Electronic Supplementary Information (ESI)

Synthesis, photophysical, electrochemical, and electrochemiluminescent properties of 5,15-bis(9-anthracenyl)porphyrins derivatives**

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Synthesis of **4a**. A solution of PhMe (6 mL) containing 9-bromoanthracene (300 mg, 1.17 mmol, 1 equiv.), bis(pinacolato)diboron (328 mg, 1.29 mmol, 1,1 equiv.), KOAc (1.15 g, 11.7 mmol, 10 equiv.) and [Pd(PPh$_3$)$_4$] (3 mg) was refluxed overnight under Ar. The catalyst was removed by filtration through Celite and the solvent evaporated *in vacuo*. The product was purified by FCC on silica gel (CH$_2$Cl$_2$/cyclohexane 50/50 + 1% NEt$_3$). The solid was re-dissolved in CH$_2$Cl$_2$ and precipitated upon addition of hexane to afford **4a** as a white solid (202 mg, 0.664 mmol, 57% yield). $C_{20}H_{21}BO_2$ (MW 304.2); IR (KBr) 3050, 2979, 1809, 1710, 1435, 1365, 1305, 1242, 1149, 989, 848, 744 cm^{-1}; ^{1}H-NMR (200 MHz, CDCl$_3$) *d* 1.57 (s, 12H, CH$_3$), 7.46 (m, 4H), 7.99 (d, $J = 9.2$ Hz, 2H), 8.45 (d, $J = 9.8$ Hz, 2H), 8.47 (s, 1H); ^{13}C-NMR (50 MHz, CDCl$_3$) *d* 25.23, 84.37, 124.81, 125.72, 128.25, 128.74, 129.43, 131.07, 135.84; UV-VIS λ_{max} (PhMe)/nm: 333 ($\varepsilon = 2910$ M^{-1}cm^{-1}), 347 ($\varepsilon = 5625$ M^{-1}cm^{-1}) 365 ($\varepsilon = 8300$ M^{-1}cm^{-1}), 384 ($\varepsilon = 7280$ M^{-1}cm^{-1}); ESI-MS *m/z*: 305.2 ([M+H]$^+$).

Synthesis of **1a**. Anthracene boronate **4a** (50 mg, 0.16 mmol, 4 equiv.), diiodoporphyrin **3** (40 mg, 0,04 mmol, 1 equiv.) and CsCO$_3$ (40 mg, 0.12 mmol, 3 equiv.) were dissolved in a mixture of DMF (2 mL) and dry PhMe (4 mL). The solution was deoxygenated by bubbling of Ar for 20 minutes and [Pd(PPh$_3$)$_4$] (5 mg) was added. The resulting mixture was heated at

130°C for 4h under Ar. The reaction was quenched by addition of H_2O and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and the solvent was evaporated in vauco. The crude mixture was then purified by FC on silica gel. Two compounds were isolated, the di-substituted porphyrin **1a** (hexane/EtOAc 90/10 + 1% NEt_3), and a fraction of mono-substituted anthracenyl-porphyrin (hexane/EtOAc 95/5 + 1% NEt_3). Compound **1a** was dissolved in CH_2Cl_2 and precipitated upon addition of MeOH to afford a violet solid (5 mg, 0.0045 mmol, 12% yield). $C_{76}H_{68}N_4Zn$ (MW 1102.8); IR (KBr) 3045, 2954, 2553, 2310, 1728, 1593, 1468, 1311, 997, 796, 729 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.44 (s, 36H, *t*Bu), 7.03 (dd, J = 6.4 and 1.2 Hz, 4H), 7.12 (d, J = 8.9 Hz, 4H), 7.46 (dd, J = 7.5 and 1.0 Hz, 4H), 7.70 (t, J = 1.7 Hz, 2H), 8.05 (d, J = 2.0 Hz, 4H), 8.29 (d, J = 8.8 Hz, 4H), 8.36 (d, J = 4.9 Hz, 4H, H_{b2}), 8.81 (d, J = 4.6 Hz, 4H, H_{b1}), 8.92 (s, 2H); ^{13}C-NMR (50 MHz, $CDCl_3$) δ 32.06, 35.35, 115.72, 121.05, 122.72, 125.27, 125.91, 128.02, 128.54, 129.13, 129.95, 131.15, 132.03, 133.26, 135.51, 137.11, 141.72, 148.77, 150.84, 151.56;_ UV-VIS λ_{max} (PhMe)/nm 426 (ε = 145240 $M^{-1}cm^{-1}$), 547 (ε = 9950 $M^{-1}cm^{-1}$), 586 (ε = 1230 $M^{-1}cm^{-1}$); ESI-MS *m/z*: 1104.0 ($[M+H]^+$).

Synthesis of **6**. A solution of PhMe (10 mL) containing 4-bromo-*N,N*-dimethylaniline (300 mg, 1.50 mmol, 1 equiv.), bis(pinacolato)diboron (419 mg, 1.65 mmol, 1.1 equiv.), KOAc (1.48 g, 15 mmol, 10 equiv.) and $[Pd(PPh_3)_4]$ (10 mg) was refluxed overnight under Ar. The catalyst was removed by filtration through Celite and the solvent evaporated *in vacuo*. The product was purified by FCC on silica gel (CH_2Cl_2/cyclohexane 20/80 + 1% NEt_3) to afford compound **6** as a white solid (192 mg, 0.777 mmol, 52% yield). $C_{14}H_{22}BNO_2$ (MW 247.1); IR (KBr) 2977, 2916, 1606, 1535, 1465, 1362, 1138, 953, 841 cm^{-1}; ^{1}H-NMR (200 MHz, $CDCl_3$) δ 1.32 (s, 12H, CH_3), 2.99 (s, 6H, CH_3 amino), 6.69 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8,2 Hz, 2H); ^{13}C-NMR (50 MHz, $CDCl_3$) *d* 24.83, 25.01, 40.08, 83.07, 83.43, 111.12, 136.01; ESI-MS *m/z*: 248.2 ($[M+H]^+$).

Synthesis of **5b**. Compound **6** (120 mg, 0.49 mmol, 1 equiv.), 9,10-dibromoanthracene (650 mg, 1.94 mmol, 4 equiv.) and Cs_2CO_3 (242 mg, 0.74 mmol, 1.5 equiv.) were dissolved in dry PhMe (30 mL). The solution was deoxygenated by bubbling Ar for 10 minutes and $[Pd(PPh_3)_4]$(15 mg) added. The resulting mixture was heated at 120°C overnight under Ar. The catalyst was removed by filtration through Celite and the solvent evaporated *in vacuo*. The product was purified by FCC on silica gel (cyclohexane/$CHCl_3$ 10/90 + 1% NEt_3). Compound **5b** was isolated as a pale yellow solid (105 mg, 0.279 mmol, 57% yield). $C_{22}H_{18}BrN$ (MW 376.3); IR (KBr) 3049, 2927, 2802, 1608, 1523, 1444, 1362, 1217, 802, 739 cm^{-1}; ^{1}H-NMR (200 MHz, $CDCl_3$) δ 3.10 (s, 6H, CH_3), 6.93 (d, J = 8.4 Hz, 2H), 7.32 (d, J =Hz, 2H), 7.38 (t, J =Hz, 2H), 7.59 (t, J = 8.8 Hz, 2H), 7.80 (d, J = 8.4 Hz, 2H), 8,59 (d, J = 8.6 Hz, 2H); ESI-MS *m/z*: 376.4 ($[M]^+$).

