Electronic Supplementary Information

Columnar bent-core liquid crystals with two oxadiazole units and two or four alkyl chains and their phase-dependent fluorescence

M. Ferreira,[a] E. Westphal,[b] M. V. Ballottin,[c] I. H. Bechtold,[c] A. J. Bortoluzzi,[a] D. Z. Mezalira [a] and H. Gallardo*[a]

[a.] Departamento de Química, Universidade Federal de Santa Catarina, 88040-900 Florianópolis, SC, Brazil. E-mail: hugo.gallardo@ufsc.br; Fax: +5548 3721 6850; Tel: +55 48 3721 6849.
[b.] Departamento Acadêmico de Química e Biologia, Universidade Tecnológica Federal do Paraná, Curitiba, PR, Brazil. E-mail: eduardw@utfpr.edu.br.
[c.] Departamento de Física, Universidade Federal de Santa Catarina, Florianópolis, Brazil. E-mail: bechtold@fsc.ufsc.br

Synthesis

General procedure for the synthesis of nitriles 1 and 2. 4-hydroxybenzonitrile (7.00 g, 119.1 g mol^{-1}, 58.8 mmol), the appropriate *n*-bromoalkane (70.6 mmol), and K_2CO_3(20.32 g, 138.2 g mol^{-1}, 147.0 mmol) were refluxed and stirred in butanone (250 mL) for 24 h. The suspension was filtered and washed with hot butanone, the solvent was evaporated and the product was purified by recrystallization from cold methanol (**1**) or ethanol (**2**).

4-(hexyloxy)benzonitrile 1. Yield: 8.70 g (203.3 g mol^{-1}, 42.8 mmol, 73%) of a white solid; IR (KBr): ν_{max} = 2933, 2827, 2225, 1608, 1510, 1469, 1303, 1259, 1172, 835 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.55 (d, *J* = 9 Hz, 2H), 6.92 (d, *J* = 9 Hz, 2H), 3.98 (t, *J* = 7 Hz, 2H), 1.78 (quint, *J* = 7 Hz, 2H), 1.45 (quint, *J* = 7 Hz, 2H), 1.38-1.27 (m, 4H), 0.90 ppm (t, *J* = 7 Hz, 3H);^{13}C NMR ($CDCl_3$, 100 MHz): δ= 162.5, 133.9, 119.3, 115.2, 103.6, 68.4, 31.5, 29.0, 25.6, 22.6, 14.0 ppm; m.p.:26-28 °C (lit., 32 °C).[1]

4-(dodecyloxy)benzonitrile 2 was synthesized and described previously.[2]

General procedure for the synthesis of nitriles3 and 4. 3,4-dihydroxybenzonitrile (6.00 g, 135.1 g mol^{-1}, 44.4 mmol), the appropriate *n*-bromoalkane (106.6 mmol), K_2CO_3(30.68 g, 138.2 g mol^{-1}, 222.0 mmol), and TBAB (0.71 g 322.4 g mol^{-1}, 2.2 mmol) was stirred and refluxed in butanone (200 mL) for 24 h. The suspension was filtered and washed with hot butanone, the solvent was evaporated and the product was purified by recrystallization from acetone (**3**) or acetonitrile (**4**).

3,4-(dihexyloxy)benzonitrile 3. Yield: 12.40 g (303.4 g mol^{-1}, 40.8 mmol, 92%) of white solid; IR (KBr): ν_{max}: 2934, 2859, 2221, 1597, 1518, 1469, 1421, 1279, 1244, 1138, 811 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ= 7.22 (dd, *J* = 8 Hz, 2 Hz, 1H), 7.07 (d, *J* = 2 Hz, 1H), 6.87 (d, *J* = 8 Hz, 1H), 4.02 (t, *J* = 7 Hz, 2H), 3.98 (t, *J* = 7 Hz, 2H), 1.83 (m, 2H), 1.81 (m, 2H), 1.48 (m, 2H), 1.46 (m, 2H), 1.38-1.30 (m, 8H), 0.91 (t, *J* = 7 Hz, 3H), 0.90 ppm (t, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ= 153.1, 149.1, 126.4, 119.5, 116.0, 112.8, 103.5, 69.5, 69.1, 31.6, 31.57, 29.1, 29.0, 25.7, 25.67, 22.66, 22.6, 14.1, 14.06 ppm; m.p.: 64 °C.

3,4-(didodecyloxy)benzonitrile 4.was synthesized and described previously.[2]

General procedure for the synthesis of tetrazoles. The appropriate nitrile **1-4** (34.5 mmol), NaN_3(6.73 g, 65.0 g mol^{-1}, 103.5 mmol), and NH_4Cl (5.54 g, 53.5 g mol^{-1}, 103.5 mmol) in DMF (100mL) was refluxed under strong stirring for 24 h. After cooling to room temperature, the suspension was poured into water/ice(400 mL) and acidified with 10 % aqueous hydrochloric acid to pH = 2. The solid was collected by filtration and washed with plenty of water and purified by recrystallization from acetonitrile (**5**) or acetone (**6**, **7**) or butanone (**8**).

5-(4-hexyloxyphenyl)tetrazole 5. Yield: 7.70 g (246.3 g mol^{-1}, 31.3 mmol, 91%) of white crystals; IR (KBr): ν_{max} = 2924, 2855, 2749, 2648, 1612, 1506, 1468, 1258, 1185, 1057, 1032, 842 cm^{-1}; ^{1}H NMR (Acetone [D6], 400 MHz): δ= 8.04 (d, *J* = 9 Hz, 2H), 7.13 (d, *J* = 9 Hz, 2H), 4.10 (t, *J* = 7 Hz, 2H), 1.80 (quint, *J* = 7 Hz, 2H), 1.49 (quint, *J* = 7 Hz, 2H), 1.41-1.30 (m, 4H), 0.90 ppm (t, *J* = 7 Hz, 3H); ^{13}C NMR (Pyridine [D5], 100 MHz): δ = 162.2, 157.7, 129.8, 118.6, 116.2, 68.9, 32.2, 29.8, 26.4, 23.3, 14.7 ppm. m.p.: 173-175 °C.

