated for 1 h. Successively the absorbance was read at 490.

Statistical analysis

Cumulative results from three determinations were used to calculate the initial compound concentration effective in inhibiting by 50% metabolic assay (IC_{50}± SD). The values were calculated according to the best-fit curve, y value versus log x, where y is the value of the examined function and x is the drug concentration.

Table S1. Crystallographic data for compounds 3 and 4

	3	4
Cambridge Database nr.	CCDC 1438070	CCDC 1437666
Temperature (K)	100(2) K	100(2)
Wavelength (Å)	0.700 A	0.700
Crystal system	Monoclinic	Monoclinic
space group	$P\,2_1/c$	$P\,2_1/n$
a (Å)	14.439(1)	10.582(2)
b (Å)	18.945(1)	26.982(5)
c (Å)	26.776(2)	25.802(5)
α (°)	90	90
β (°)	104.056(4)	94.7(3)
γ (°)	90	90
V (Å^3)	7105.2(8)	7342(3)
Z, $\rho_{calc.}$ (g·mm^{-3})	4	4
F(000)	2816	880
Crystal size (mm)	0.14 x 0.11 x 0.08	0.21 x 0.13 x 0.11
Shape, Colour	block, yellow	block, colourless

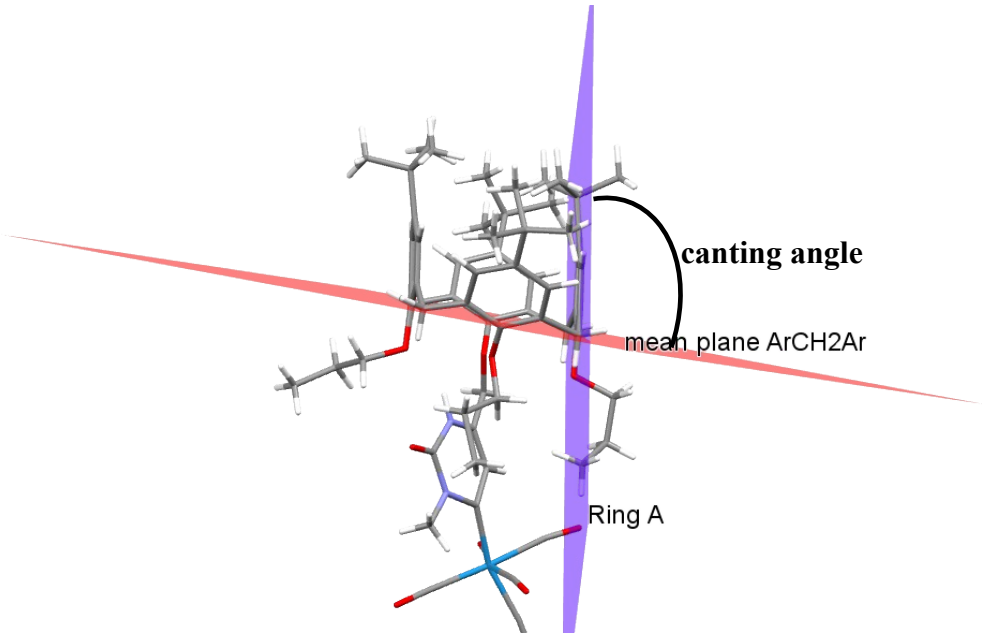


Figure S22. Canting Angle for A 98.2(1)°

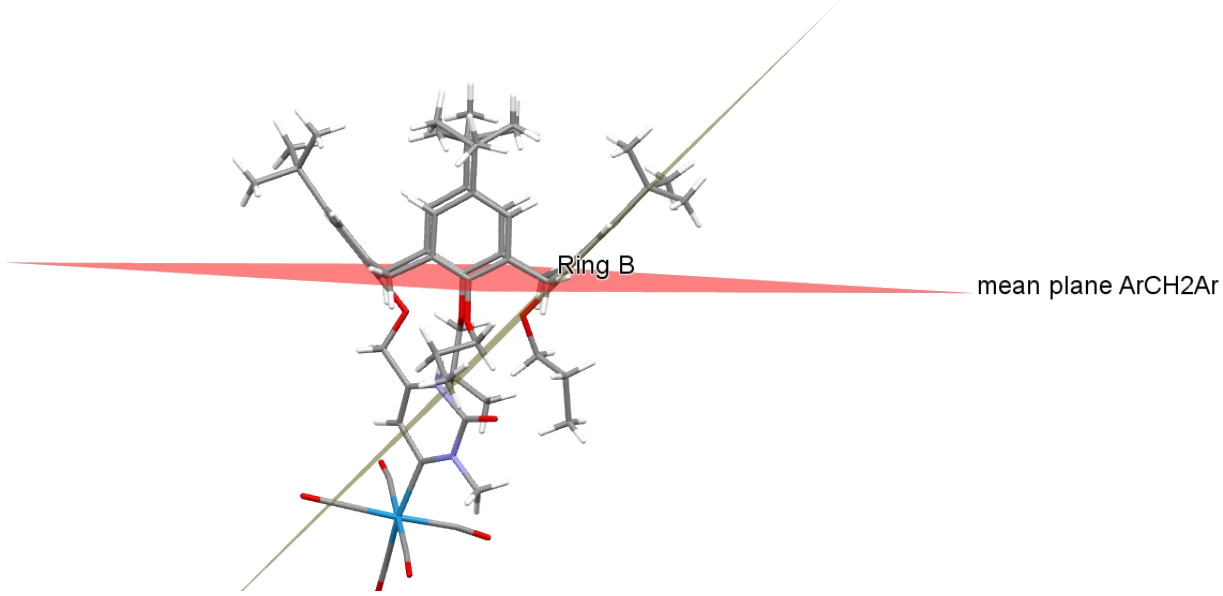


Figure S23. Canting Angle for B 47(2)°

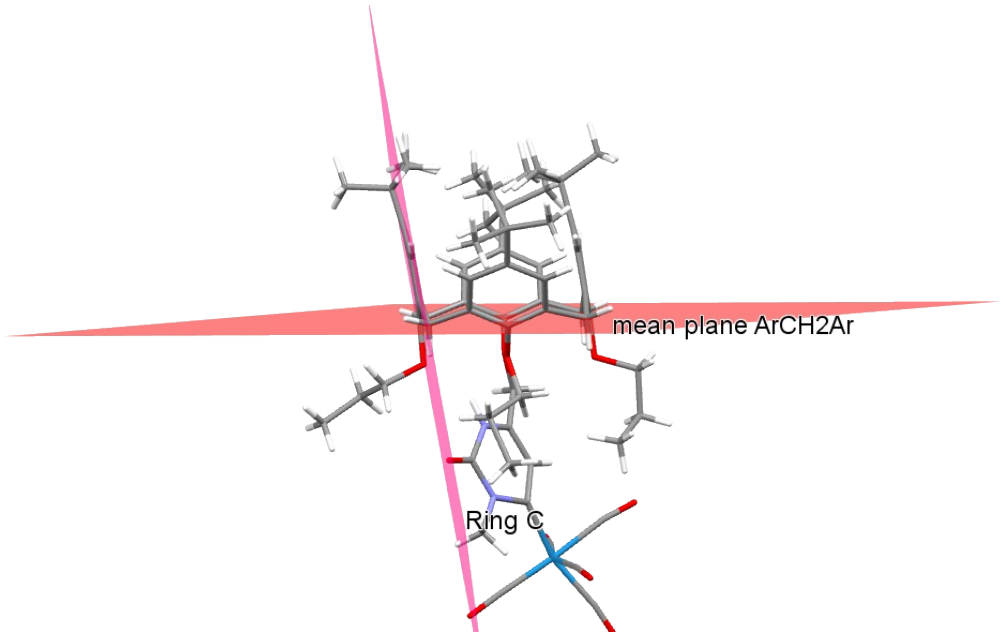


Figure S24. Canting Angle for C 82.8(2)°

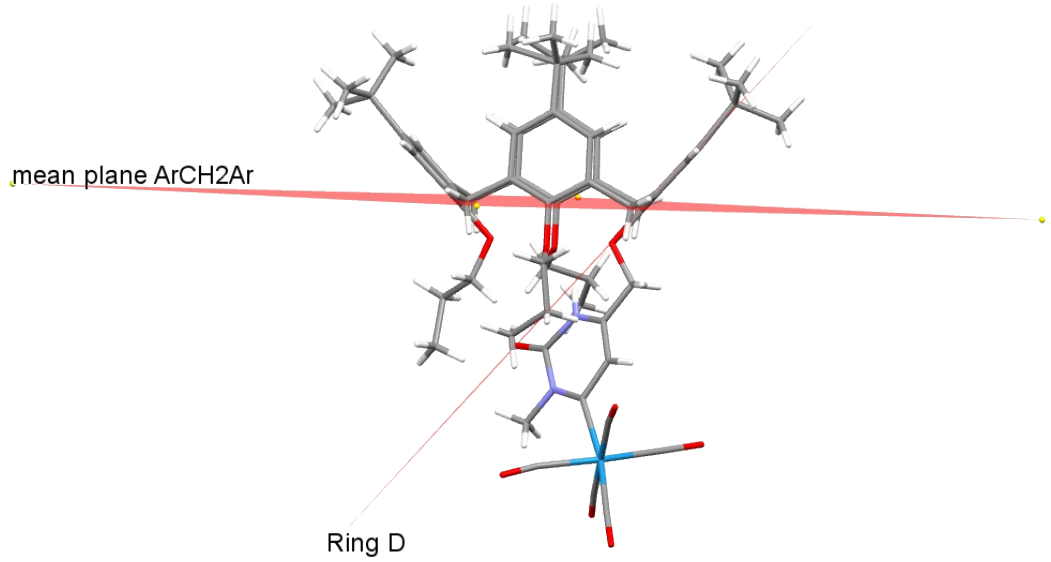


Figure S25. Canting Angle for D 49.5(2)°

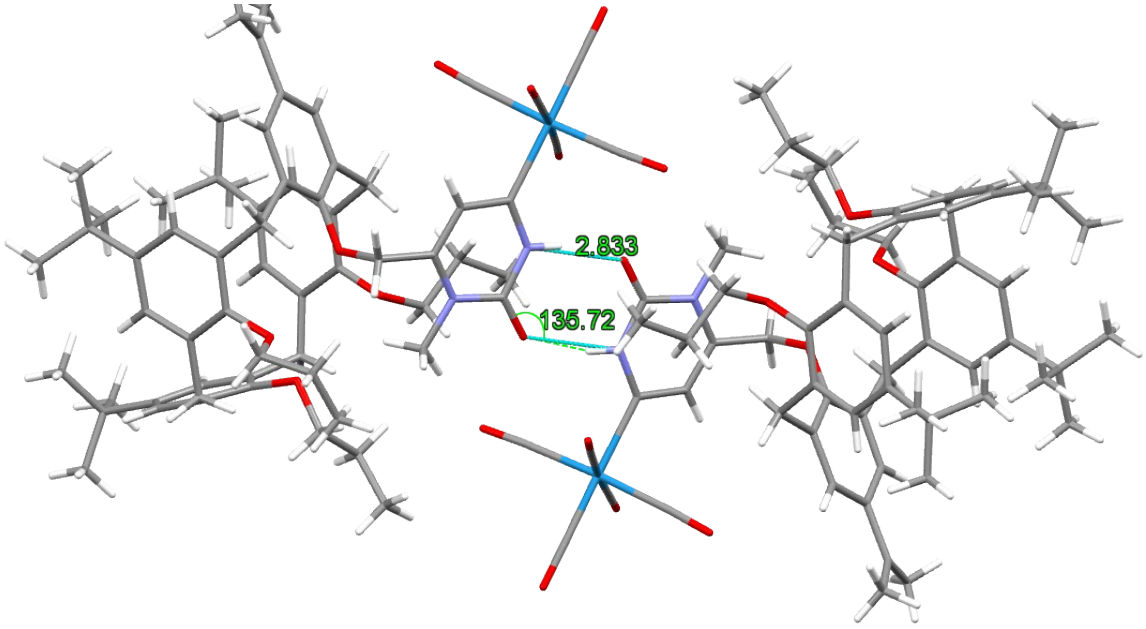


Figure S26. Intermolecular H-bond distance and C=O▪▪▪H-N angle (°) for the self-assembled dimer 4▪4.

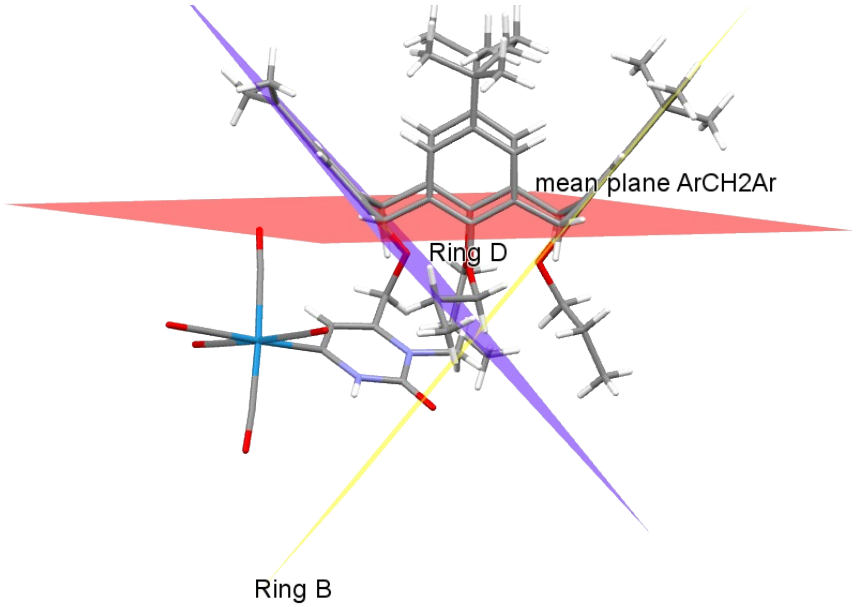


Figure S27. Canting Angles for B 52.21(6)° and D 46.95(5)°.

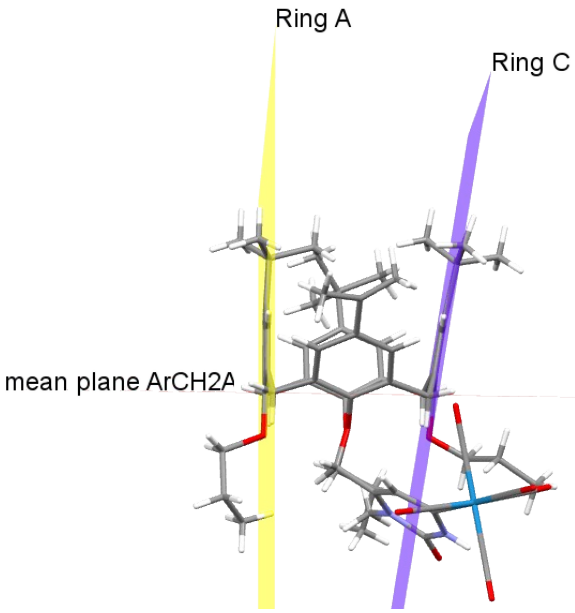


Figure S28. Canting Angles for A 90.27(6)° and C 81.44(6)°.

Bond distances and angles

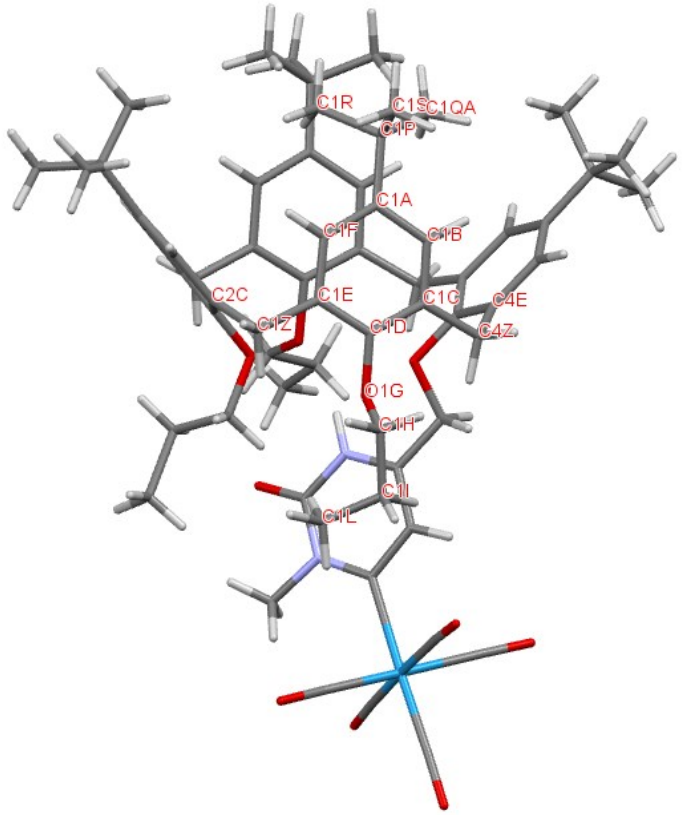


Figure S29. X-ray structure of **3.** Atoms labeling.