Synthesis of **4b**. A solution of PhMe (20 mL) containing **5b** (105 mg, 0.280 mmol, 1 equiv.), bis(pinacolato)diboron (86 mg, 0.335 mmol, 1.2 equiv.), KOAc (275 mg, 2.80 mmol, 10 equiv.) and $[Pd(PPh_3)_4]$ (10 mg) was refluxed overnight under Ar. The catalyst was removed by filtration through Celite and the solvent evaporated *in vacuo*. The product was purified by FCC on silica gel (CH_2Cl_2/cyclohexane 60/40 + 1% NEt_3). The solid was dissolved in CH_2Cl_2 and precipitated upon addition of hexane to afford **4b** as a yellow solid (55 mg, 0.130 mmol, 48% yield). $C_{28}H_{30}BNO_2$ (MW 423.3); IR (KBr) 3049, 2981, 2923, 2808, 1608, 1523, 1429, 1313, 1230, 1142, 968, 769 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.59 (s, 12H, CH_3 boron), 3.08 (s, 6H, CH_3 amino), 6.92 (d, J = 8.8 Hz, 2H), 7.26 (d, J = 8,7 Hz, 2H), 7.30 (m, 2H), 7.44 (td, J = 8.8 and 1.2 Hz, 2H), 7.76 (d, J = 8.6 Hz, 2H), 8.41 (d, J = 8.8 Hz, 2H); ^{13}C-NMR (50 MHz, $CDCl_3$) δ 25.27, 40.68, 84.36, 112.13, 124.42, 125.26, 126.66, 127.75, 128.26, 130.21, 131.81, 135.47, 149.70; UV-VIS λ_{max} (PhMe)/nm : 358 (ε = 5130 $M^{-1}cm^{-1}$), 375 (ε = 8300 $M^{-1}cm^{-1}$), 394 (ε = 9295 $M^{-1}cm^{-1}$); ESI-MS *m/z*: 424.2 ($[M+H]^+$).

Synthesis of **1b**. Compound **4b** (50 mg, 0.118 mmol, 4 equiv.), diiodoporphyrin **2** (30 mg, 0,030 mmol, 1 equiv.) and $CsCO_3$ (30 mg, 0.090 mmol, 3 equiv.) were dissolved in a mixture of DMF (2 mL) and dry PhMe (4 mL). The solution was deoxygenated by bubbling Ar for 20 minutes and $[Pd(PPh_3)_4]$ (4 mg) added. The resulting mixture was heated at 130°C overnight under Ar. The reaction was quenched upon addition of H_2O and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and the solvent evaporated *in vacuo*. The crude mixture then purified by FCC on silica gel. Two compounds were isolated, di-substituted porphyrin **1b** (hexane/EtOAc 90/10 + 1% NEt_3), and a mono-substituted anthracenyl-porphyrin (hexane/EtOAc 95/5 + 1% NEt_3). Compound **1b** was dissolved in CH_2Cl_2 and precipitated upon addition of MeOH to afford a violet solid (4 mg, 0.003 mmol, 10% yield). $C_{92}H_{86}N_6Zn$ (MW 1341.1); IR (KBr) 3454, 3141, 3049, 2954, 2536, 2287, 1934, 1712, 1603, 1510, 1354, 1215, 945, 818 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$) *d* 1.45 (s, 36H, *t*Bu), 3.16 (s, 12H, CH_3 amino), 6.98 (m, 4H), 7.09 (d, *J* = 8.7 Hz, 4H), 7.16 (d, *J* = 9.0 Hz, 4H), 7.31 (m, 4H), 7.65 (d, *J* = 8.6 Hz, 4H), 7.71 (t, *J* = 2.0 Hz, 2H, H_p), 8.07 (d, *J* = 1.7 Hz, 4H, H_o), 8.09 (d, *J* = 7.8 Hz, 4H), 8.48 (d, *J* = 4.6 Hz, 4H, H_{b2}), 8.84 (d, *J* = 4.6 Hz, 4H, H_{b1}); UV-VIS λ_{max} (PhMe)/nm 427 (ε = 169615 $M^{-1}cm^{-1}$), 548(ε =14259 $M^{-1}cm^{-1}$), 586(ε = 2121 $M^{-1}cm^{-1}$); ESI-MS *m/z*: 1342.2 ($[M+H]^+$).

Synthesis of **9**. Compound **6** (70 mg, 0.283 mmol, 4 equiv.), diiodoporphyrin **3** (70 mg, 0.071 mmol, 1 equiv.) and Cs_2CO_3 (36 mg, 0.110 mmol, 1.5 equiv.) were dissolved in a mixture of DMF (4 mL) and dry toluene (8 mL). The solution was deoxygenated by bubbling of Ar for 20 minutes and $[Pd(PPh_3)_4]$ catalyst (8 mg) added. The resulting mixture was heated at 130°C overnight under Ar. The catalyst was removed by filtration through Celite and the solvent evaporated *in vacuo*. The crude mixture was purified by flash column chromatography on silica gel, (eluent CH_2Cl_2 / cyclohexane 60/40) and precipitated by addition of MeOH to afford **9** as a violet solid (48 mg, 0.049 mmol, 67 % yield) : IR (KBr) 3049, 2954, 2875, 1599, 1508, 1348, 1205, 1005, 939, 808 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$) δ 1.55 (s, 36H, tBu), 3.22 (s, 12H, CH_3 amino), 7.10 (d, *J* = 8.4 Hz, 4H), 7.78 (s, 2H, H_p), 8.09 (d, *J* = 6.5 Hz, 4H), 8.10 (s, 4H, H_o), 8.96 (d, *J* = 4.4 Hz, 4H, $H_{\beta1}$), 9.04 (d, *J* = 4.8 Hz, 2H, $H_{\beta2}$); FABMS *m/z* [M$^+$]; 988.2 $C_{64}H_{70}N_6Zn$ (MW 988.7).

X-ray data for porphyrin 1a. Red prismatic crystals, 0.2×0.3×0.4 mm in dimension, grown from a solution of Dichloromethane. X-ray of Anode Cu k/α radiation was used (monochromatic wavelength λ = 1.54 Å) using a Mar CCD detector. The crystal soaked with PEG 200 was mounted in a loop and flash frozen to 100 K with a nitrogen stream. The diffraction data were indexed and integrated using DENZO[1] and scaled with SCALEPACK[1]. The structure was solved by direct methods using SHELXS[3] and Fourier analyses and refined by the full-matrix least-squares based on F^2 using SHELXL-97[3]. The electron density maps and the refinement of the structure revealed in the asymmetric unit the presence of half functionalised porphyrine with a Zinc ion coordinated in the centre of the tetrapyrrole ring with an average distance of 2.04 Å from the four nitrogen atoms. The unit cell is triclinic with P-1 symmetry and contains one molecular porphyrin unit. It is symmetry related by a symmetry centre positioned on the Zinc anion. Both the planes of anthracene molecules and of aryl groups are perpendicular to the porphyrin plane. The refinement was performed introducing in the asymmetric unit 2,5 dichloromethane molecules from solvent. In the final refinement, all non-hydrogen atoms were treated anisotropically and the hydrogen atoms were included at calculated positions with isotropic *U* factors = $1.2U_{eq}$.

Table S1. Essential crystal and refinement data.