5-(4-dodecyloxyphenyl)tetrazole 6. Yield:9.7 g (330.5 g mol^{-1}, 29.3 mmol. 85%) of white solid; IR (KBr): ν_{max} = 2920, 2857, 2549, 2467, 1903, 1615, 1493, 1461, 1402, 1254, 1168, 1045, 988, 829 cm^{-1}; ^{1}H NMR(Acetone [D6], 400 MHz): δ= 8.04 (d, *J* = 9 Hz, 2H), 8.02 (s, 1H), 7.12 (d, *J* = 9 Hz, 2H), 4.09 (t, *J* = 6 Hz, 2H), 1.80 (quint, *J* = 6 Hz, 2H), 1.57-1.43

(m, 2H), 1.43-1.16 (br, 16H), 0.86 ppm (t, *J* = 7 Hz, 3H);[13]C NMR(Acetone [D6], 100 MHz): 162.2, 157.7, 129.8, 118.7, 116.2, 69.0, 32.6, 30.43, 30.4, 30.36, 30.35, 30.13, 30.1, 30.0, 26.8, 23.4, 14.8 ppm; m.p.: 149-151 °C (lit. 154 °C).[3]

5-(3,4-dihexyloxyphenyl)tetrazole 7. Yield:10.80 g (346.5 g mol^{-1},31.2 mmol, 90%) of white solid; IR (KBr): ν_{max} =2956, 2924, 2858, 2750, 2488, 1608, 1511, 1465, 1272, 1237 cm^{-1}; ^{1}H NMR(Pyridine [D5], 400 MHz): δ= 8.58 (s, 1H), 8.00 (dd, *J* = 8 Hz, 2 Hz, 1H), 7.97 (d, *J* = 2 Hz, 1H) 7.20 (d, *J* = 8 Hz, 1H), 4.04 (t, *J* = 7 Hz, 2H), 3.95 (t, *J* = 7 Hz, 2H), 1.77 (m, 2H), 1.76 (m, 2H), 1.43 (m, 2H), 1.40 (m, 2H), 1.31-1.15 (m, 8H), 0.85 (t, *J* = 7 Hz, 3H), 0.85 ppm (t, *J* = 7 Hz, 3H); ^{13}C NMR(Pyridine [D5], 100 MHz): δ = 157.9, 152.4, 121.3, 118.8, 114.4, 113.0, 69.6, 32.2, 32.16, 30.0, 29.97, 26.5, 26.4, 23.3, 14.6 ppm; m.p.:171-172 °C.

5-(3,4-didodecyloxyphenyl)tetrazole 8. Yield:16.00 g (514.8 g mol^{-1}, 31.0 mmol, 90%) of white solid; IR (KBr): ν_{max} = 2921, 2848, 2744, 2613, 1607, 1512, 1465, 1272, 1239, 1133, 1039, 812, 746 cm^{-1}; ^{1}H NMR(Pyridine [D5], 400 MHz): δ= 8.58 (s, 1H), 8.01 (d, *J* = 9 Hz, 1H), 7.99 (s, 1H), 7.21 (d, *J* = 9 Hz, 1H), 4.09 (t, *J* = 6 Hz, 2H), 4.00 (t, *J* = 6 Hz, 2H), 1.92-1.74 (m, 4H), 1.60-1.40 (m, 4H), 1.38-1.18 (m, 32H), 0.88 ppm (t, *J* = 7 Hz, 6H);[13]C NMR(Pyridine [D5], 100 MHz): δ = 157.9, 152.1, 121.3, 118.8, 114.4, 113.1, 69.7, 32.6, 30.5, 30.47, 30.42, 30.4, 30.2, 30.15, 30.13, 30.11, 30.1,26.9, 26.88, 23.4, 14.8 ppm; m.p.:158-159 °C (lit. 157.8-159.0°C)[4]

5-iodoisophthalic acid 9b. 5-aminoisophthalic acid (10.00 g, 181.1 g mol^{-1}, 55.2 mmol) was refluxed in metanol (300 mL) and sulfuric acid. (4.00 mL) for 24 h. Then the solvent was evaporated and the crude product was dissolved in chloroform and washed with 10% aqueous NaOH. The organic phase was washed with water and dried over sodium sulfate, the solvent was evaporated and the product was purified by recrystallization from MeOH:H_2O. Yield: 10.28 g (209.2 g mol^{-1}, 49.1 mmol, 80 %) of beige crystals; m.p. 178-180 °C.
A solution of $NaNO_2$ (3.46g, 69.0 g mol^{-1}, 50.2 mmol) in water (10 mL) was added to a stirred cooling suspension (-5 °C) of dimethyl 5-aminoisophthalate (10.0 g, 209.2 g mol^{-1}, 47.8 mmol) in 10% aqueous hydrochloric acid (170 mL). The mixture was stirredat 0 °C for 1 h, then a solution of KI (31.54 g, 166.0 g mol^{-1}, 0.19 mol) in water (80 mL) was added dropwise, then the mixture was strongly stirred at room temperature for further 12 h. The orange precipitate was filtered off and washed with plenty of water and purified by recrystallization from methanol. Yield: 13.60 g (320.1g mol^{-1}, 42.5 mmol, 89 %) of white solid; m.p.: 102-103 °C; (lit., 103-105 °C);[5] ^{1}H NMR (DMSO[D6], 200 MHz): δ= 8.68-8.61 (m, 3H), 3.93 ppm (s, 6H).
Dimethyl 5-iodoisophthalate (13.00 g, 320.1g mol^{-1}, 40.6 mmol) was refluxed in methanol and NaOH for 2 h, then acidified with 10% aqueous hydrochloric acid to pH = 3. The precipitate was filtered off and washed with water and dried. Yield: 11.26 g (292.0 g mol^{-1}, 38.6 mmol, 95 %) of white solid; m.p.: 286-287 °C; (lit., 285-288°C).[6] ^{1}H NMR (DMSO [D6], 200 MHz): δ = 8.4 (m, 3H) ppm.

5-acetoxyisophthalic acid 9d. 5-hydroxyisophthalic acid (7 g, 182.1 g mol^{-1}, 38.4 mmol) was dissolved in 1:1 pyridine/acetic anhydride (26 mL) and stirred at room temperature for 1 h. Then, the solution was poured into10% aqueous hydrochloric acid (300 mL) and the product was extracted with ethyl acetate (3 × 100 mL). The solution was dried with sodium sulfate, the solvent was evaporated and the product was purified by recrystallization from water. Yield: 6.81 g (224.2 g mol^{-1}, 30.4 mmol, 79 %) of white solid; m.p.: 238-240 °C.[7]

Additional data

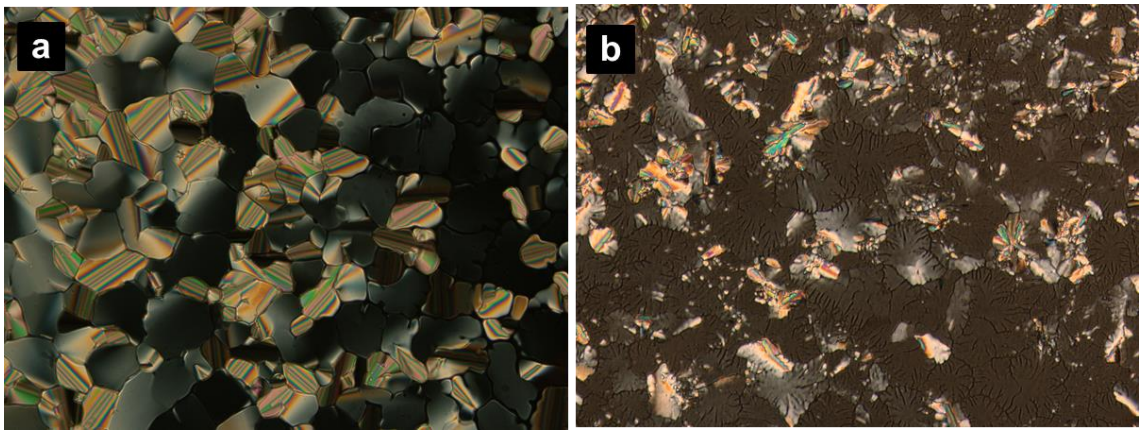

Figure S1. Optical textures of the Col_h mesophases of **NO2-4C12** at 144 °C (a) and **NH2-4C12** (b - slightly uncrossed polarizers) at 150 °C.