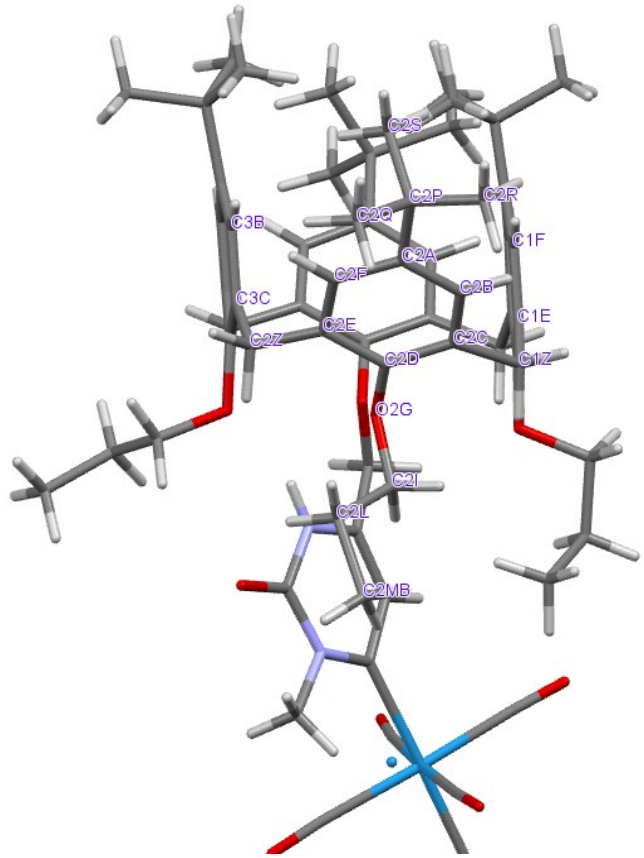


Figure S30. X-ray structure of **3.** Atoms labeling

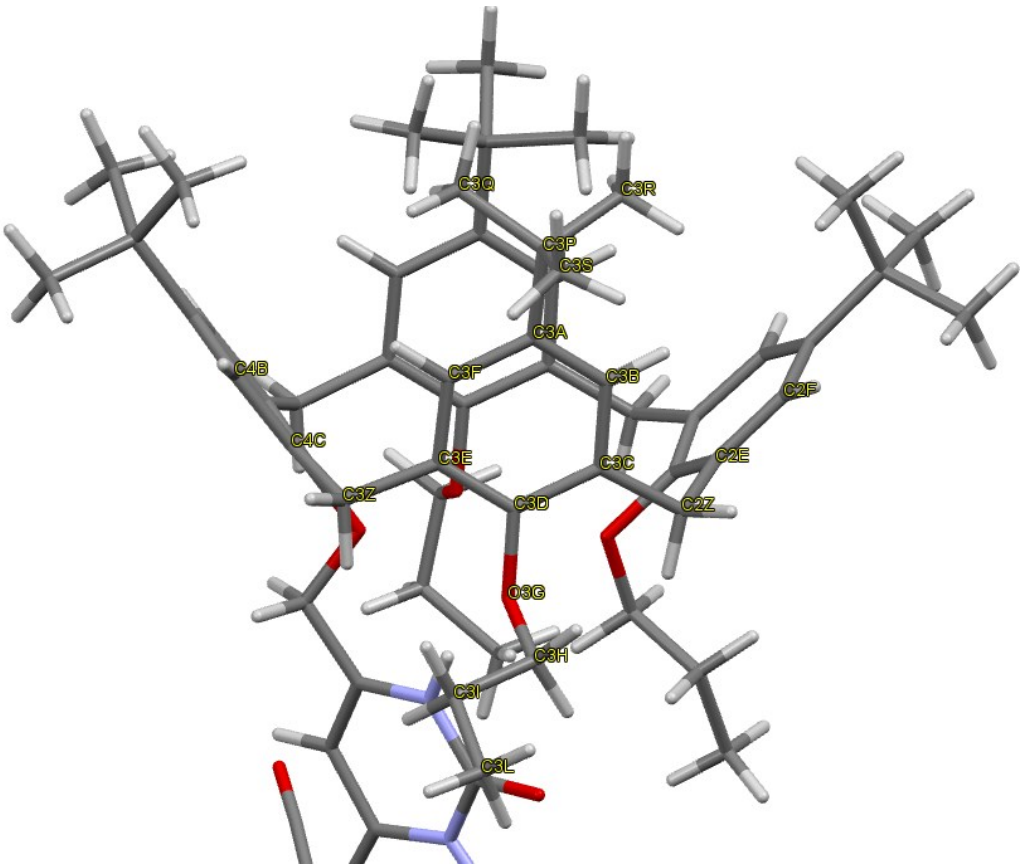


Figure S31. X-ray structure of **3.** Atoms labeling

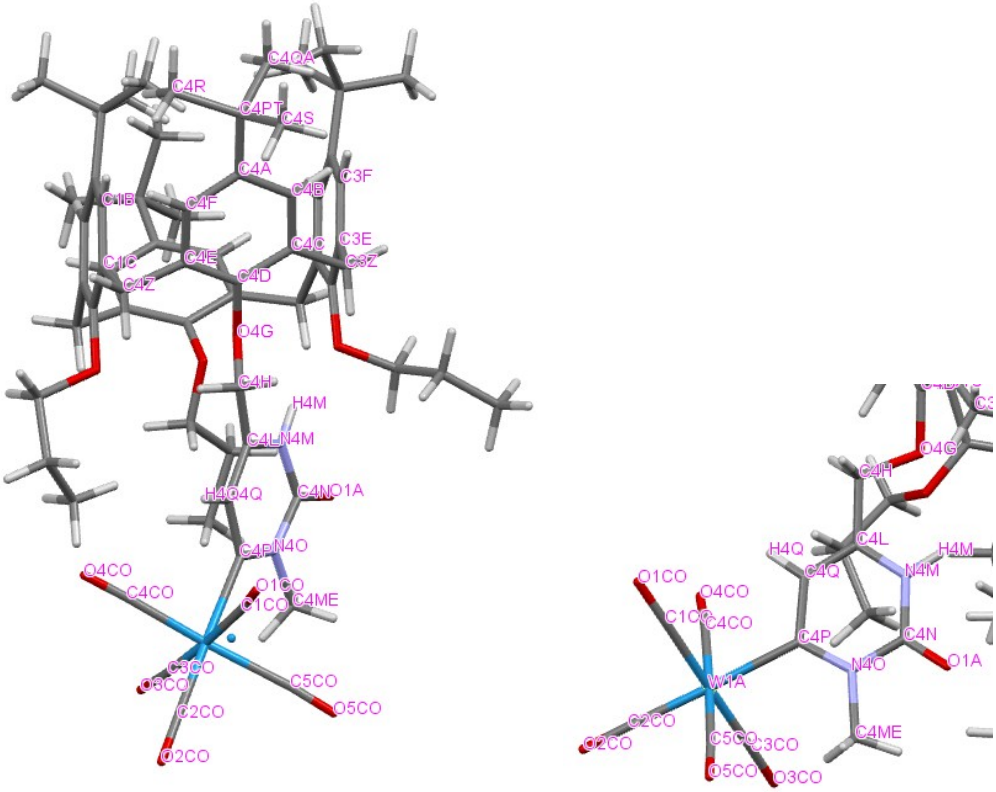


Figure S32. X-ray structure of **3.** Atoms labeling

Table S2. Bond distances for 3

Atom1	Atom2	Length
1 C1A	C1B	1.39(1)
2 C1A	C1F	1.36(1)
3 C1A	C1P	1.55(1)
5 C1B	C1C	1.39(1)
6 C1C	C1D	1.37(1)
7 C1C	C4Z	1.53(1)
8 C1D	C1E	1.39(1)
9 C1D	O1G	1.395(9)
10 C1E	C1F	1.40(1)
11 C1E	C1Z	1.50(1)
19 C1I	C1L	1.53(2)
23 C1P	C1S	1.47(1)
24 C1P	C1QA	1.47(2)
25 C1P	C1R	1.58(2)
31 C1Z	C2C	1.53(1)
35 C1CO	O1CO	1.16(2)
36 C1CO	W1A	2.05(1)
40 C2A	C2B	1.37(1)
41 C2A	C2F	1.39(2)
42 C2A	C2P	1.55(1)
44 C2B	C2C	1.41(1)
45 C2C	C2D	1.38(1)
46 C2D	C2E	1.40(1)
47 C2D	O2G	1.388(9)
48 C2E	C2F	1.41(1)
49 C2E	C2Z	1.50(1)
53 C2I	C2L	1.49(2)
54 C2I	O2G	1.42(1)
57 C2L	C2MB	1.61(2)
58 C2P	C2Q	1.58(2)
59 C2P	C2R	1.48(2)
60 C2P	C2S	1.49(2)
75 C2Z	C3C	1.50(1)
76 C2CO	O2CO	1.14(1)
77 C2CO	W1A	1.98(1)
78 C3A	C3B	1.37(1)
79 C3A	C3F	1.40(1)
80 C3A	C3P	1.55(1)
82 C3B	C3C	1.40(1)
83 C3C	C3D	1.41(1)
84 C3D	C3E	1.37(1)
85 C3D	O3G	1.399(9)
86 C3E	C3F	1.39(1)
87 C3E	C3Z	1.53(1)
91 C3H	C3I	1.52(1)
92 C3H	O3G	1.44(1)

95 C3I C3L 1.50(1)
99 C3P C3Q 1.54(2)
100 C3P C3R 1.50(1)
101 C3P C3S 1.49(2)
113 C3Z C4C 1.50(1)
114 C3CO O3CO 1.15(2)
115 C3CO W1A 1.99(1)
116 C4A C4B 1.40(1)
117 C4A C4F 1.37(1)
118 C4A C4PT 1.54(1)
120 C4B C4C 1.41(1)
121 C4C C4D 1.40(1)
122 C4D C4E 1.37(1)
123 C4D O4G 1.41(1)
124 C4E C4F 1.41(1)
125 C4E C4Z 1.51(1)
131 C4L C4Q 1.38(1)
132 C4L N4M 1.34(1)
133 C4N O1A 1.21(2)
134 C4N N4M 1.38(1)
135 C4N N4O 1.39(1)
136 C4P C4Q 1.41(1)
137 C4P W1A 2.267(8)
138 C4P N4O 1.38(1)
143 C4R C4PT 1.61(2)
147 C4S C4PT 1.53(2)
150 C4CO O4CO 1.09(1)
151 C4CO W1A 2.03(1)
155 C4ME N4O 1.47(2)
159 C4QA C4PT 1.53(1)
160 C5CO O5CO 1.12(2)
161 C5CO W1A 2.12(1)
162 N4M H4M 0.879