Formula	$(C_{76}H_{68}N_4Zn)\cdot5(CH_2Cl_2)$

F w	1527.34
T, K	100 (2)
λ, Å	1.542
Cryst. system, space group	Triclinic, *P -1*
a, b, c Å	9.0583(4) 15.2181(7) 15.4705(5)
α,, β,, ,γ deg	114.522(2) 106.688(2) 91.036(3)
V, Å^3	1835.33(13)
Z, D$_{calc}$	1, 1.382mg/m^3
μ, mm^{-1}	4.18
F(000)	792
λ/2senθ_{max}, Å	0.90
Reflns measured	5014
Reflns unique	5010
Reflns *I*>2.0s(*I*)	3934
Rmerge	0.099 (0.139, in shell 0.93 Å-0.90 Å)
Params	469
restraints	0
G.o.f.	1.075
R [I>2s(I)]	R1 = 0.0641, wR2 = 0.1839
R (all data)	R1 = 0.0782, wR2 = 0.1943

X-ray data for porphyrin 1b. Red prismatic crystals, 0.1×0.1×0.05 mm in dimension, grown from a solution of Chloroform, Dichloromethane and Pyridine. The crystals used for diffraction experiment were totally dried. X-ray of Synchrotron radiation was used (monochromatic wavelength λ = 1.0000 Å) using a Mar CCD detector with the rotating crystal method. The crystal soaked with PEG 200 was mounted in a loop and flash frozen to 100 K with a nitrogen stream. The diffraction data were indexed and integrated using DENZO[1] and scaled with SCALEPACK[1]. The structure was solved by direct methods using Sir92[3] and Fourier analyses and refined by the full-matrix least-squares based on F^2 using SHELXL-97[4]. In the final refinement, all non-hydrogen atoms were treated anisotropically and the hydrogen atoms of the porphyrin were included at calculated positions with isotropic U factors = $1.2U_{eq}$, while the hydrogen atoms of the solvent were introduced in restrained positions, with isotropic U factors = $1.2U_{eq}$.

Table S2. Essential crystal and refinement data.

Formula	$(C_{92}H_{86}N_6Zn)(C_5H_5N)\cdot 3.25(CHCl_3)$
F w	1808.1
T, K	100 (2)
λ, Å	1.000
Cryst. system, space group	Monoclinic, *C 2/c*
a, b, c Å	42.923(1) 18.262(1) 29.806(1)
β deg	129.10(1)
V, $Å^3$	18130(3)
Z, D_{calc}	8, 1.325mg/m^3
μ, mm^{-1}	1.494
F(000)	7524
λ/2senθ_{max} , Å	0.95
Reflns measured	10329
Reflns unique	10045

Reflns *I*>2.0s(*I*)	9060
Rmerge	0.038 (0.123, in shell 0.98 Å-0.95 Å)
Params	1154
restraints	50
G.o.f.	1.029
R [I>2s(I)]	R1 = 0.0776, wR2 = 0.2111
R (all data)	R1 = 0.0853, wR2 = 0.2219

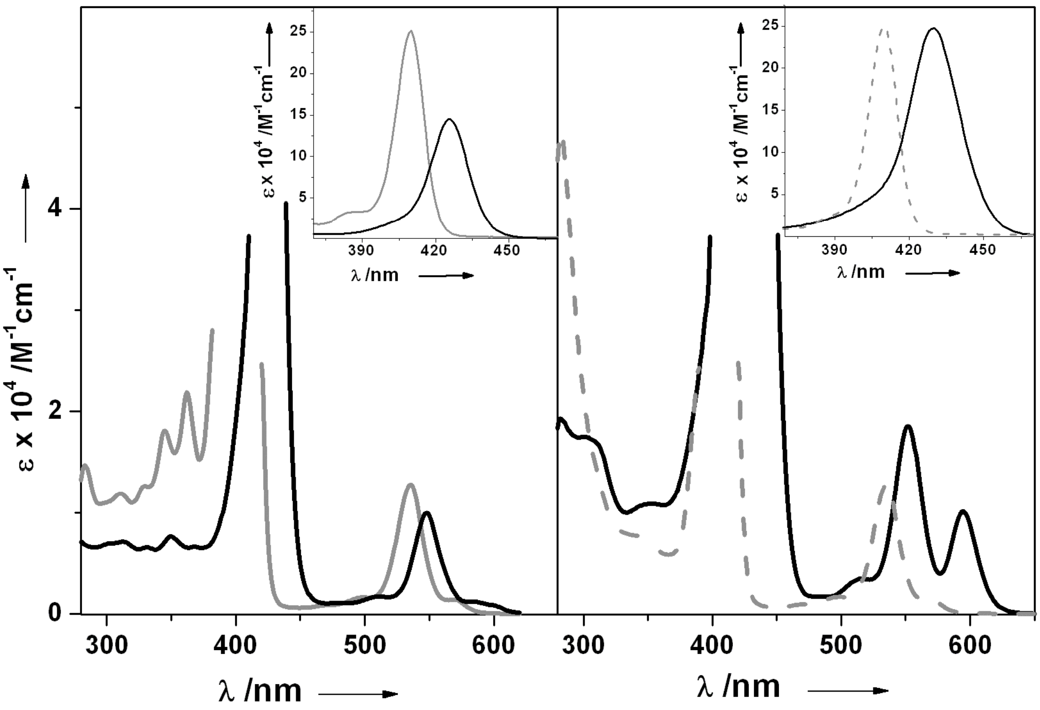


Figure S1. Left panel: Comparison of the absorption spectrum of **1a** (black) relative to the sum of its component units (**3** plus 2×**4a**, grey) at 298 K in PhMe. Right panel: Comparison of the absorption spectrum of **7** (black) relative to the sum of its component units (**3** plus 2×**6**, grey); 298 K in PhMe.

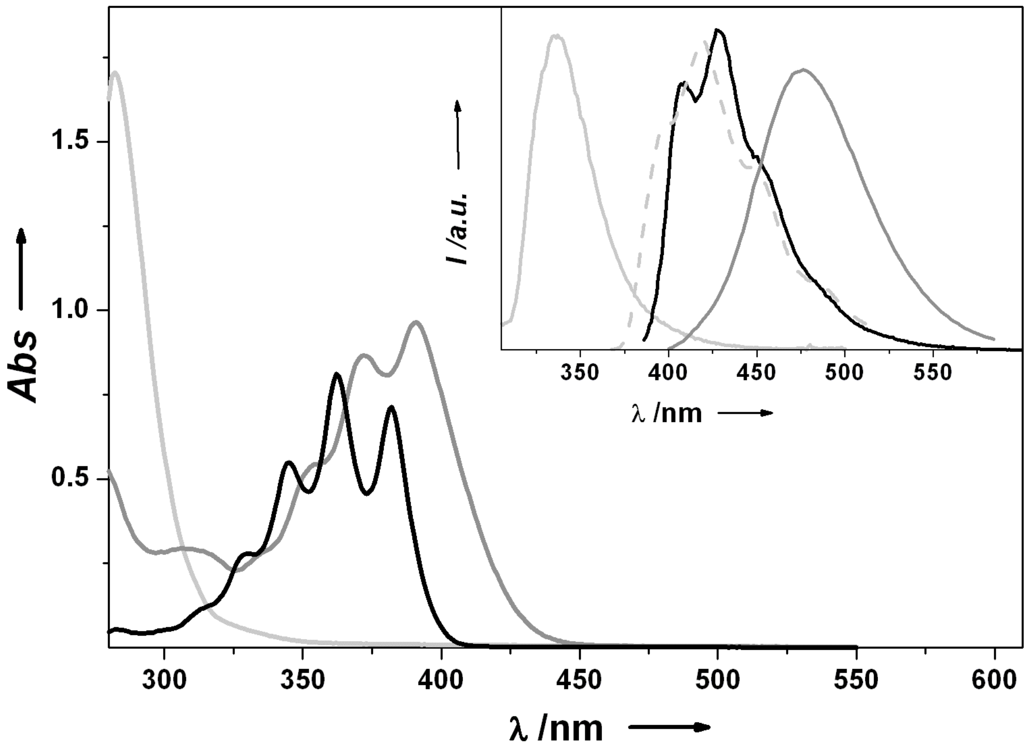


Figure S2. Absorption and (inset) emission spectra (fluorescence, 298 K, full lines; phosphorescence, 77 K, dashed line) of **5a** (black), **4b** (grey), **6** (light grey) recorded in PhMe.