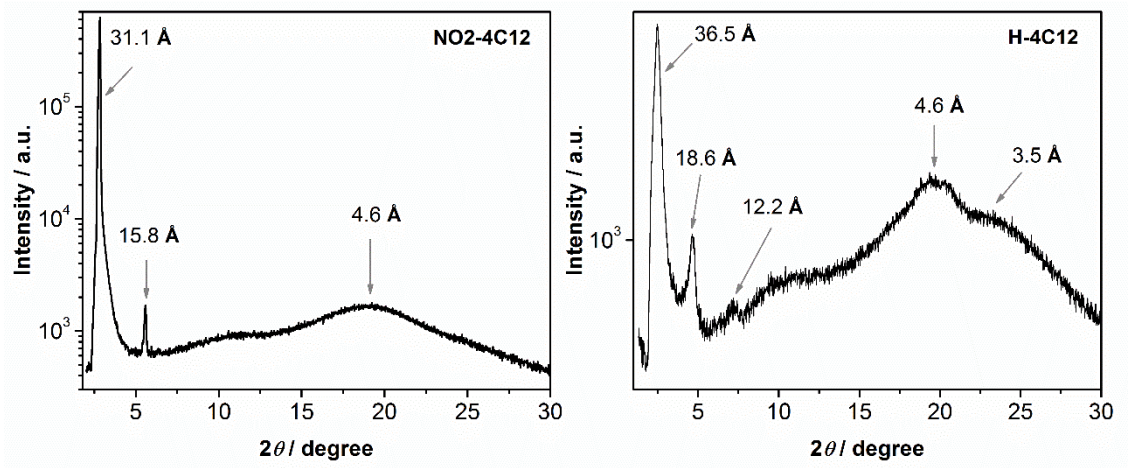

Figure S2. XRD spectra of **NO2-4C12** (left) and**H-4C12** (right) after cooling from the isotropic liquid (logarithmic intensity scaling).

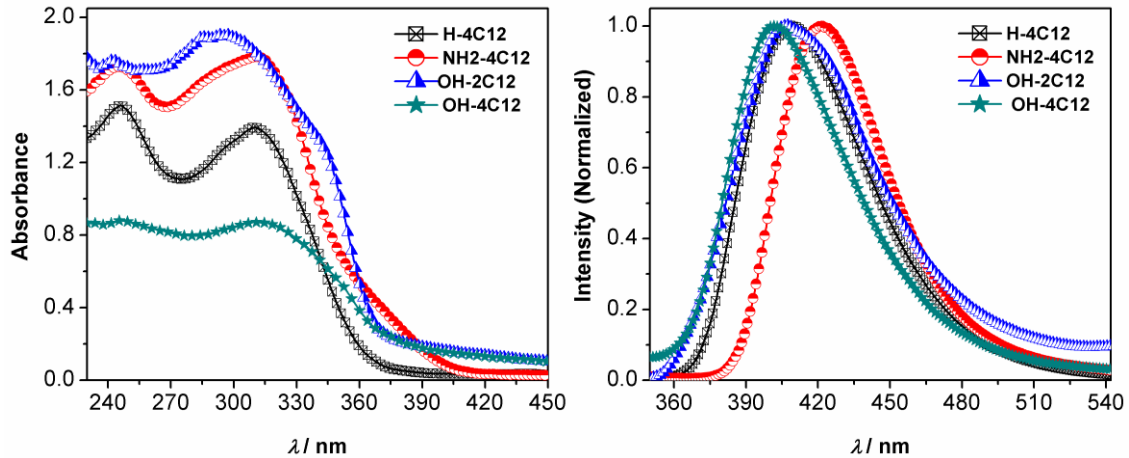

Figure S3. Absorption spectrum (left) and emission spectrum (right) for thin solid film of fluorescent columnar compound with four dodecyloxy chains.

^{1}H and ^{13}C NMR spectra of final compounds

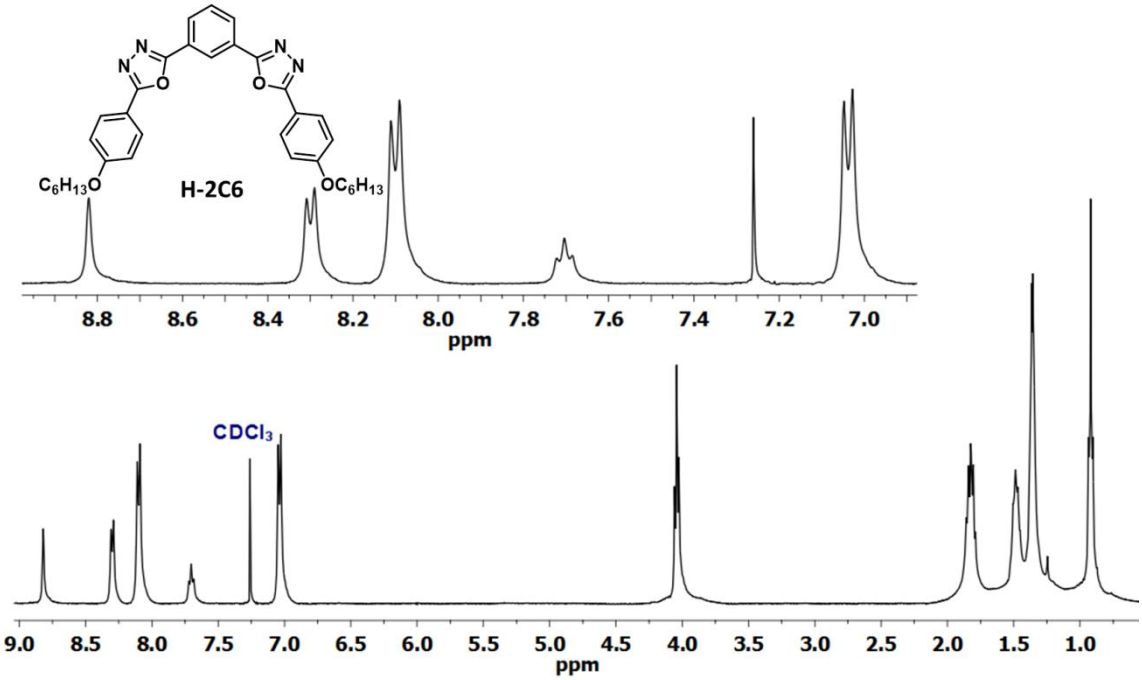

Figure S4. ^{1}H NMR spectrum ($CDCl_3$, 400MHz) of compound **H-2C6**.