Bond Angles for 3

Atom1	Atom2	Atom3	Angle
1	C1B	C1A	C1F 116.3(9)
2	C1B	C1A	C1P 119.9(8)
3	C1F	C1A	C1P 123.8(9)
4	C1A	C1B	H1B 119.0
5	C1A	C1B	C1C 122.0(8)
6	H1B	C1B	C1C 119.0
7	C1B	C1C	C1D 119.9(8)
8	C1B	C1C	C4Z 117.2(7)
9	C1D	C1C	C4Z 122.5(7)
10	C1C	C1D	C1E 120.3(8)
11	C1C	C1D	O1G 120.1(7)
12	C1E	C1D	O1G 119.5(7)
13	C1D	C1E	C1F 117.3(8)
14	C1D	C1E	C1Z 122.2(8)
15	C1F	C1E	C1Z 120.4(8)
16	C1A	C1F	C1E 124.0(9)
17	C1A	C1F	H1F 118.0
18	C1E	C1F	H1F 118.0
19	H1H1	C1H	H1H2 108.3
20	H1H1	C1H	C1I 110.0
21	H1H1	C1H	O1G 109.9
22	H1H2	C1H	C1I 109.9
23	H1H2	C1H	O1G 109.8
24	C1I	C1H	O1G 108.9(7)
25	C1H	C1I	H1I1 108.9
26	C1H	C1I	H1I2 108.9
27	C1H	C1I	C1L 113.8(8)
28	H1I1	C1I	H1I2 107.6
29	H1I1	C1I	C1L 108.8
30	H1I2	C1I	C1L 108.8
31	C1I	C1L	H1L1 109
32	C1I	C1L	H1L2 110
33	C1I	C1L	H1L3 110
34	H1L1	C1L	H1L2 109
35	H1L1	C1L	H1L3 109
36	H1L2	C1L	H1L3 109
37	C1A	C1P	C1S 111.0(8)
38	C1A	C1P	C1QA 112(1)
39	C1A	C1P	C1R 108.4(9)
40	C1S	C1P	C1QA 112(1)
41	C1S	C1P	C1R 105(1)
42	C1QA	C1P	C1R 108(1)
43	C1P	C1S	H1S1 109
44	C1P	C1S	H1S2 109
45	C1P	C1S	H1S3 110
46	H1S1	C1S	H1S2 109

47 H1S1 C1S H1S3 109
48 H1S2 C1S H1S3 109
49 C1E C1Z H1Z1 110.1
50 C1E C1Z H1Z2 110.1
51 C1E C1Z C2C 108.0(7)
52 H1Z1 C1Z H1Z2 108.5
53 H1Z1 C1Z C2C 110.1
54 H1Z2 C1Z C2C 110.0
55 C1P C1QA H1Q1 109
56 C1P C1QA H1Q2 109
57 C1P C1QA H1Q3 109
58 H1Q1 C1QA H1Q2 109
59 H1Q1 C1QA H1Q3 110
60 H1Q2 C1QA H1Q3 110
61 O1CO C1CO W1A 174(1)
62 C1P C1R H1R1 109
63 C1P C1R H1R2 109
64 C1P C1R H1R3 109
65 H1R1 C1R H1R2 110
66 H1R1 C1R H1R3 109
67 H1R2 C1R H1R3 110
68 C2B C2A C2F 118.9(9)
69 C2B C2A C2P 121.3(9)
70 C2F C2A C2P 119.7(9)
71 C2A C2B H2B 119.1
72 C2A C2B C2C 121.6(9)
73 H2B C2B C2C 119.2
74 C1Z C2C C2B 119.3(8)
75 C1Z C2C C2D 121.7(8)
76 C2B C2C C2D 118.3(8)
77 C2C C2D C2E 122.1(8)
78 C2C C2D O2G 120.2(7)
79 C2E C2D O2G 117.4(7)
80 C2D C2E C2F 116.8(8)
81 C2D C2E C2Z 121.2(8)
82 C2F C2E C2Z 121.9(8)
83 C2A C2F C2E 121.9(9)
84 C2A C2F H2F 119.1
85 C2E C2F H2F 119.0
86 H2I1 C2I H2I2 108
87 H2I1 C2I C2L 109
88 H2I1 C2I O2G 108.7
89 H2I2 C2I C2L 109
90 H2I2 C2I O2G 108.8
91 C2L C2I O2G 114(1)
92 C2I C2L H2L1 110
93 C2I C2L H2L2 110
94 C2I C2L C2MB 109(1)
95 H2L1 C2L H2L2 108
96 H2L1 C2L C2MB 110
97 H2L2 C2L C2MB 110

98 C2A C2P C2Q 108(1)
99 C2A C2P C2R 113(1)
100 C2A C2P C2S 108.8(9)
101 C2Q C2P C2R 107(1)
102 C2Q C2P C2S 108(1)
103 C2R C2P C2S 113(1)
104 C2P C2Q H2Q1 109
105 C2P C2Q H2Q2 109
106 C2P C2Q H2Q3 109
107 H2Q1 C2Q H2Q2 109
108 H2Q1 C2Q H2Q3 110
109 H2Q2 C2Q H2Q3 109
110 C2P C2R H2R1 109
111 C2P C2R H2R2 109
112 C2P C2R H2R3 109
113 H2R1 C2R H2R2 110
114 H2R1 C2R H2R3 110
115 H2R2 C2R H2R3 110
116 C2P C2S H2S1 109
117 C2P C2S H2S2 109
118 C2P C2S H2S3 110
119 H2S1 C2S H2S2 110
120 H2S1 C2S H2S3 109
121 H2S2 C2S H2S3 110
122 C2L C2MB H2M1 110
123 C2L C2MB H2M2 109
124 C2L C2MB H2M3 109
125 H2M1 C2MB H2M2 110
126 H2M1 C2MB H2M3 110
127 H2M2 C2MB H2M3 109
128 C2E C2Z H2Z1 109.0
129 C2E C2Z H2Z2 109.1
130 C2E C2Z C3C 112.7(7)
131 H2Z1 C2Z H2Z2 107.8
132 H2Z1 C2Z C3C 109.1
133 H2Z2 C2Z C3C 109.1
134 O2CO C2CO W1A 178(1)
135 C3B C3A C3F 117.8(7)
136 C3B C3A C3P 121.5(7)
137 C3F C3A C3P 120.6(7)
138 C3A C3B H3B 118.7
139 C3A C3B C3C 122.5(7)
140 H3B C3B C3C 118.8
141 C2Z C3C C3B 119.4(7)
142 C2Z C3C C3D 123.3(7)
143 C3B C3C C3D 117.2(7)
144 C3C C3D C3E 121.7(7)
145 C3C C3D O3G 117.9(7)
146 C3E C3D O3G 120.4(7)
147 C3D C3E C3F 118.6(7)
148 C3D C3E C3Z 122.9(7)

149 C3F C3E C3Z 118.5(7)
150 C3A C3F C3E 121.8(7)
151 C3A C3F H3F 119.2
152 C3E C3F H3F 119.1
153 H3H1 C3H H3H2 108.1
154 H3H1 C3H C3I 109.2
155 H3H1 C3H O3G 109.3
156 H3H2 C3H C3I 109.2
157 H3H2 C3H O3G 109.3
158 C3I C3H O3G 111.7(7)
159 C3H C3I H3I1 109.4
160 C3H C3I H3I2 109.5
161 C3H C3I C3L 110.6(9)
162 H3I1 C3I H3I2 108
163 H3I1 C3I C3L 110
164 H3I2 C3I C3L 110
165 C3I C3L H3L1 110
166 C3I C3L H3L2 109
167 C3I C3L H3L3 109
168 H3L1 C3L H3L2 109
169 H3L1 C3L H3L3 110
170 H3L2 C3L H3L3 109
171 C3A C3P C3Q 111.5(8)
172 C3A C3P C3R 111.6(8)
173 C3A C3P C3S 108.6(9)
174 C3Q C3P C3R 107.2(9)
175 C3Q C3P C3S 109(1)
176 C3R C3P C3S 108(1)
177 C3P C3Q H3Q1 109
178 C3P C3Q H3Q2 110
179 C3P C3Q H3Q3 110
180 H3Q1 C3Q H3Q2 109
181 H3Q1 C3Q H3Q3 109
182 H3Q2 C3Q H3Q3 109
183 C3P C3R H3R1 110
184 C3P C3R H3R2 109
185 C3P C3R H3R3 110
186 H3R1 C3R H3R2 109
187 H3R1 C3R H3R3 109
188 H3R2 C3R H3R3 109
189 C3P C3S H3S1 109
190 C3P C3S H3S2 109
191 C3P C3S H3S3 110
192 H3S1 C3S H3S2 109
193 H3S1 C3S H3S3 110
194 H3S2 C3S H3S3 109
195 C3E C3Z H3Z1 109.3
196 C3E C3Z H3Z2 109.3
197 C3E C3Z C4C 111.3(7)
198 H3Z1 C3Z H3Z2 108.0
199 H3Z1 C3Z C4C 109.3