Table S3. Selected structural parameters (bonds (Å) and dihedral angles (deg)) of complexes **1a-b** in the neutral, single and double oxidation states. All structures in table (a) are optimised with BLYP [5,6] exchange correlation functional, those in table (b) with the B3LYP [6,7] exchange correlation functional. Zn-N distance is the average distance over the four bonds. For atom labelling see Figure S5. With a and b, we refer to the C atoms which are proximal to the labelled carbon on the rings and that points towards the reader and in the opposite direction, respectively.

Atoms	**1a**	**1a$^+$**	**1a^{2+}**	**1b**	**1b$^+$**	**1b^{2+}**	**1b$_{qc}^{2+}$**	**1b$_{qt}^{2+}$**
Bonds lengths (Å)								
Zn-N	2.07	2.06	2.06	2.07	2.04	2.07	2.09	2.08
C1-C2	1.51	1.51	1.51	1.51	1.50	1.51	1.40	1.40
C1-C2’	1.51	1.51	1.51	1.51	1.50	1.51	1.40	1.40
C3-C4				1.50	1.50	1.48	1.45	1.45
C3-C4’				1.50	1.50	1.49	1.45	1.45
Dihedral angles (deg)								
C1a-C1-C2aC2	89	87	86	90	90	92	2	4
C1b-C1-C2b-C2	90	88	88	90	90	92	3	-6
C3a-C3-C4-C4a				107	114	115	-3	1
C3b-C3-C4-C4b				107	114	115	2	-6
C1a’-C1’-C2a-C2	91	88	88	90	89	88	6	4

C1b'-C1'-C2b-C2'	90	88	87	90	89	88	5	-6
C3a'-C3'-C4'-C4a'				78	70	70	-1	1
C3b'-C3'-C4'-C4b'				77	71	70	-5	-6

Atoms	**1a**	**1a**$^{+}$	**1a**$^{2+}$	**1b**	**1b**$^{+}$	**1b**$^{2+}$
Bonds lengths (Å)						
Zn-N	2.05	2.05	2.06	2.05	2.05	2.07
C1-C2	1.51	1.50	1.48	1.51	1.51	1.51
C1-C2'	1.51	1.50	1.48	1.51	1.51	1.51
C3-C4				1.50	1.48	1.48
C3-C4'				1.50	1.48	1.48
Dihedral angles (deg)						
C1a-C1-C2aC2	90	72	66	90	90	72
C1b-C1-C2b-C2	90	72	66	90	90	72
C3a-C3-C4-C4a				106	122	128
C3b-C3-C4-C4b				106	122	128
C1a'-C1'-	90	73	66	90	89	108

C2a-C2						
C1b’-C1’-C2b-C2’	90	73	66	90	89	108
C3a’-C3’-C4’-C4a’				73	58	53
C3b’-C3’-C4’-C4b’				73	58	53

Table S4. Selected structural parameters, bonds (Å) and dihedral angles (deg), of complexes **$1b_{qc}^{2+}$** complex in which one H on the tetraphyrroilc and one on the anthracenyl moiety have been progressively removed. The complex in which 8 H atoms have been removed correspond to porphyrin **8**. For atom labelling see Figure S5.

	$1b_{qc}^{2+}$-2H	$1b_{qc}^{2+}$-4H	$1b_{qc}^{2+}$-6H	$1b_{qc}^{2+}$-8H (**8**)
Zn-N	2.07	2.07	2.07	2.06
C1-C2	1.51	1.50	1.44	1.44
C1’-C2’	1.45	1.44	1.44	1.44
C3-C4	1.48	1.50	1.47	1.47

Figure S3a

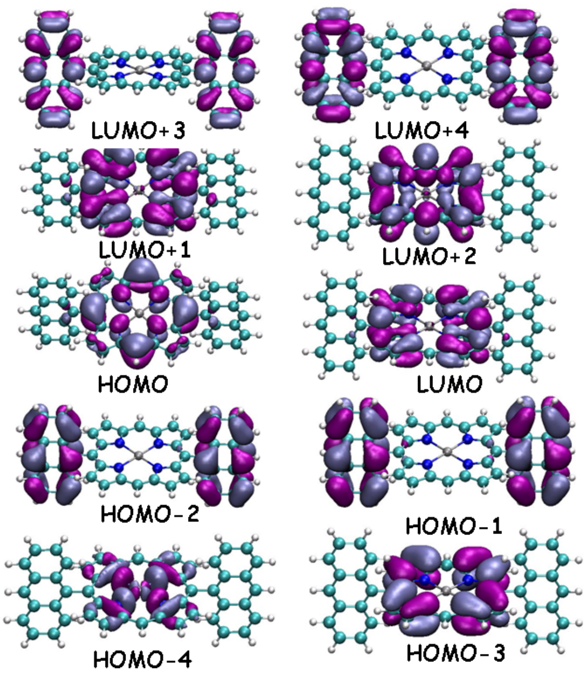


Figure S3b

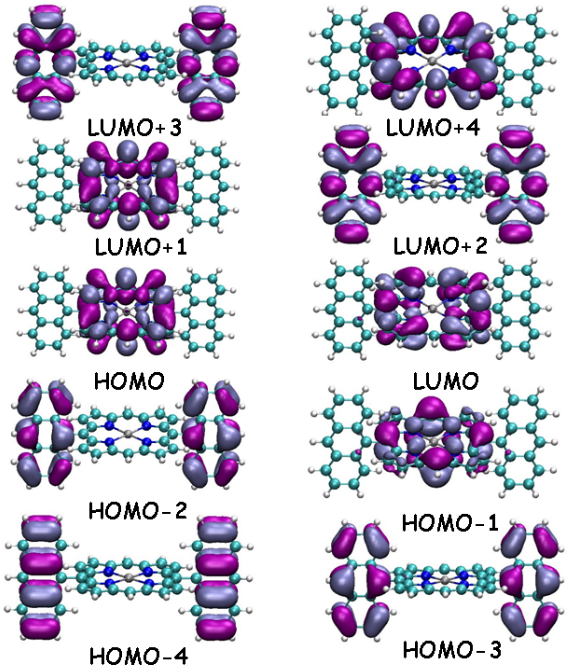


Figure S3. Kohn-Sham orbitals of **1a** calculated with the B3LYP exchange-correlation functional (a) and molecular orbitals calculated at Hartree-Fock level of theory (b). The 3,5-di(*tert*-butyl)phenyl groups have been replaced by H atoms.