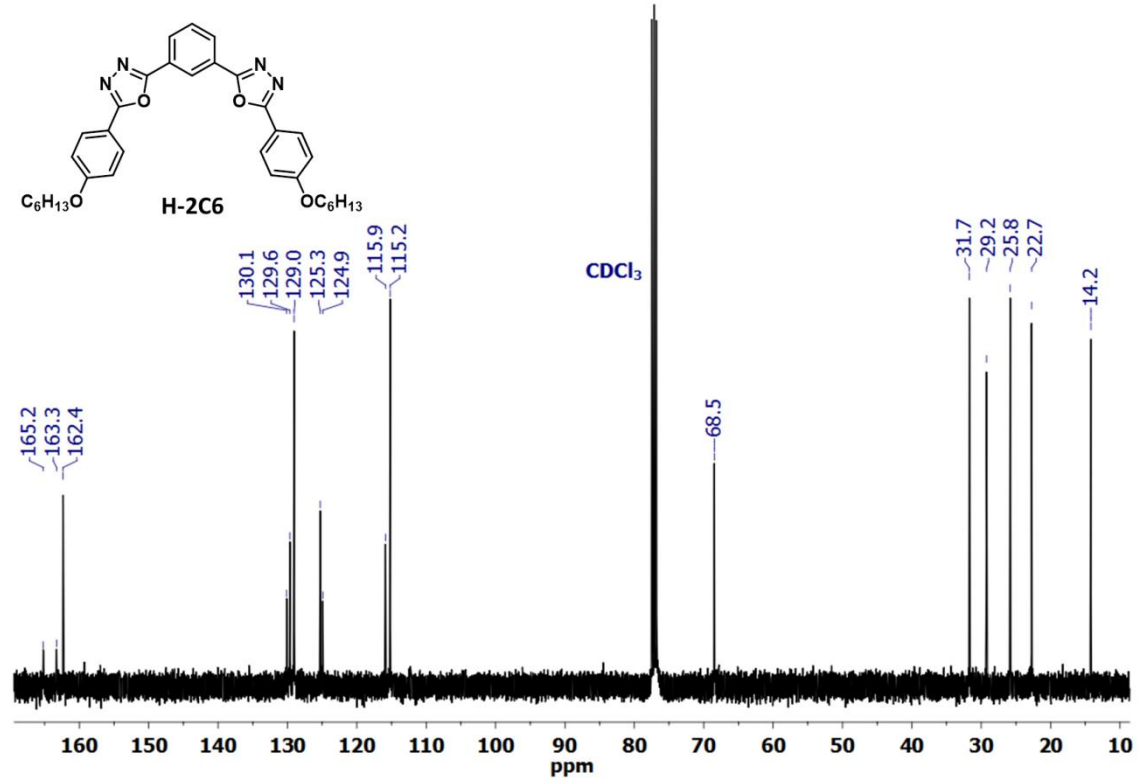

Figure S5. ^{13}C NMR spectrum ($CDCl_3$, 100MHz) of compound **H-2C6**.

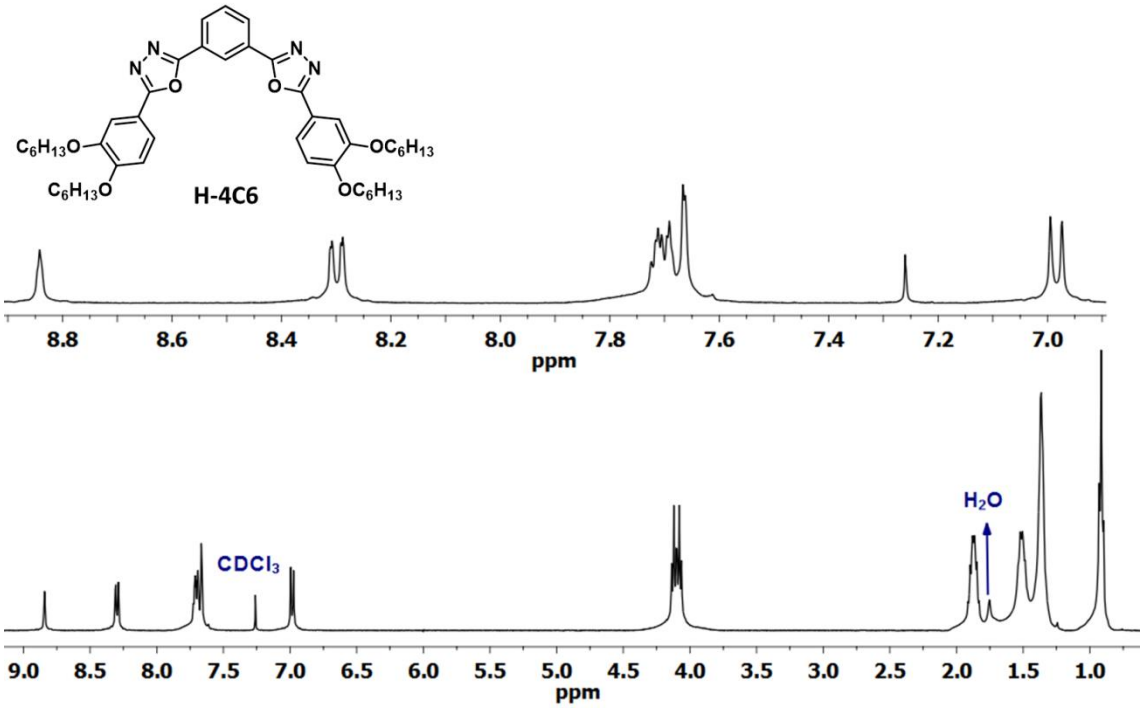

Figure S6.^{1}H NMR spectrum ($CDCl_3$, 400MHz) of compound **H-4C6**.