200 H3Z2 C3Z C4C 109.4
201 O3CO C3CO W1A 175(1)
202 C4B C4A C4F 119.1(8)
203 C4B C4A C4PT 119.9(8)
204 C4F C4A C4PT 121.0(8)
205 C4A C4B H4B 118.9
206 C4A C4B C4C 121.9(8)
207 H4B C4B C4C 119.1
208 C3Z C4C C4B 121.8(7)
209 C3Z C4C C4D 121.7(7)
210 C4B C4C C4D 116.2(7)
211 C4C C4D C4E 123.3(7)
212 C4C C4D O4G 117.2(7)
213 C4E C4D O4G 119.5(7)
214 C4D C4E C4F 118.6(7)
215 C4D C4E C4Z 122.4(7)
216 C4F C4E C4Z 118.1(7)
217 C4A C4F C4E 120.9(8)
218 C4A C4F H4F 119.6
219 C4E C4F H4F 119.6
220 H4H1 C4H H4H2 107.8
221 H4H1 C4H C4L 109.4
222 H4H1 C4H O4G 109.4
223 H4H2 C4H C4L 109.4
224 H4H2 C4H O4G 109.5
225 C4L C4H O4G 111.3(7)
226 C4H C4L C4Q 121.1(8)
227 C4H C4L N4M 118.2(7)
228 C4Q C4L N4M 120.2(8)
229 O1A C4N N4M 122(1)
230 O1A C4N N4O 122(1)
231 N4M C4N N4O 116.0(9)
232 C4Q C4P W1A 118.7(6)
233 C4Q C4P N4O 113.0(7)
234 W1A C4P N4O 128.3(6)
235 C4L C4Q C4P 122.8(8)
236 C4L C4Q H4Q 118.6
237 C4P C4Q H4Q 118.6
238 H4R1 C4R H4R2 110
239 H4R1 C4R H4R3 109
240 H4R1 C4R C4PT 109
241 H4R2 C4R H4R3 109
242 H4R2 C4R C4PT 109
243 H4R3 C4R C4PT 109
244 H4S1 C4S H4S2 110
245 H4S1 C4S H4S3 109
246 H4S1 C4S C4PT 110
247 H4S2 C4S H4S3 110
248 H4S2 C4S C4PT 109
249 H4S3 C4S C4PT 109
250 C1C C4Z C4E 108.2(7)

251 C1C C4Z H4Z1 110.0
252 C1C C4Z H4Z2 110.2
253 C4E C4Z H4Z1 110.1
254 C4E C4Z H4Z2 110.1
255 H4Z1 C4Z H4Z2 108.3
256 O4CO C4CO W1A 176.7(8)
257 H4M1 C4ME H4M2 109
258 H4M1 C4ME H4M3 110
259 H4M1 C4ME N4O 109
260 H4M2 C4ME H4M3 109
261 H4M2 C4ME N4O 109
262 H4M3 C4ME N4O 109
263 H4Q1 C4QA H4Q2 110
264 H4Q1 C4QA H4Q3 109
265 H4Q1 C4QA C4PT 109.5
266 H4Q2 C4QA H4Q3 109
267 H4Q2 C4QA C4PT 109.5
268 H4Q3 C4QA C4PT 109.4
269 C4A C4PT C4R 111.2(9)
270 C4A C4PT C4S 108(1)
271 C4A C4PT C4QA 110.6(8)
272 C4R C4PT C4S 113(1)
273 C4R C4PT C4QA 106.3(9)
274 C4S C4PT C4QA 108(1)
275 O5CO C5CO W1A 176(1)
276 C1D O1G C1H 112.9(6)
277 C2D O2G C2I 113.4(7)
278 C3D O3G C3H 112.3(6)
279 C4D O4G C4H 109.8(6)
280 C1CO W1A C2CO 90.3(5)
281 C1CO W1A C3CO 177.2(5)
282 C1CO W1A C4P 87.2(4)
283 C1CO W1A C4CO 88.0(4)
284 C1CO W1A C5CO 87.5(5)
285 C2CO W1A C3CO 89.5(5)
286 C2CO W1A C4P 177.3(4)
287 C2CO W1A C4CO 92.2(4)
288 C2CO W1A C5CO 88.4(5)
289 C3CO W1A C4P 93.0(4)
290 C3CO W1A C4CO 89.2(4)
291 C3CO W1A C5CO 95.3(5)
292 C4P W1A C4CO 88.7(3)
293 C4P W1A C5CO 90.6(4)
294 C4CO W1A C5CO 175.5(4)
295 C4L N4M C4N 121.8(8)
296 C4L N4M H4M 119.0
297 C4N N4M H4M 119.2
298 C4N N4O C4P 126.0(8)
299 C4N N4O C4ME 112.3(9)
300 C4P N4O C4ME 121.5(8)
301 H1C1 C1CE H1C2 106

302 H1C1 C1CE C2CE 105
303 H1C1 C1CE C6CE 105
304 H1C2 C1CE C2CE 106
305 H1C2 C1CE C6CE 106
306 C2CE C1CE C6CE 127(3)
307 C1CE C2CE H2C1 109
308 C1CE C2CE H2C2 109
309 C1CE C2CE C3CE 113(4)
310 H2C1 C2CE H2C2 108
311 H2C1 C2CE C3CE 108
312 H2C2 C2CE C3CE 109
313 C2CE C3CE H3C1 106
314 C2CE C3CE H3C2 107
315 C2CE C3CE C4CE 124(5)
316 H3C1 C3CE H3C2 106
317 H3C1 C3CE C4CE 106
318 H3C2 C3CE C4CE 107
319 C3CE C4CE H4C1 108
320 C3CE C4CE H4C2 108
321 C3CE C4CE C5CE 114(4)
322 H4C1 C4CE H4C2 108
323 H4C1 C4CE C5CE 109
324 H4C2 C4CE C5CE 110
325 C4CE C5CE H5C1 106
326 C4CE C5CE H5C2 107
327 C4CE C5CE C6CE 123(3)
328 H5C1 C5CE H5C2 107
329 H5C1 C5CE C6CE 107
330 H5C2 C5CE C6CE 107
331 C1CE C6CE C5CE 116(3)
332 C1CE C6CE H6C1 108
333 C1CE C6CE H6C2 109
334 C5CE C6CE H6C1 108
335 C5CE C6CE H6C2 108
336 H6C1 C6CE H6C2 107
337 H1E1 C1EX H1E2 109
338 H1E1 C1EX C2EX 110
339 H1E1 C1EX C6EX 109
340 H1E2 C1EX C2EX 109
341 H1E2 C1EX C6EX 110
342 C2EX C1EX C6EX 111(5)
343 C1EX C2EX H2E1 108
344 C1EX C2EX H2E2 107
345 C1EX C2EX C3EX 118(5)
346 H2E1 C2EX H2E2 108
347 H2E1 C2EX C3EX 108
348 H2E2 C2EX C3EX 108
349 C2EX C3EX H3E1 110
350 C2EX C3EX H3E2 111
351 C2EX C3EX C4EX 107(4)
352 H3E1 C3EX H3E2 108

353 H3E1 C3EX C4EX 110
354 H3E2 C3EX C4EX 111
355 C3EX C4EX H4E1 109
356 C3EX C4EX H4E2 108
357 C3EX C4EX C5EX 114(4)
358 H4E1 C4EX H4E2 108
359 H4E1 C4EX C5EX 109
360 H4E2 C4EX C5EX 109
361 C4EX C5EX H5E1 108
362 C4EX C5EX H5E2 108
363 C4EX C5EX C6EX 116(4)
364 H5E1 C5EX H5E2 108
365 H5E1 C5EX C6EX 109
366 H5E2 C5EX C6EX 108
367 C1EX C6EX C5EX 107(4)
368 C1EX C6EX H6E1 110
369 C1EX C6EX H6E2 110
370 C5EX C6EX H6E1 110
371 C5EX C6EX H6E2 110
372 H6E1 C6EX H6E2 109