Figure S4a

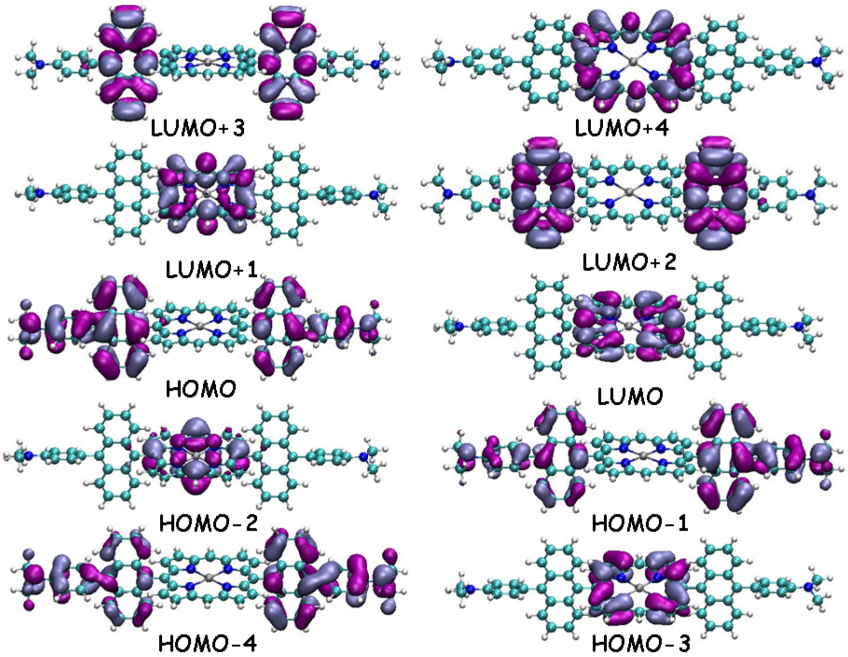


Figure S4b

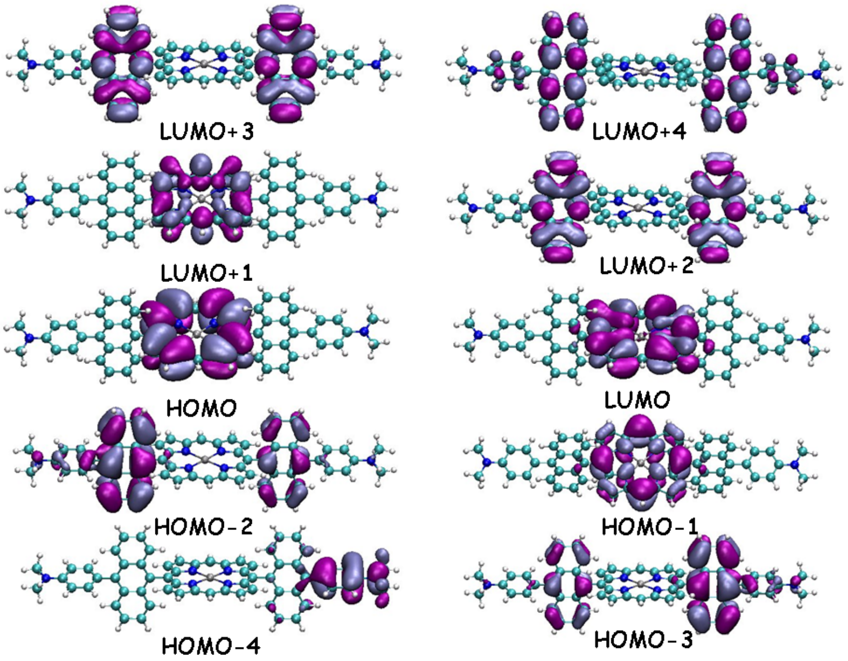
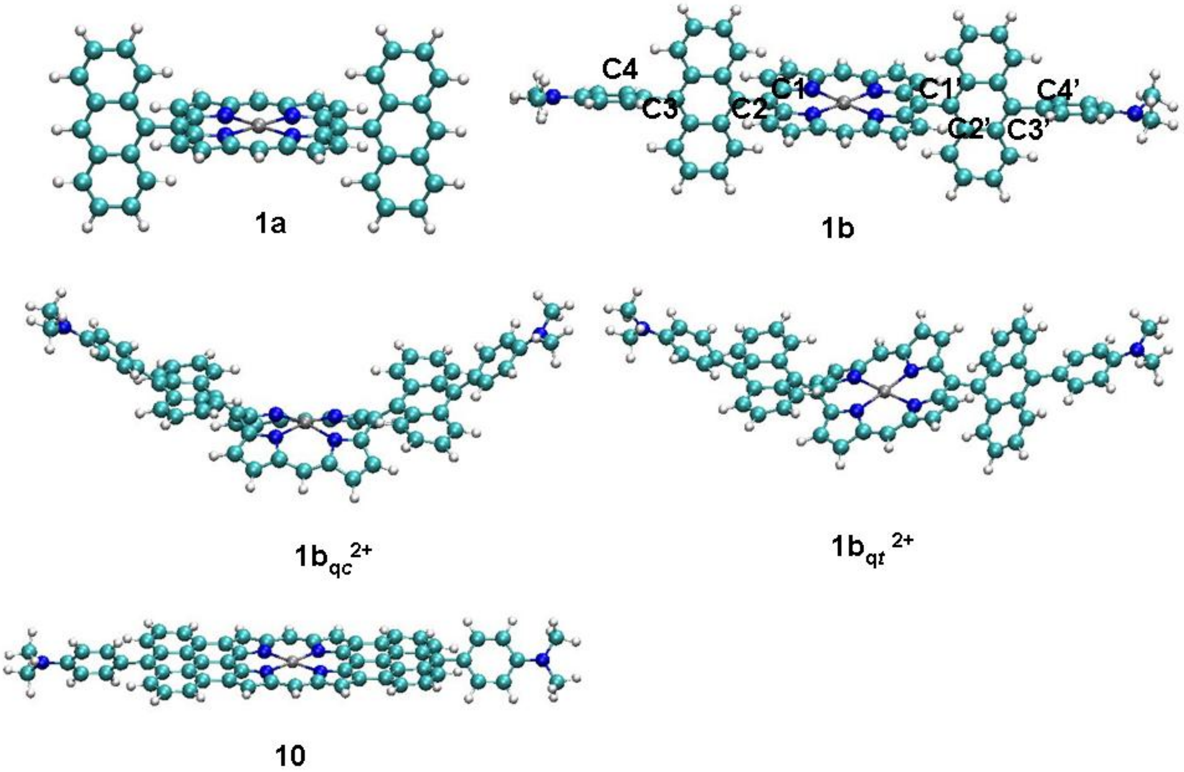


Figure S4. Kohn-Sham orbitals of **1b** calculated with the B3LYP exchange-correlation functional (a) and molecular orbitals calculated at Hartree-Fock level of theory (b). The 3,5-di(*tert*-butyl)phenyl groups have been replaced by H atoms.



1a

1b

1b$_{qc}^{2+}$

1b$_{qt}^{2+}$

10

Figure S5. Optimized structures of **1a**, **1b, 1b^{2+}qc**, **1b^{2+}qc** and **8.**

[1] Z. Otwinowski and W. Minor **1997** *Methods in Enzymology*, Vol. 276,307-326.

[2] G. M. Sheldrick, **1990***, Acta Cryst*. A46, 467-473.

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[4] G. M. Sheldrick, SHELXL-97, **1997**, University of Göttingen.