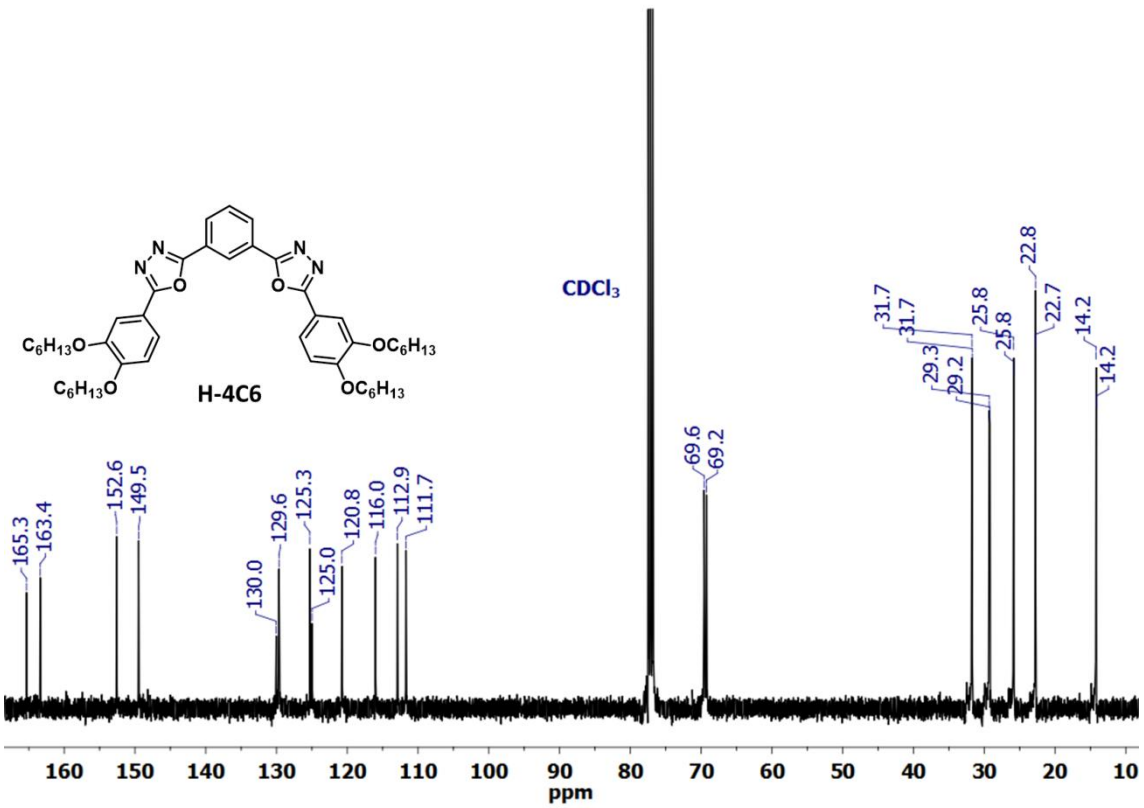

Figure S7.^{13}C NMR spectrum ($CDCl_3$, 100MHz) of compound **H-4C6**.

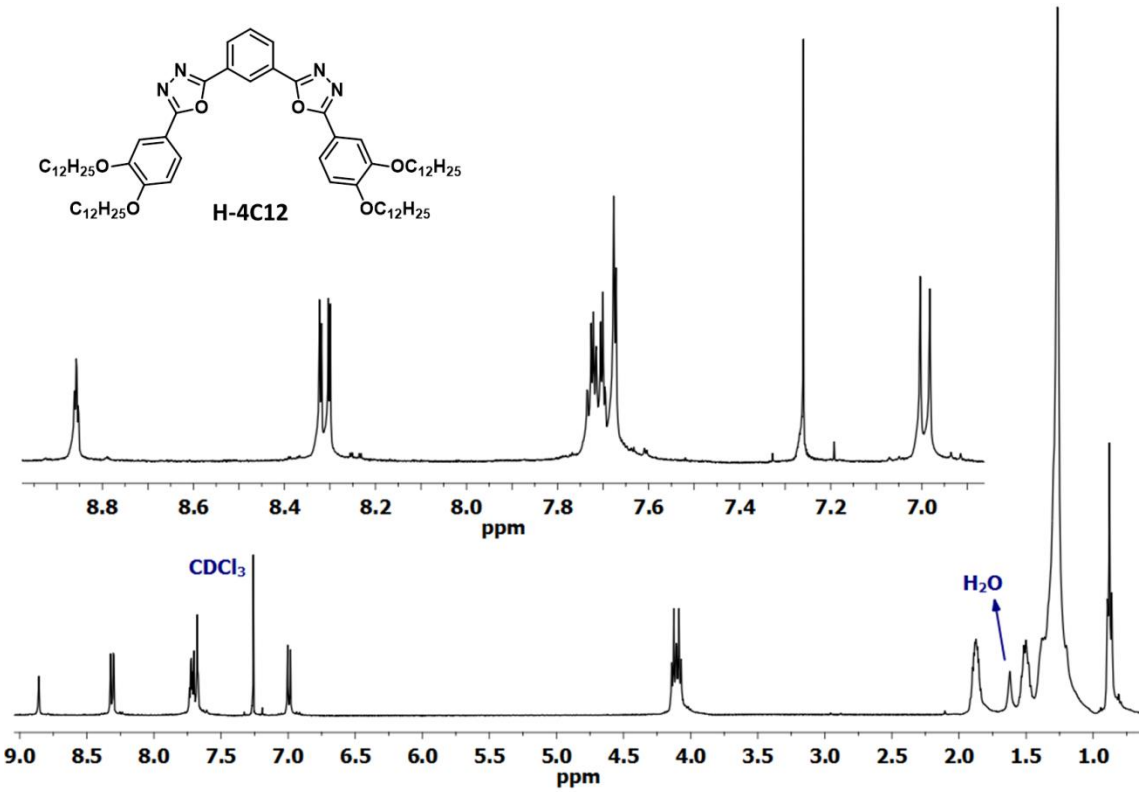

Figure S8.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **H-4C12**.

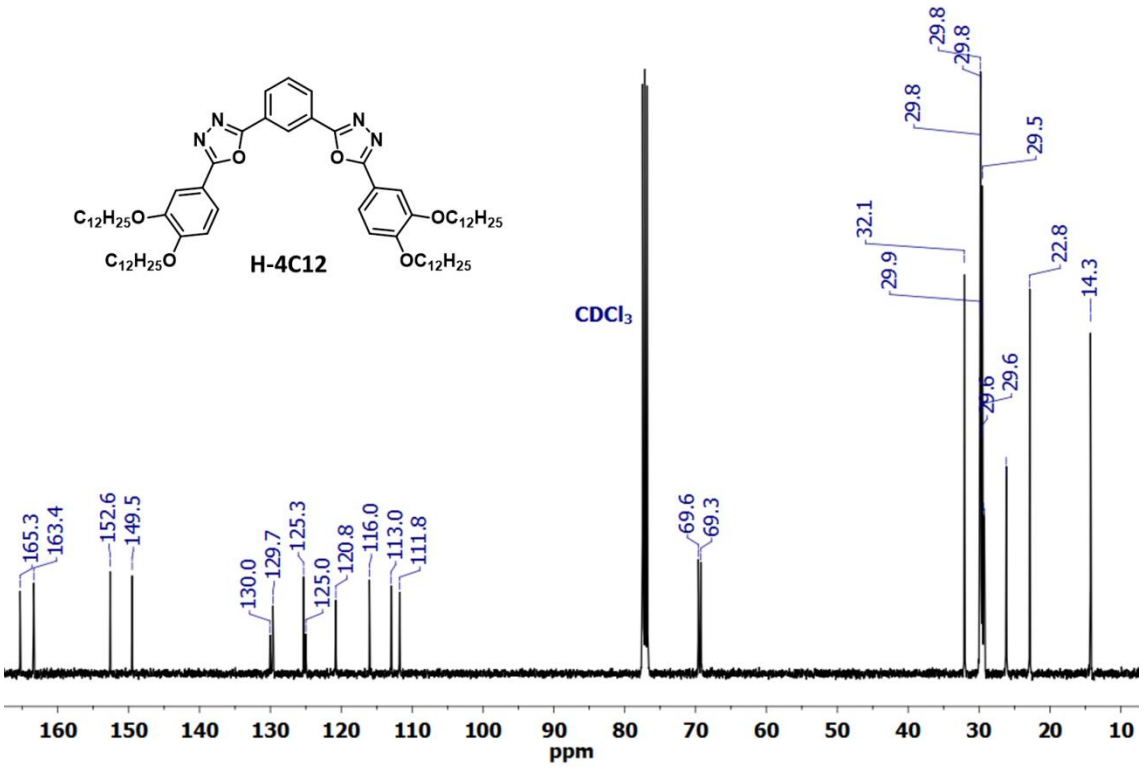

Figure S9.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **H-4C12**.