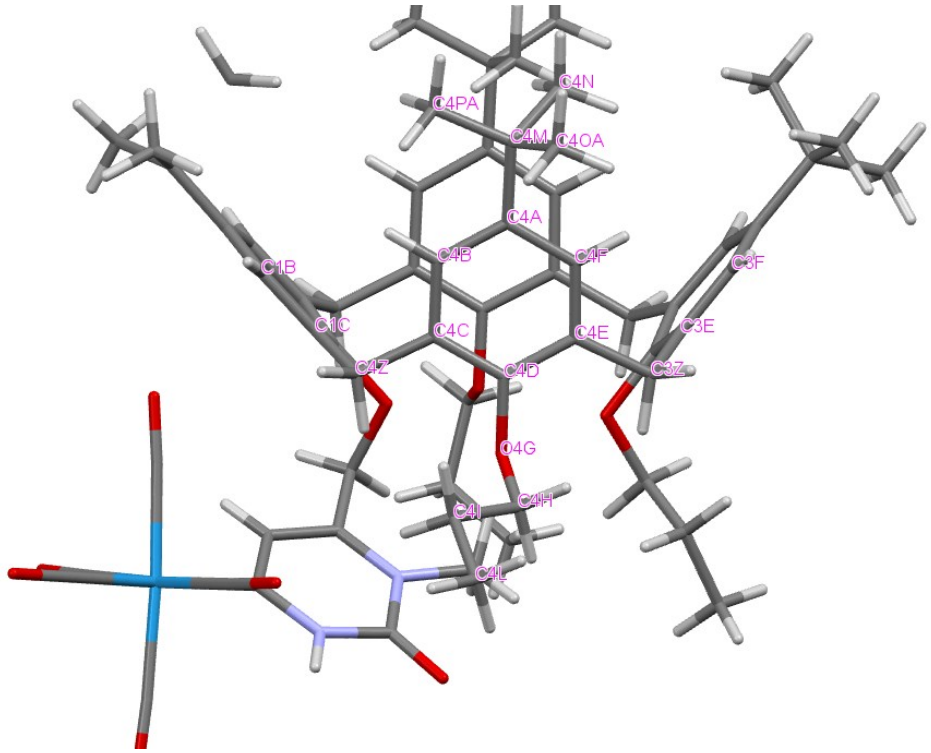


Figure S33. X-ray structure of **4.** Atoms labeling

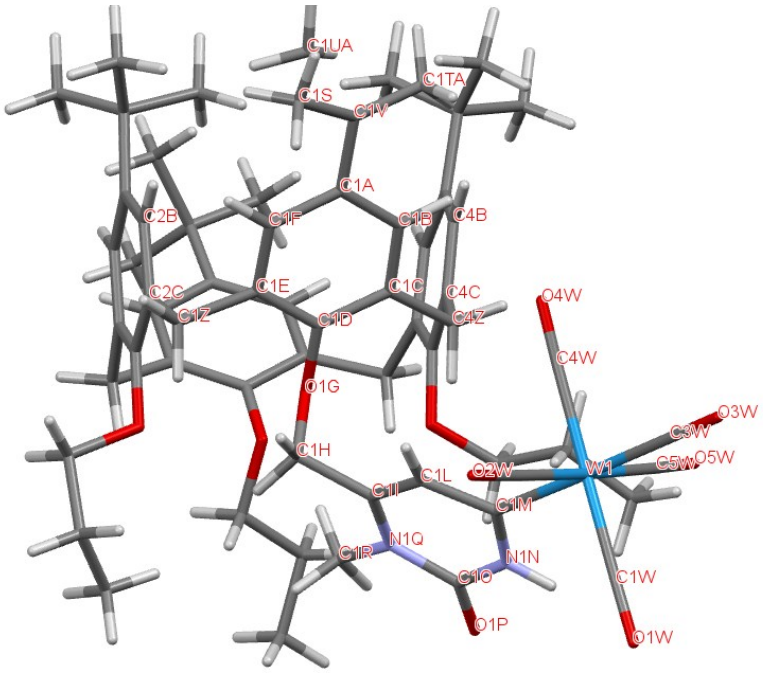


Figure S34. X-ray structure of **4.** Atoms labeling

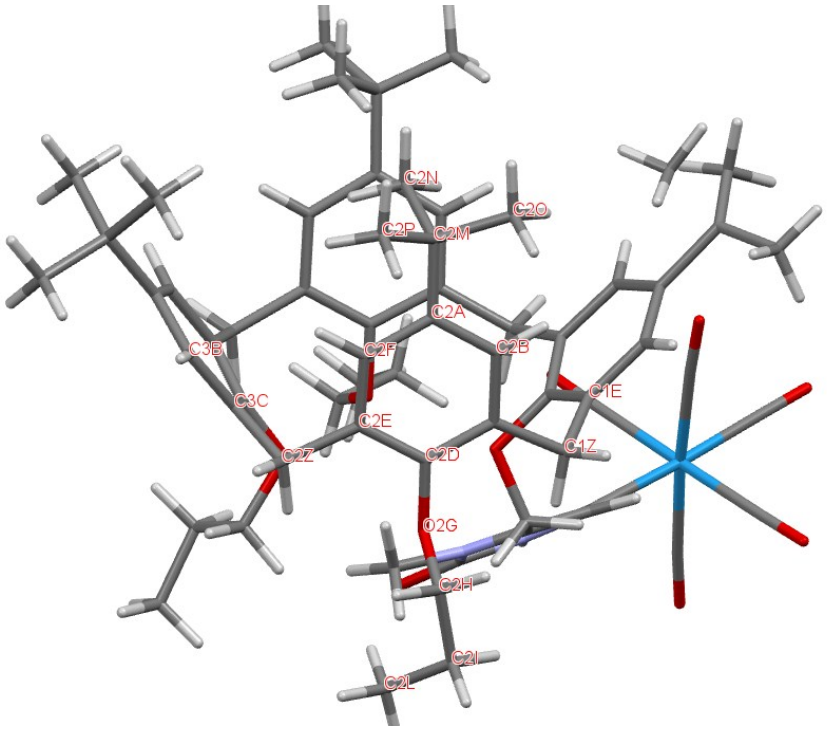


Figure S34. X-ray structure of **4.** Atoms labeling

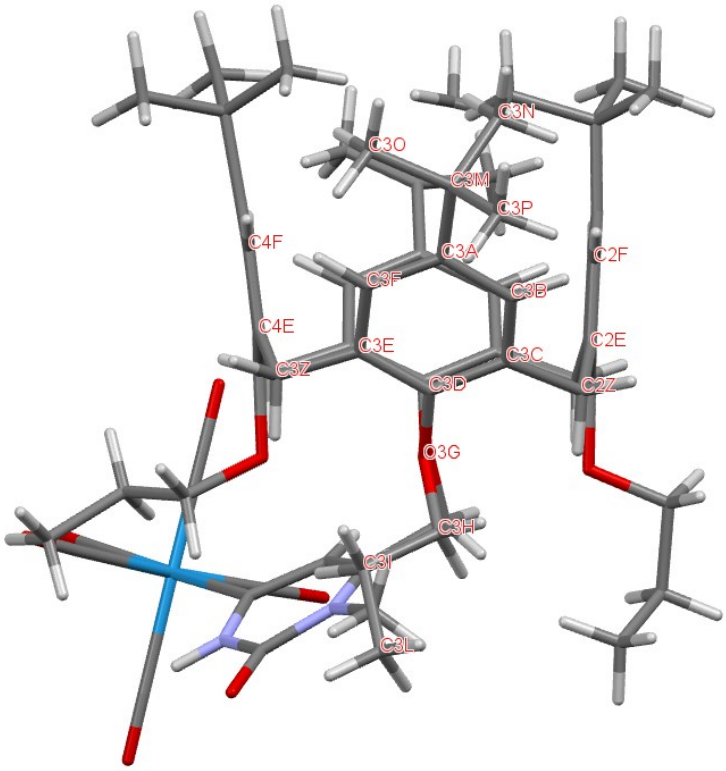


Figure S35. X-ray structure of **4.** Atoms labeling

Table S3. Bond distances for 4

Atom1	Atom2	Length
W1	C1W	2.038(2)
W1	C2W	2.024(2)
W1	C3W	2.023(3)
W1	C4W	2.048(2)
W1	C5W	2.050(3)
W1	C1M	2.206(2)
C1W	O1W	1.140(3)
C2W	O2W	1.131(3)
C3W	O3W	1.143(3)
C4W	O4W	1.141(3)
C5W	O5W	1.140(4)
C1A	C1B	1.391(3)
C1A	C1F	1.394(3)
C1A	C1V	1.523(4)
C1B	C1C	1.396(3)
C1C	C1D	1.397(3)
C1C	C4Z	1.512(3)
C1D	C1E	1.394(3)
C1D	O1G	1.391(2)
C1E	C1F	1.392(3)
C1E	C1Z	1.515(3)
C1H	C1I	1.507(3)
C1H	O1G	1.435(2)
C1I	C1L	1.366(3)
C1I	N1Q	1.363(3)
C1L	C1M	1.422(3)
C1M	N1N	1.367(3)
C1O	N1N	1.383(3)
C1O	N1Q	1.375(3)
C1O	O1P	1.226(2)
C1R	N1Q	1.475(2)
C1S	C1V	1.486(5)
C1TA	C1V	1.34(1)
C1Z	C2C	1.519(3)
C2A	C2B	1.388(3)
C2A	C2F	1.391(3)
C2A	C2M	1.528(3)
C2B	C2C	1.397(3)
C2C	C2D	1.393(3)
C2D	C2E	1.396(3)