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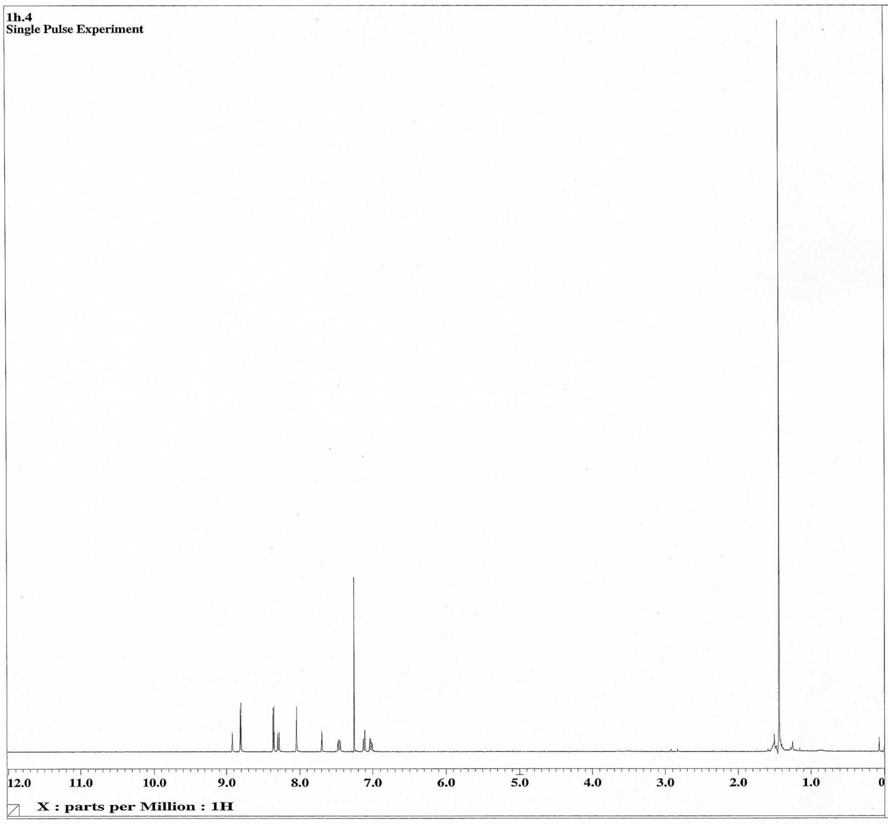
[7] A.D. Becke, J.Chem.Phys. 98 (1993) 5648-5652

^{1}H-NMR characterization spectra for the synthesized compounds.

Compound **1a** (whole range and zoomed region)



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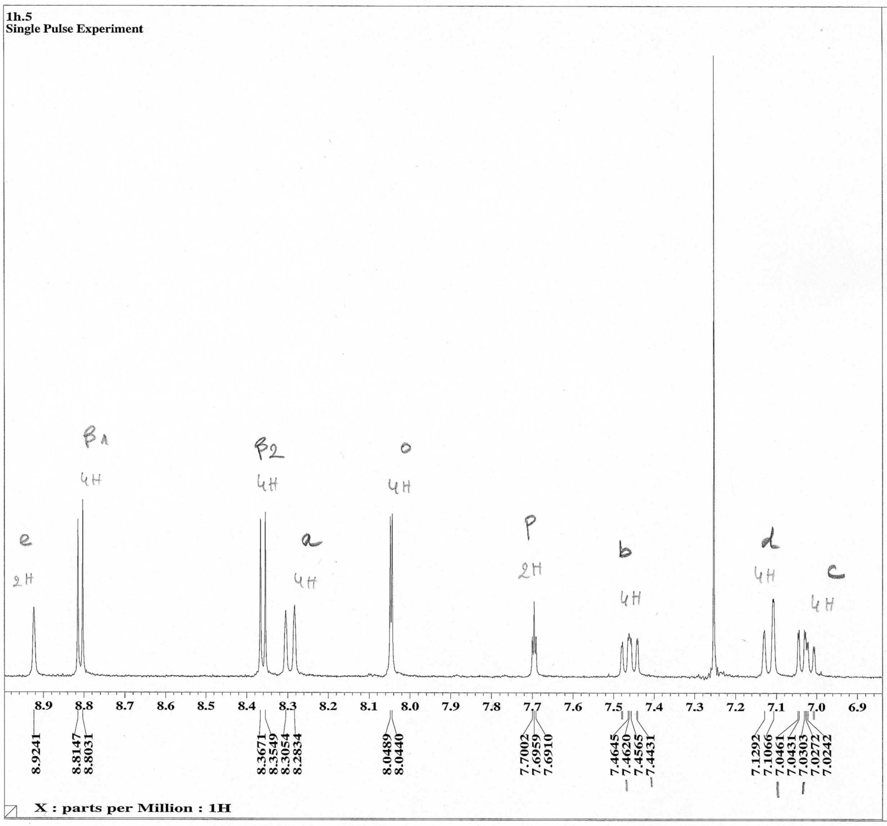
Temp_get           = 20.7[dC]
Temp_set           = 25[dC]
Temp_state         = TEMP OFF
Temp_status        = TEMP OFF
X90                = 12.5[us]
X90_hi             = 1[us]
X90_lo             = 70[us]
X_acq_duration     = 2.048[s]
X_domain           = 1H
X_freq             = 399.7841973[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_pulse            = 6.2[us]
X_resolution       = 0.48831105[Hz]
X_sweep            = 8[kHz]
```

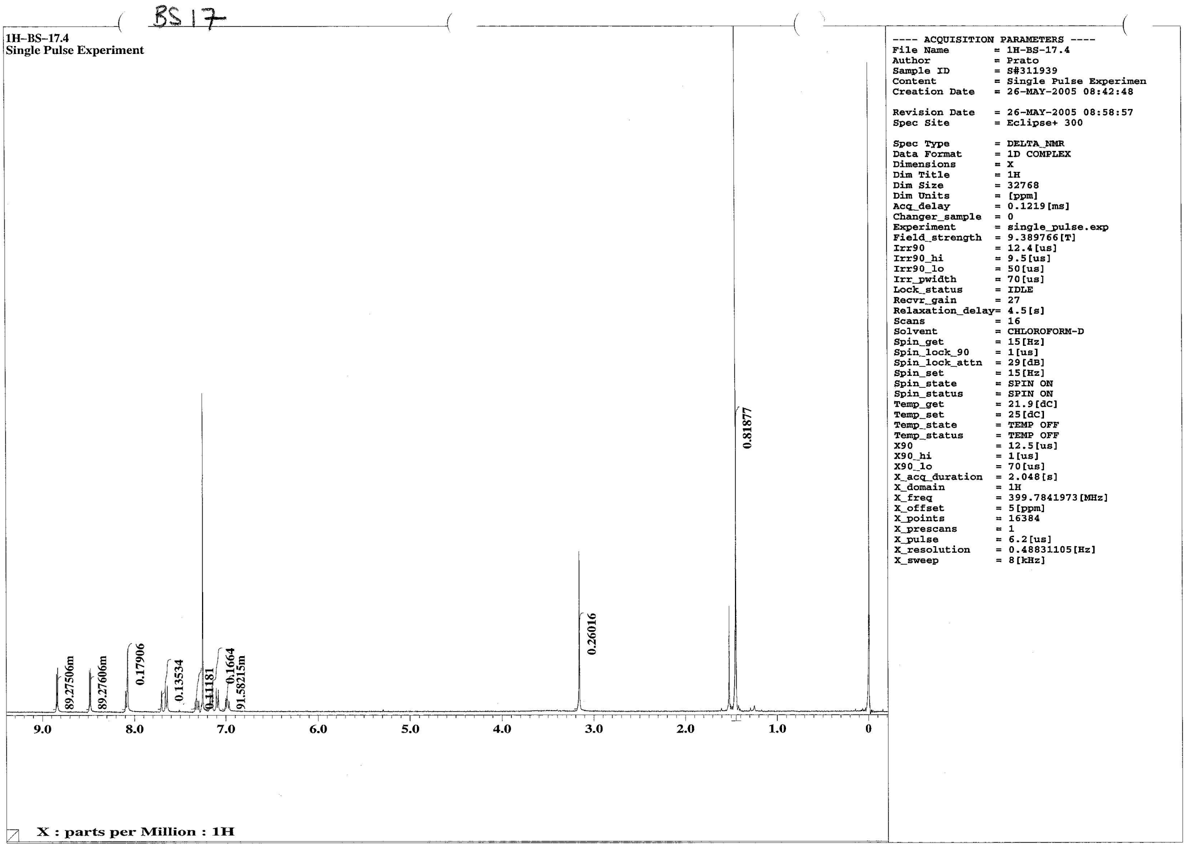