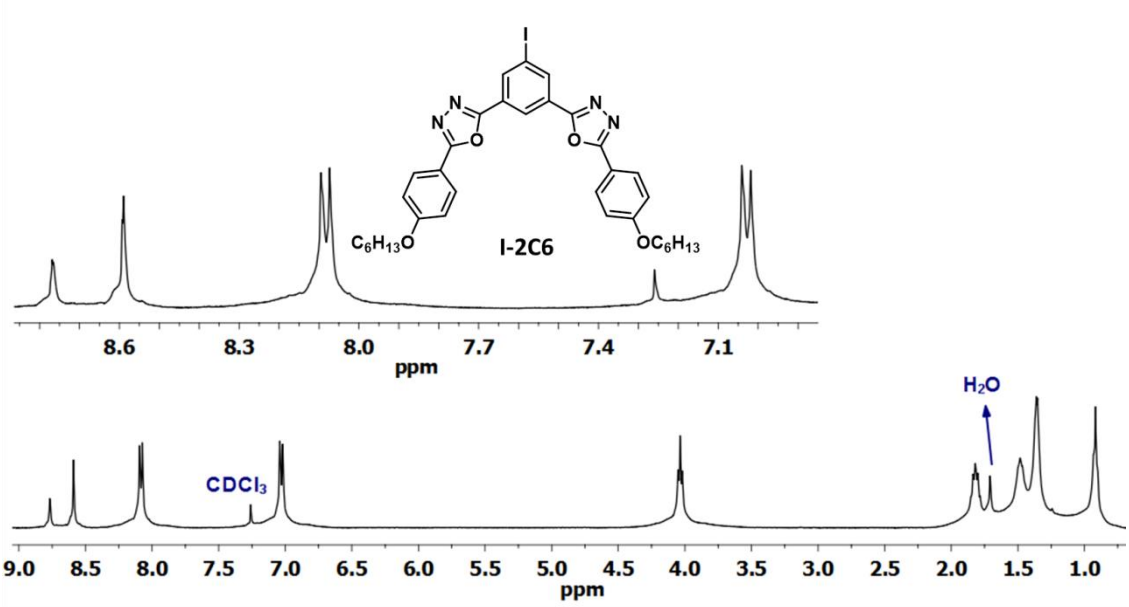

Figure S10.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **I-2C6**.

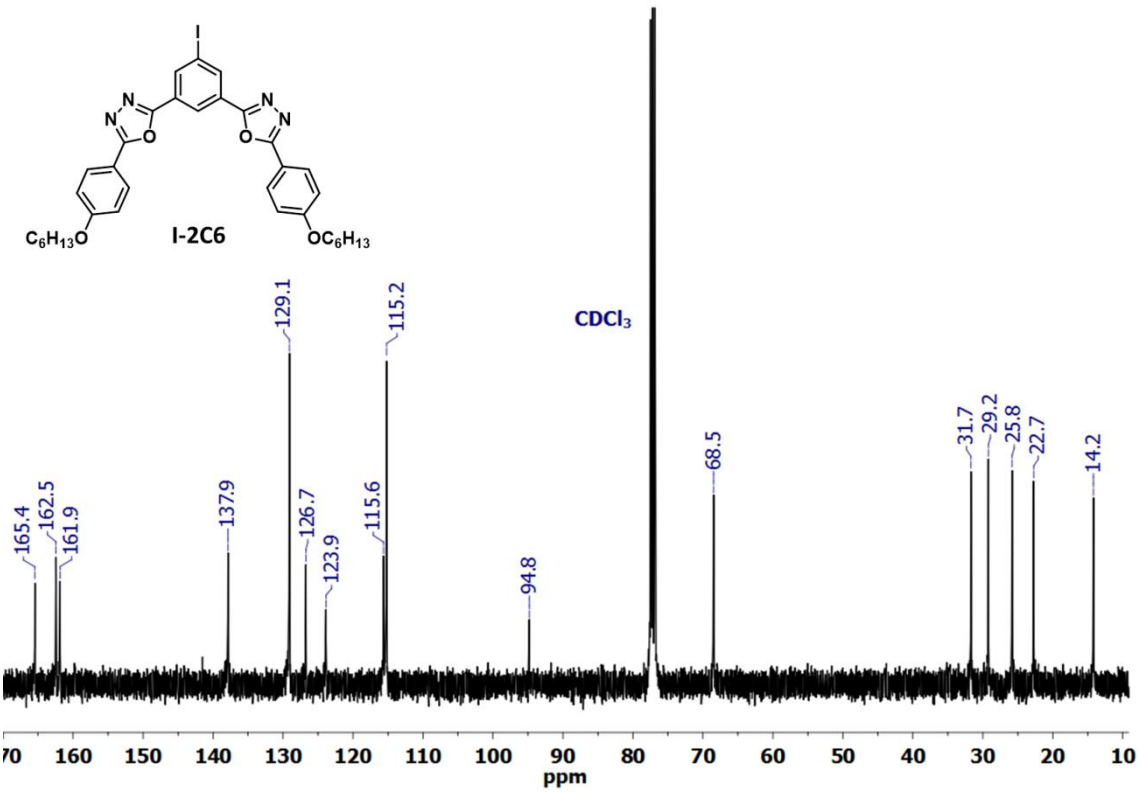

Figure S11.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **I-2C6**.

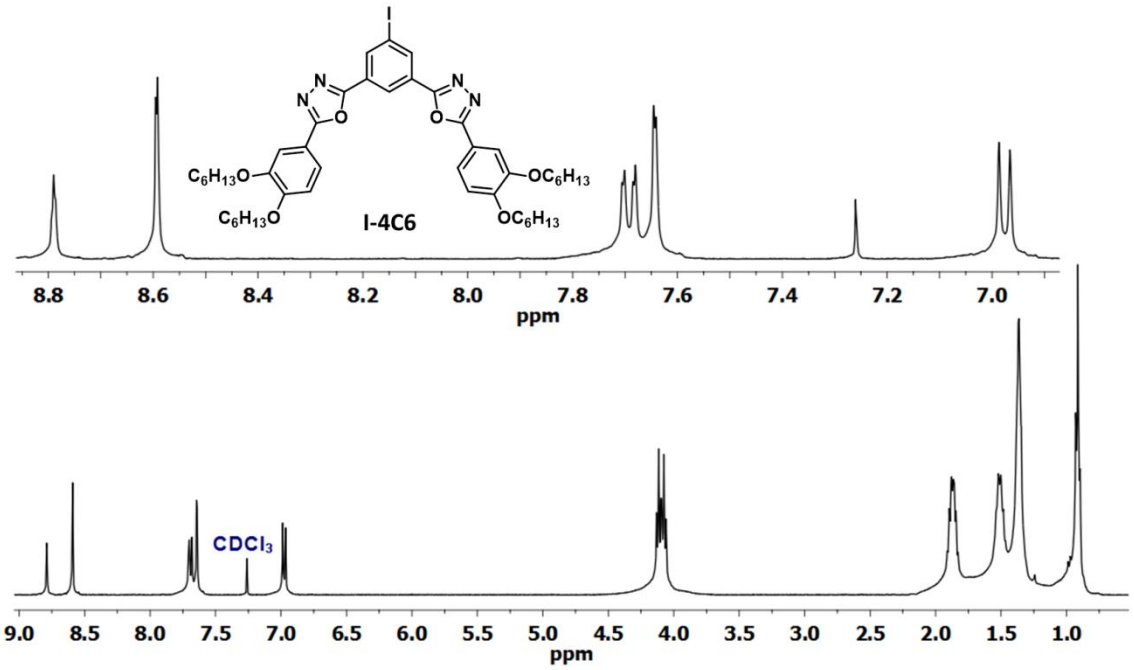

Figure S12.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **I-4C6**.