58 C2D O2G 1.394(2)
59 C2E C2F 1.399(3)
60 C2E C2Z 1.521(3)
64 C2H C2I 1.515(3)
65 C2H O2G 1.438(3)
68 C2I C2L 1.498(4)
72 C2M C2N 1.544(4)
73 C2M C2O 1.526(4)
74 C2M C2P 1.539(4)
86 C2Z C3C 1.516(3)
87 C3A C3B 1.399(3)
88 C3A C3F 1.391(3)
89 C3A C3M 1.531(3)
91 C3B C3C 1.387(3)
92 C3C C3D 1.403(3)
93 C3D C3E 1.387(3)
94 C3D O3G 1.386(2)
95 C3E C3F 1.400(3)
96 C3E C3Z 1.517(3)
100 C3H C3I 1.512(3)
101 C3H O3G 1.443(2)
104 C3I C3L 1.517(4)
108 C3M C3N 1.535(4)
109 C3M C3O 1.530(4)
110 C3M C3P 1.534(4)
122 C3Z C4E 1.524(3)
123 C4A C4B 1.396(3)
124 C4A C4F 1.390(3)
125 C4A C4M 1.532(3)
127 C4B C4C 1.399(3)
128 C4C C4D 1.393(3)
129 C4C C4Z 1.521(3)
130 C4D C4E 1.401(3)
131 C4D O4G 1.385(2)
132 C4E C4F 1.396(3)
136 C4H C4I 1.517(3)
137 C4H O4G 1.437(3)
140 C4I C4L 1.527(4)
144 C4M C4N 1.509(4)
145 C4M C4OA 1.570(6)
146 C4M C4PA 1.482(6)
158 N1N H1N 0.880

Table S4. Bond Angles for 4

	Atom1	Atom2	Atom3	Angle
1	C1W	W1	C2W	90.26(9)
2	C1W	W1	C3W	89.9(1)
3	C1W	W1	C4W	176.14(9)
4	C1W	W1	C5W	92.6(1)
5	C1W	W1	C1M	91.55(9)
6	C2W	W1	C3W	91.1(1)
7	C2W	W1	C4W	87.01(9)
8	C2W	W1	C5W	176.7(1)
9	C2W	W1	C1M	90.60(8)
10	C3W	W1	C4W	87.5(1)
11	C3W	W1	C5W	90.6(1)
12	C3W	W1	C1M	177.8(1)
13	C4W	W1	C5W	90.3(1)
14	C4W	W1	C1M	91.20(8)
15	C5W	W1	C1M	87.7(1)
16	W1	C1W	O1W	176.7(2)
17	W1	C2W	O2W	178.9(2)
18	W1	C3W	O3W	179.3(2)
19	W1	C4W	O4W	176.9(2)
20	W1	C5W	O5W	178.3(3)
21	C1B	C1A	C1F	117.7(2)
22	C1B	C1A	C1V	120.5(2)
23	C1F	C1A	C1V	121.7(2)
24	C1A	C1B	H1B	118.6
25	C1A	C1B	C1C	122.8(2)
26	H1B	C1B	C1C	118.6
27	C1B	C1C	C1D	117.2(2)
28	C1B	C1C	C4Z	120.6(2)
29	C1D	C1C	C4Z	122.1(2)
30	C1C	C1D	C1E	121.8(2)
31	C1C	C1D	O1G	119.4(2)
32	C1E	C1D	O1G	118.7(2)
33	C1D	C1E	C1F	118.5(2)
34	C1D	C1E	C1Z	120.2(2)
35	C1F	C1E	C1Z	120.9(2)
36	C1A	C1F	C1E	121.7(2)
37	C1A	C1F	H1F	119.2
38	C1E	C1F	H1F	119.2
39	H1H1	C1H	H1H2	108.3
40	H1H1	C1H	C1I	109.9

41 H1H1 C1H O1G 110.0
42 H1H2 C1H C1I 110.0
43 H1H2 C1H O1G 109.9
44 C1I C1H O1G 108.6(2)
45 C1H C1I C1L 121.2(2)
46 C1H C1I N1Q 117.8(2)
47 C1L C1I N1Q 120.9(2)
48 C1I C1L H1L 119.0
49 C1I C1L C1M 122.1(2)
50 H1L C1L C1M 118.9
51 W1 C1M C1L 124.6(1)
52 W1 C1M N1N 123.2(1)
53 C1L C1M N1N 112.0(2)
54 N1N C1O N1Q 115.2(2)
55 N1N C1O O1P 122.2(2)
56 N1Q C1O O1P 122.6(2)
57 H1R1 C1R H1R2 109.4
58 H1R1 C1R H1R3 109.6
59 H1R1 C1R N1Q 109.5
60 H1R2 C1R H1R3 109.5
61 H1R2 C1R N1Q 109.4
62 H1R3 C1R N1Q 109.4
63 H1S1 C1S H1S2 109.5
64 H1S1 C1S H1S3 109.5
65 H1S1 C1S C1V 109.5
66 H1S2 C1S H1S3 109.4
67 H1S2 C1S C1V 109.5
68 H1S3 C1S C1V 109.5
69 H1T1 C1TA H1T2 109
70 H1T1 C1TA H1T3 109
71 H1T1 C1TA C1V 110
72 H1T2 C1TA H1T3 109
73 H1T2 C1TA C1V 110
74 H1T3 C1TA C1V 110
75 C1A C1V C1S 114.1(3)
76 C1A C1V C1TA 118.6(9)
77 C1S C1V C1TA 124.1(9)
78 C1E C1Z H1Z1 109.9
79 C1E C1Z H1Z2 110.0
80 C1E C1Z C2C 108.7(2)
81 H1Z1 C1Z H1Z2 108.3
82 H1Z1 C1Z C2C 110.0
83 H1Z2 C1Z C2C 109.9
84 C2B C2A C2F 117.2(2)
85 C2B C2A C2M 122.1(2)
86 C2F C2A C2M 120.5(2)
87 C2A C2B H2B 119.0
88 C2A C2B C2C 122.0(2)
89 H2B C2B C2C 119.0
90 C1Z C2C C2B 118.3(2)
91 C1Z C2C C2D 122.2(2)

92 C2B C2C C2D 119.3(2)
93 C2C C2D C2E 120.3(2)
94 C2C C2D O2G 119.3(2)
95 C2E C2D O2G 120.4(2)
96 C2D C2E C2F 118.3(2)
97 C2D C2E C2Z 122.1(2)
98 C2F C2E C2Z 119.4(2)
99 C2A C2F C2E 122.8(2)
100 C2A C2F H2F 118.7
101 C2E C2F H2F 118.6
102 H2H1 C2H H2H2 108.4
103 H2H1 C2H C2I 109.9
104 H2H1 C2H O2G 109.9
105 H2H2 C2H C2I 109.8
106 H2H2 C2H O2G 109.9
107 C2I C2H O2G 109.0(2)
108 C2H C2I H2I1 108.7
109 C2H C2I H2I2 108.7
110 C2H C2I C2L 113.8(2)
111 H2I1 C2I H2I2 107.8
112 H2I1 C2I C2L 108.8
113 H2I2 C2I C2L 108.8
114 C2I C2L H2L1 109.5
115 C2I C2L H2L2 109.5
116 C2I C2L H2L3 109.5
117 H2L1 C2L H2L2 109.5
118 H2L1 C2L H2L3 109.5
119 H2L2 C2L H2L3 109.4
120 C2A C2M C2N 107.3(2)
121 C2A C2M C2O 112.3(2)
122 C2A C2M C2P 110.6(2)
123 C2N C2M C2O 109.0(2)
124 C2N C2M C2P 108.6(2)
125 C2O C2M C2P 108.9(2)
126 C2M C2N H2N1 109.4
127 C2M C2N H2N2 109.5
128 C2M C2N H2N3 109.5
129 H2N1 C2N H2N2 109.5
130 H2N1 C2N H2N3 109.4
131 H2N2 C2N H2N3 109.5
132 C2M C2O H2O1 109.5
133 C2M C2O H2O2 109.5
134 C2M C2O H2O3 109.4
135 H2O1 C2O H2O2 109.5
136 H2O1 C2O H2O3 109.5
137 H2O2 C2O H2O3 109.4
138 C2M C2P H2P1 109.4
139 C2M C2P H2P2 109.5
140 C2M C2P H2P3 109.5
141 H2P1 C2P H2P2 109.5
142 H2P1 C2P H2P3 109.5