```
---- ACQUISITION PARAMETERS ----
File Name          = 1h.9
Author             = Prato
Sample ID          = davide1
Content            = Single Pulse Experimen
Creation Date      =  7-APR-2005 09:04:06

Revision Date      =  7-APR-2005 09:11:47
Spec Site          = Eclipse+ 300

Spec Type          = DELTA_NMR
Data Format        = 1D COMPLEX
Dimensions         = X
Dim Title          = 1H
Dim Size           = 32768
Dim Units          = [ppm]
Acq_delay          = 0.1219[ms]
Changer_sample     = 0
Experiment         = single_pulse.exp
Field_strength     = 9.389766[T]
Irr90              = 12.5[us]
Irr90_hi           = 1[us]
Irr90_lo           = 70[us]
Irr_pwidth         = 70[us]
Lock_status        = IDLE
Recvr_gain         = 25
Relaxation_delay= 4.5[s]
Scans              = 4
Solvent            = CHLOROFORM-D
Spin_get           = 15[Hz]
Spin_lock_90       = 1[us]
Spin_lock_attn     = 29[dB]
Spin_set           = 15[Hz]
Spin_state         = SPIN ON
Spin_status        = SPIN ON
Temp_get           = 20.7[dC]
Temp_set           = 25[dC]
Temp_state         = TEMP OFF
Temp_status        = TEMP OFF
X90                = 12.5[us]
X90_hi             = 1[us]
X90_lo             = 70[us]
X_acq_duration     = 2.048[s]
X_domain           = 1H
X_freq             = 399.7841973[MHz]
X_offset           = 5[ppm]
X_points           = 16384
X_prescans         = 1
X_pulse            = 6.2[us]
X_resolution       = 0.48831105[Hz]
X_sweep            = 8[kHz]
```

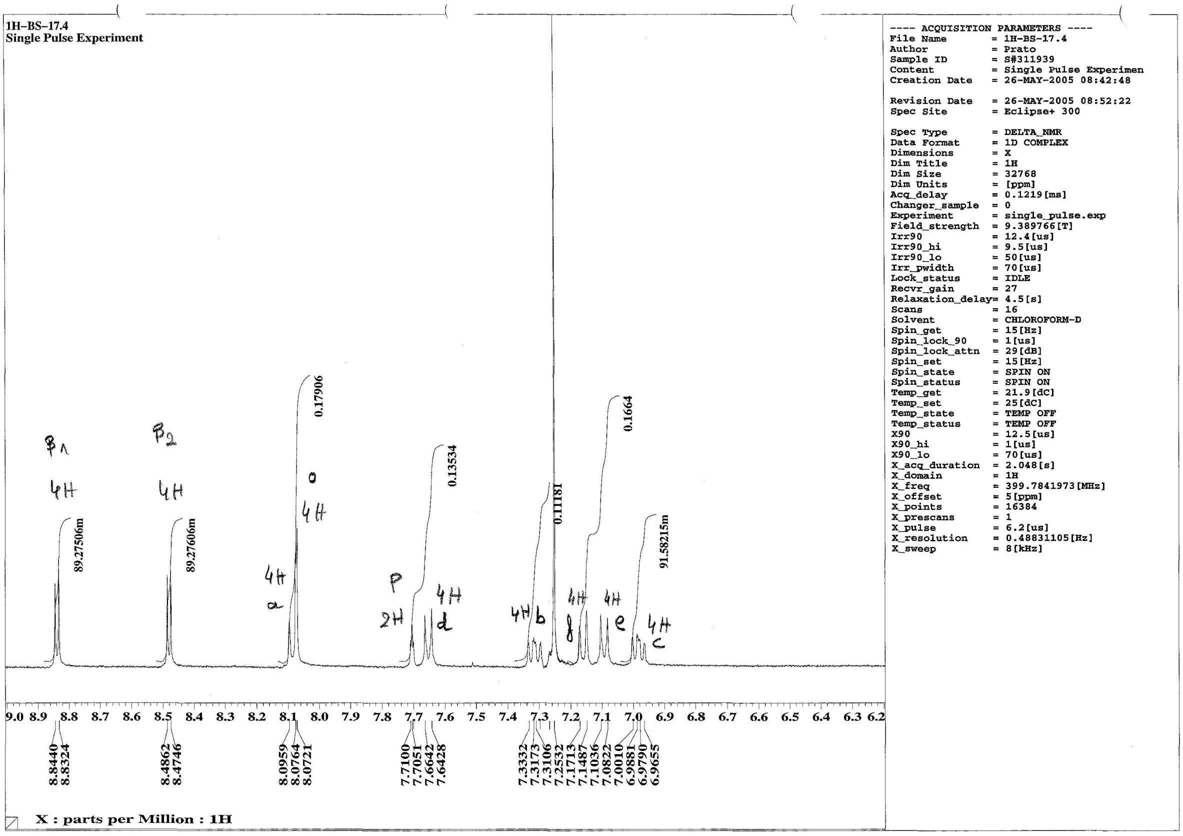
Compound **1b** (whole range and zoomed region)



```
---- ACQUISITION PARAMETERS ----
File Name         = 1H-BS-17.4
Author            = Prato
Sample ID         = S#311939
Content           = Single Pulse Experimen
Creation Date     = 26-MAY-2005 08:42:48