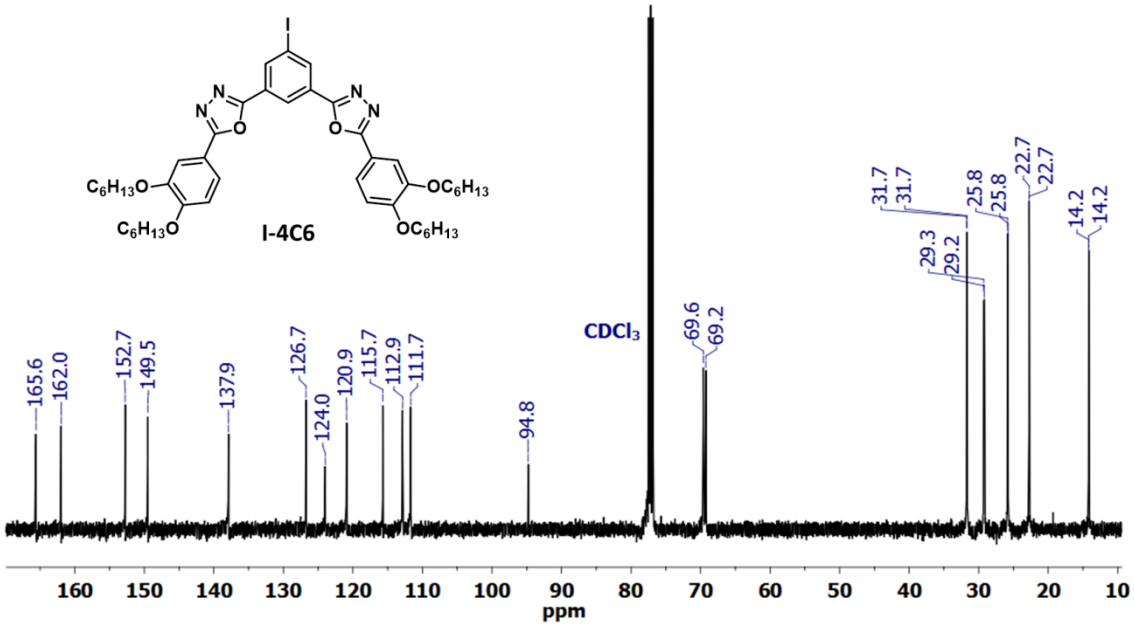

Figure S13.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **I-4C6**.

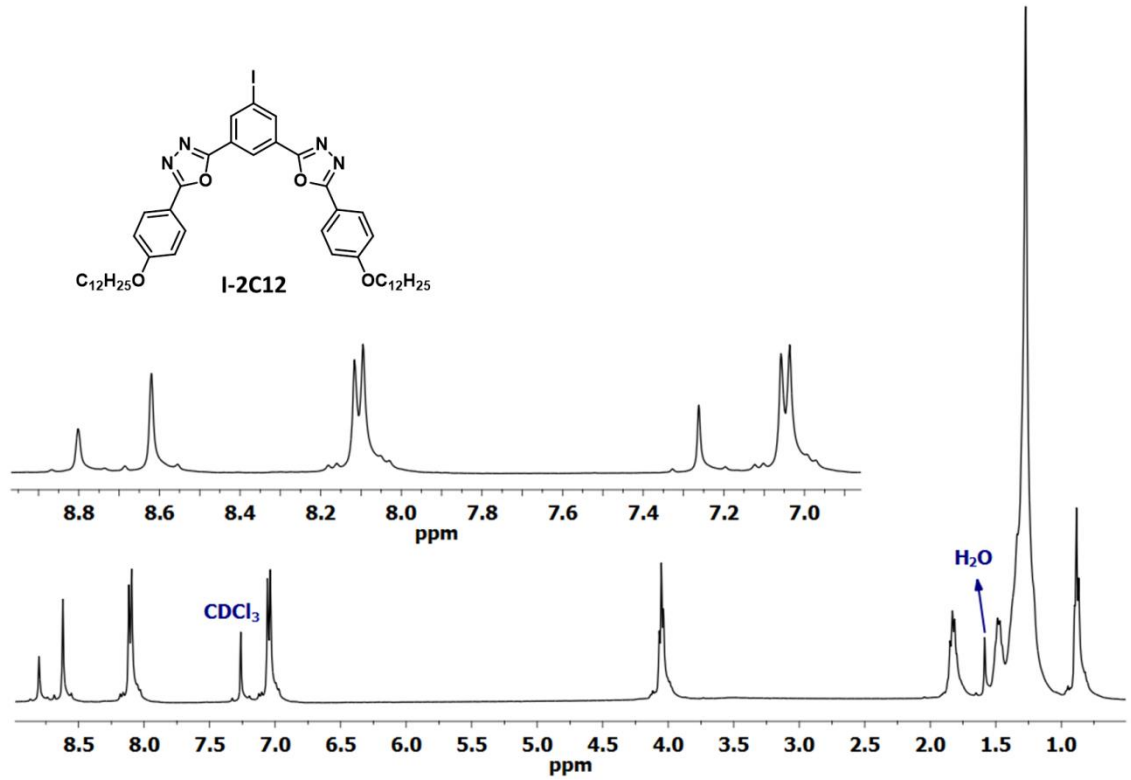

Figure S14.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **I-2C12**.