143 H2P2 C2P H2P3 109.4
144 C2E C2Z H2Z1 109.9
145 C2E C2Z H2Z2 109.9
146 C2E C2Z C3C 108.7(2)
147 H2Z1 C2Z H2Z2 108.5
148 H2Z1 C2Z C3C 109.9
149 H2Z2 C2Z C3C 109.9
150 C3B C3A C3F 117.1(2)
151 C3B C3A C3M 119.8(2)
152 C3F C3A C3M 123.1(2)
153 C3A C3B H3B 118.8
154 C3A C3B C3C 122.5(2)
155 H3B C3B C3C 118.7
156 C2Z C3C C3B 120.6(2)
157 C2Z C3C C3D 120.5(2)
158 C3B C3C C3D 118.4(2)
159 C3C C3D C3E 120.8(2)
160 C3C C3D O3G 119.1(2)
161 C3E C3D O3G 120.0(2)
162 C3D C3E C3F 118.6(2)
163 C3D C3E C3Z 120.9(2)
164 C3F C3E C3Z 120.4(2)
165 C3A C3F C3E 122.3(2)
166 C3A C3F H3F 118.9
167 C3E C3F H3F 118.8
168 H3H1 C3H H3H2 107.9
169 H3H1 C3H C3I 109.0
170 H3H1 C3H O3G 108.9
171 H3H2 C3H C3I 109.0
172 H3H2 C3H O3G 108.9
173 C3I C3H O3G 113.1(2)
174 C3H C3I H3I1 109.5
175 C3H C3I H3I2 109.5
176 C3H C3I C3L 110.9(2)
177 H3I1 C3I H3I2 108.0
178 H3I1 C3I C3L 109.5
179 H3I2 C3I C3L 109.4
180 C3I C3L H3L1 109.4
181 C3I C3L H3L2 109.4
182 C3I C3L H3L3 109.5
183 H3L1 C3L H3L2 109.4
184 H3L1 C3L H3L3 109.5
185 H3L2 C3L H3L3 109.5
186 C3A C3M C3N 109.1(2)
187 C3A C3M C3O 112.2(2)
188 C3A C3M C3P 109.5(2)
189 C3N C3M C3O 107.2(2)
190 C3N C3M C3P 110.1(2)
191 C3O C3M C3P 108.7(2)
192 C3M C3N H3N1 109.4
193 C3M C3N H3N2 109.4

194 C3M C3N H3N3 109.5
195 H3N1 C3N H3N2 109.5
196 H3N1 C3N H3N3 109.5
197 H3N2 C3N H3N3 109.5
198 C3M C3O H3O1 109.5
199 C3M C3O H3O2 109.5
200 C3M C3O H3O3 109.5
201 H3O1 C3O H3O2 109.4
202 H3O1 C3O H3O3 109.5
203 H3O2 C3O H3O3 109.4
204 C3M C3P H3P1 109.5
205 C3M C3P H3P2 109.5
206 C3M C3P H3P3 109.5
207 H3P1 C3P H3P2 109.4
208 H3P1 C3P H3P3 109.5
209 H3P2 C3P H3P3 109.4
210 C3E C3Z H3Z1 109.7
211 C3E C3Z H3Z2 109.6
212 C3E C3Z C4E 110.3(2)
213 H3Z1 C3Z H3Z2 108.1
214 H3Z1 C3Z C4E 109.6
215 H3Z2 C3Z C4E 109.6
216 C4B C4A C4F 117.3(2)
217 C4B C4A C4M 122.5(2)
218 C4F C4A C4M 120.2(2)
219 C4A C4B H4B 118.9
220 C4A C4B C4C 122.1(2)
221 H4B C4B C4C 119.0
222 C4B C4C C4D 118.7(2)
223 C4B C4C C4Z 119.9(2)
224 C4D C4C C4Z 121.4(2)
225 C4C C4D C4E 120.5(2)
226 C4C C4D O4G 119.2(2)
227 C4E C4D O4G 120.3(2)
228 C3Z C4E C4D 122.1(2)
229 C3Z C4E C4F 119.3(2)
230 C4D C4E C4F 118.4(2)
231 C4A C4F C4E 122.4(2)
232 C4A C4F H4F 118.9
233 C4E C4F H4F 118.7
234 H4H1 C4H H4H2 107.6
235 H4H1 C4H C4I 108.6
236 H4H1 C4H O4G 108.6
237 H4H2 C4H C4I 108.7
238 H4H2 C4H O4G 108.6
239 C4I C4H O4G 114.5(2)
240 C4H C4I H4I1 109.6
241 C4H C4I H4I2 109.7
242 C4H C4I C4L 110.1(2)
243 H4I1 C4I H4I2 108.2
244 H4I1 C4I C4L 109.6

245 H4I2 C4I C4L 109.7
246 C4I C4L H4L1 109.4
247 C4I C4L H4L2 109.4
248 C4I C4L H4L3 109.5
249 H4L1 C4L H4L2 109.6
250 H4L1 C4L H4L3 109.5
251 H4L2 C4L H4L3 109.4
252 C4A C4M C4N 110.7(2)
253 C4A C4M C4OA 109.1(3)
254 C4A C4M C4PA 113.4(3)
255 C4N C4M C4OA 104.0(3)
256 C4N C4M C4PA 112.4(3)
257 C4OA C4M C4PA 106.7(3)
258 C4M C4N H4N1 109.4
259 C4M C4N H4N2 109.6
260 C4M C4N H4N3 109.5
261 H4N1 C4N H4N2 109.5
262 H4N1 C4N H4N3 109.5
263 H4N2 C4N H4N3 109.3
264 C4M C4OA H4O1 109.5
265 C4M C4OA H4O2 109.4
266 C4M C4OA H4O3 109.5
267 H4O1 C4OA H4O2 109.5
268 H4O1 C4OA H4O3 109.6
269 H4O2 C4OA H4O3 109.3
270 C4M C4PA H4P1 109.5
271 C4M C4PA H4P2 109.5
272 C4M C4PA H4P3 109.4
273 H4P1 C4PA H4P2 109.4
274 H4P1 C4PA H4P3 109.5
275 H4P2 C4PA H4P3 109.6
276 C1C C4Z C4C 111.4(2)
277 C1C C4Z H4Z1 109.4
278 C1C C4Z H4Z2 109.3
279 C4C C4Z H4Z1 109.4
280 C4C C4Z H4Z2 109.4
281 H4Z1 C4Z H4Z2 107.9
282 C1M N1N C1O 128.4(2)
283 C1M N1N H1N 115.8
284 C1O N1N H1N 115.8
285 C1I N1Q C1O 120.8(2)
286 C1I N1Q C1R 121.5(2)
287 C1O N1Q C1R 117.6(2)
288 C1D O1G C1H 112.8(1)
289 C2D O2G C2H 112.0(2)
290 C3D O3G C3H 112.2(1)
291 C4D O4G C4H 113.5(1)
292 H5A1 C5A H5A2 108.0
293 H5A1 C5A C5B 109.4
294 H5A1 C5A C5F 109.5
295 H5A2 C5A C5B 109.5

296 H5A2 C5A C5F 109.5
297 C5B C5A C5F 111.0(3)
298 C5A C5B H5B1 109.3
299 C5A C5B H5B2 109.4
300 C5A C5B C5C 111.7(3)
301 H5B1 C5B H5B2 107.9
302 H5B1 C5B C5C 109.3
303 H5B2 C5B C5C 109.2
304 C5B C5C H5C1 109.5
305 C5B C5C H5C2 109.5
306 C5B C5C C5D 110.7(3)
307 H5C1 C5C H5C2 108.0
308 H5C1 C5C C5D 109.5
309 H5C2 C5C C5D 109.5
310 C5C C5D H5D1 109.3
311 C5C C5D H5D2 109.3
312 C5C C5D C5E 111.5(3)
313 H5D1 C5D H5D2 108.0
314 H5D1 C5D C5E 109.4
315 H5D2 C5D C5E 109.3
316 C5D C5E H5E1 109.4
317 C5D C5E H5E2 109.4
318 C5D C5E C5F 111.1(3)
319 H5E1 C5E H5E2 108.0
320 H5E1 C5E C5F 109.4
321 H5E2 C5E C5F 109.4
322 C5A C5F C5E 111.2(3)
323 C5A C5F H5F1 109.3
324 C5A C5F H5F2 109.4
325 C5E C5F H5F1 109.4
326 C5E C5F H5F2 109.5
327 H5F1 C5F H5F2 108.0
328 H1U1 C1UA H1U2 110
329 H1U1 C1UA H1U3 109
330 H1U2 C1UA H1U3 110
331 H6A1 C6A H6A2 110.2
332 H6A1 C6A C6B 112.6
333 H6A1 C6A C6FA 112.7
334 H6A2 C6A C6B 112.7
335 H6A2 C6A C6FA 112.7
336 C6B C6A C6FA 95.4(9)
337 C6A C6B H6B1 110
338 C6A C6B H6B2 110
339 C6A C6B C6CA 107.8(9)
340 H6B1 C6B H6B2 108
341 H6B1 C6B C6CA 110
342 H6B2 C6B C6CA 110
343 C6B C6CA H6C1 112
344 C6B C6CA H6C2 111
345 C6B C6CA C6DA 100.7(9)
346 H6C1 C6CA H6C2 110

347 H6C1 C6CA C6DA 112
348 H6C2 C6CA C6DA 112
349 C6CA C6DA H6D1 110
350 C6CA C6DA H6D2 110
351 C6CA C6DA C6EA 110(1)
352 H6D1 C6DA H6D2 108
353 H6D1 C6DA C6EA 110
354 H6D2 C6DA C6EA 110
355 C6DA C6EA H6E1 111
356 C6DA C6EA H6E2 111
357 C6DA C6EA C6FA 105(1)
358 H6E1 C6EA H6E2 109
359 H6E1 C6EA C6FA 111
360 H6E2 C6EA C6FA 111
361 C6A C6FA C6EA 110(1)
362 C6A C6FA H6F1 110
363 C6A C6FA H6F2 110
364 C6EA C6FA H6F1 110
365 C6EA C6FA H6F2 110
366 H6F1 C6FA H6F2 108