Revision Date     = 26-MAY-2005 08:58:57
Spec Site         = Eclipse+ 300

Spec Type         = DELTA_NMR
Data Format       = 1D COMPLEX
Dimensions        = X
Dim Title         = 1H
Dim Size          = 32768
Dim Units         = [ppm]
Acq_delay         = 0.1219[ms]
Changer_sample    = 0
Experiment        = single_pulse.exp
Field_strength    = 9.389766[T]
Irr90             = 12.4[us]
Irr90_hi          = 9.5[us]
Irr90_lo          = 50[us]
Irr_pwidth        = 70[us]
Lock_status       = IDLE
Recvr_gain        = 27
Relaxation_delay= 4.5[s]
Scans             = 16
Solvent           = CHLOROFORM-D
Spin_get          = 15[Hz]
Spin_lock_90      = 1[us]
Spin_lock_attn    = 29[dB]
Spin_set          = 15[Hz]
Spin_state        = SPIN ON
Spin_status       = SPIN ON
Temp_get          = 21.9[dC]
Temp_set          = 25[dC]
Temp_state        = TEMP OFF
Temp_status       = TEMP OFF
X90               = 12.5[us]
X90_hi            = 1[us]
X90_lo            = 70[us]
X_acq_duration    = 2.048[s]
X_domain          = 1H
X_freq            = 399.7841973[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_pulse           = 6.2[us]
X_resolution      = 0.48831105[Hz]
X_sweep           = 8[kHz]
```

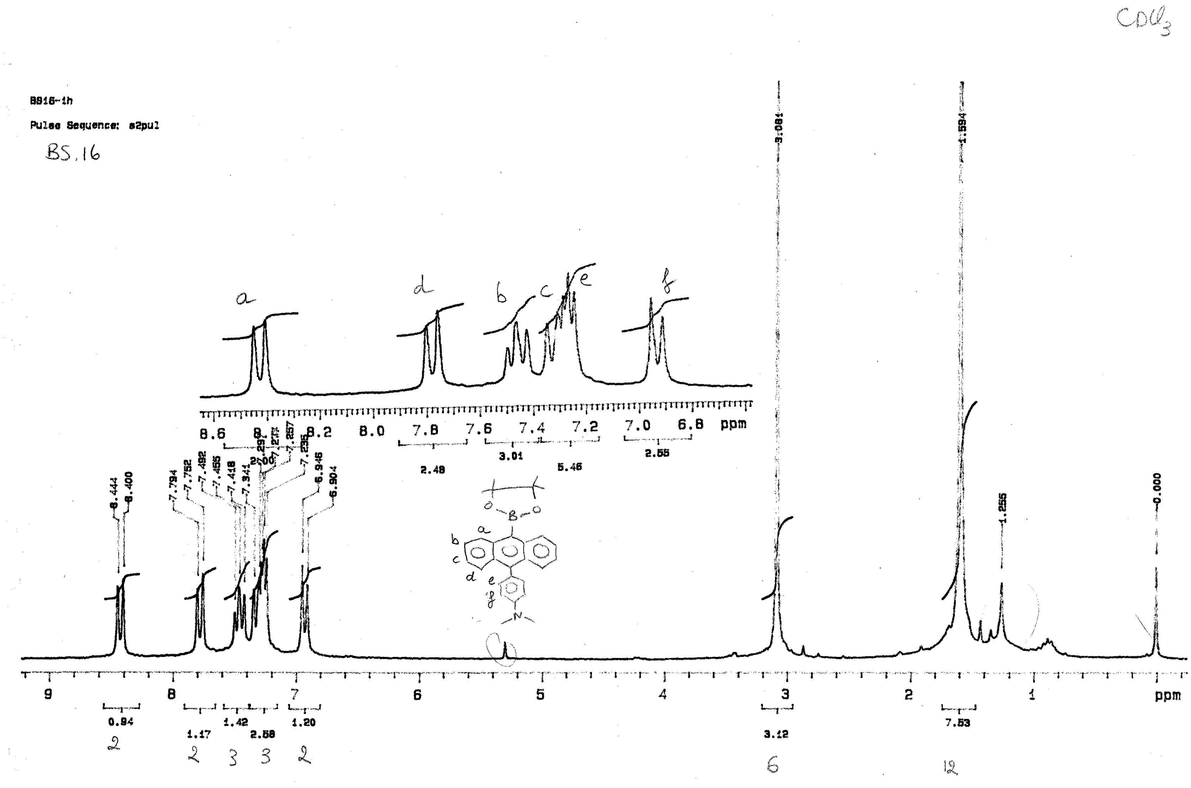


```
---- ACQUISITION PARAMETERS ----
File Name         = 1H-BS-17.4
Author            = Prato
Sample ID         = S#311939
Content           = Single Pulse Experimen
Creation Date     = 26-MAY-2005 08:42:48

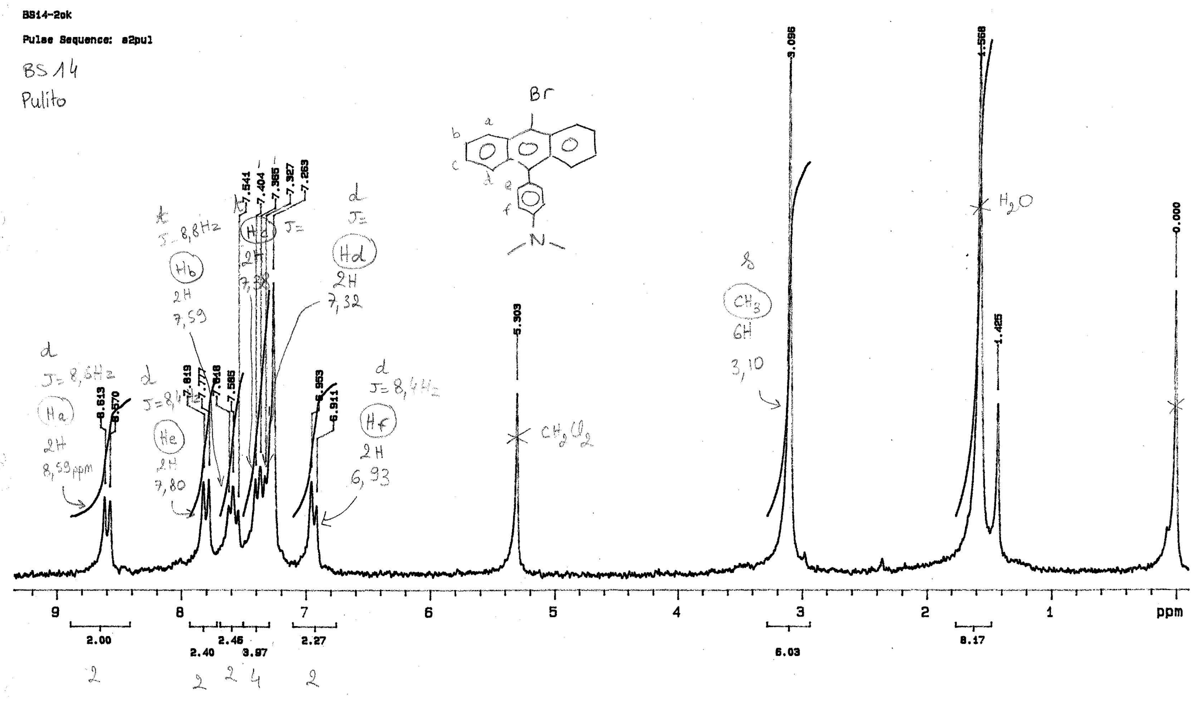
Revision Date     = 26-MAY-2005 08:52:22
Spec Site         = Eclipse+ 300

Spec Type         = DELTA_NMR
Data Format       = 1D COMPLEX
Dimensions        = X
Dim Title         = 1H
Dim Size          = 32768
Dim Units         = [ppm]
Acq_delay         = 0.1219[ms]
Changer_sample    = 0
Experiment        = single_pulse.exp
Field_strength    = 9.389766[T]
Irr90             = 12.4[us]
Irr90_hi          = 9.5[us]
Irr90_lo          = 50[us]
Irr_pwidth        = 70[us]
Lock_status       = IDLE
Recvr_gain        = 27
Relaxation_delay= 4.5[s]
Scans             = 16
Solvent           = CHLOROFORM-D
Spin_get          = 15[Hz]
Spin_lock_90      = 1[us]
Spin_lock_attn    = 29[dB]
Spin_set          = 15[Hz]
Spin_state        = SPIN ON
Spin_status       = SPIN ON
Temp_get          = 21.9[dC]
Temp_set          = 25[dC]
Temp_state        = TEMP OFF
Temp_status       = TEMP OFF
X90               = 12.5[us]
X90_hi            = 1[us]
X90_lo            = 70[us]
X_acq_duration    = 2.048[s]
X_domain          = 1H
X_freq            = 399.7841973[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_pulse           = 6.2[us]
X_resolution      = 0.48831105[Hz]
X_sweep           = 8[kHz]
```

Compound **4b** (whole range and zoomed region)



Compound **5b** (whole range and zoomed region)



Compound **7** (whole range and zoomed region)