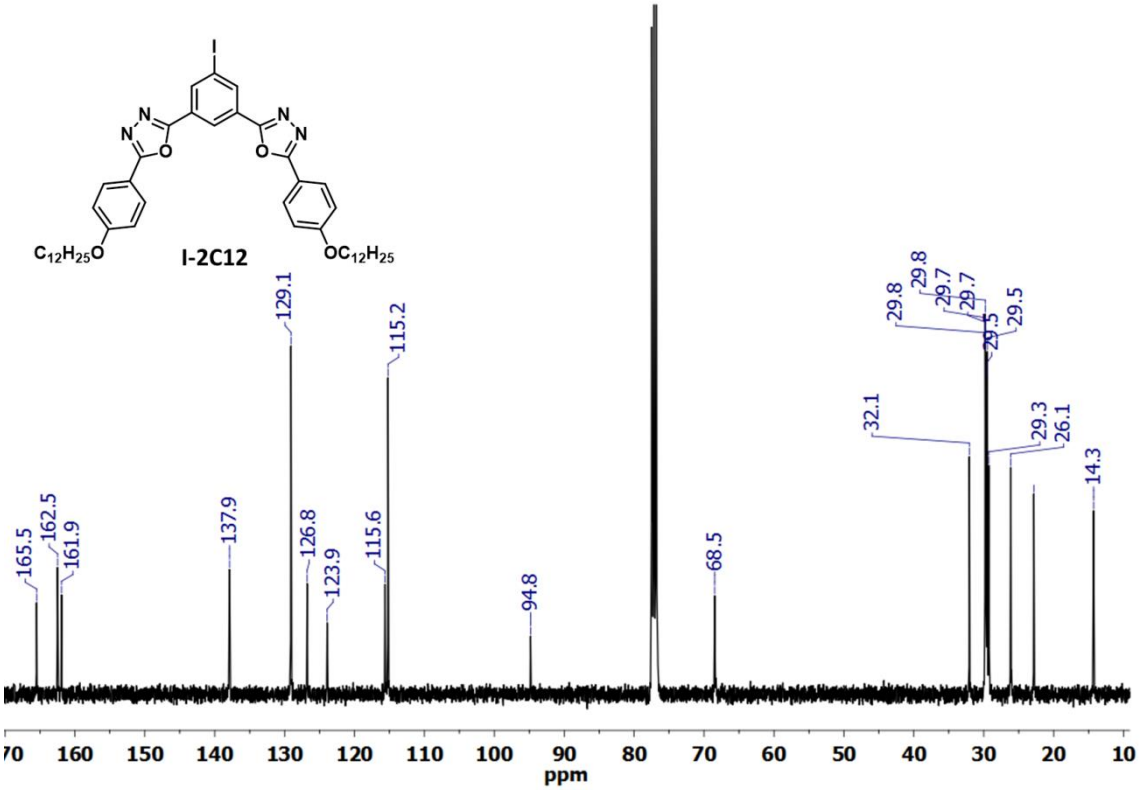

Figure S15.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **I-2C12.**

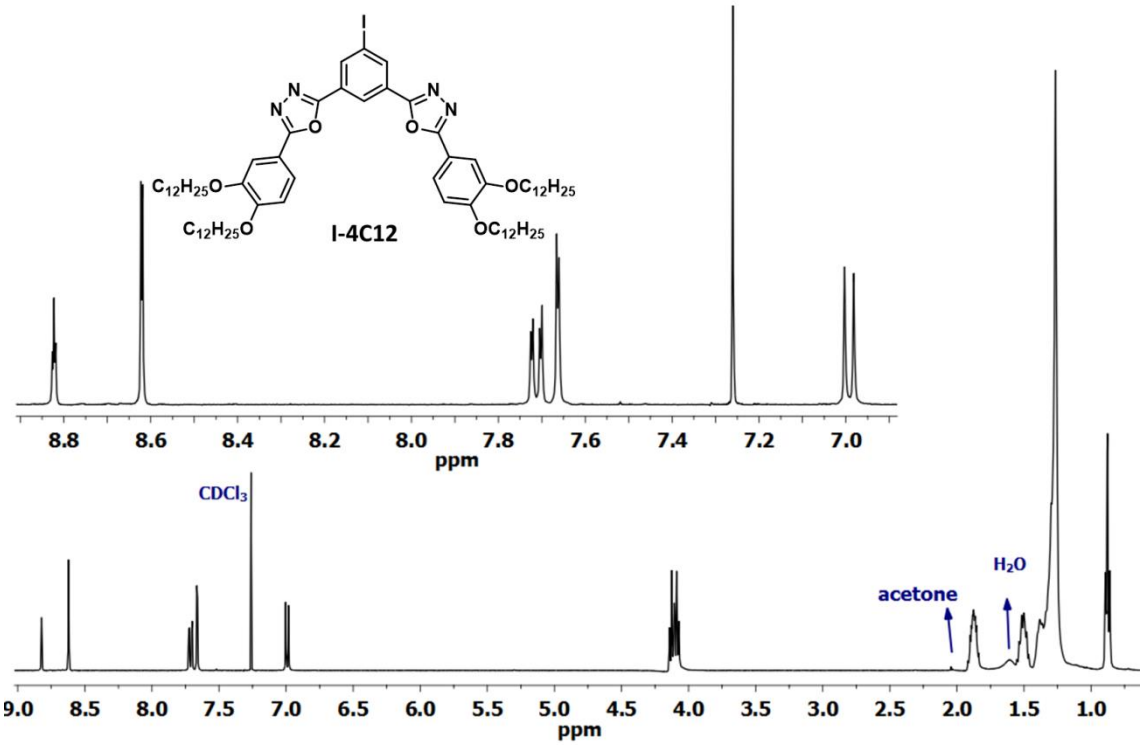

Figure S16.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **I-4C12**.

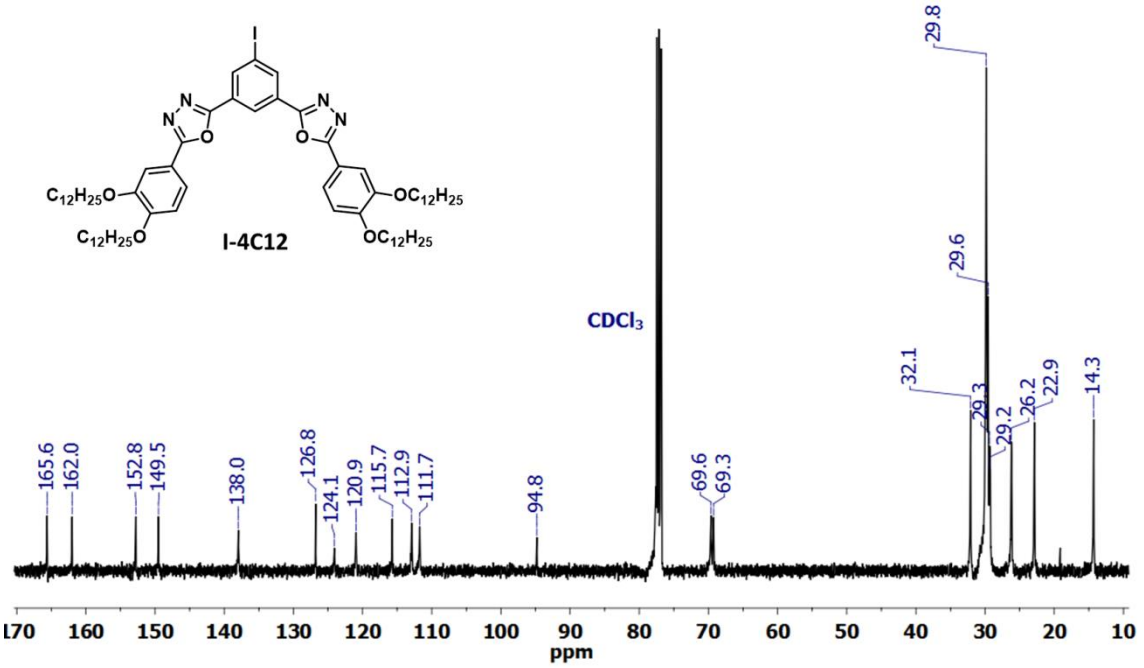

Figure S17.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **I-4C12**.

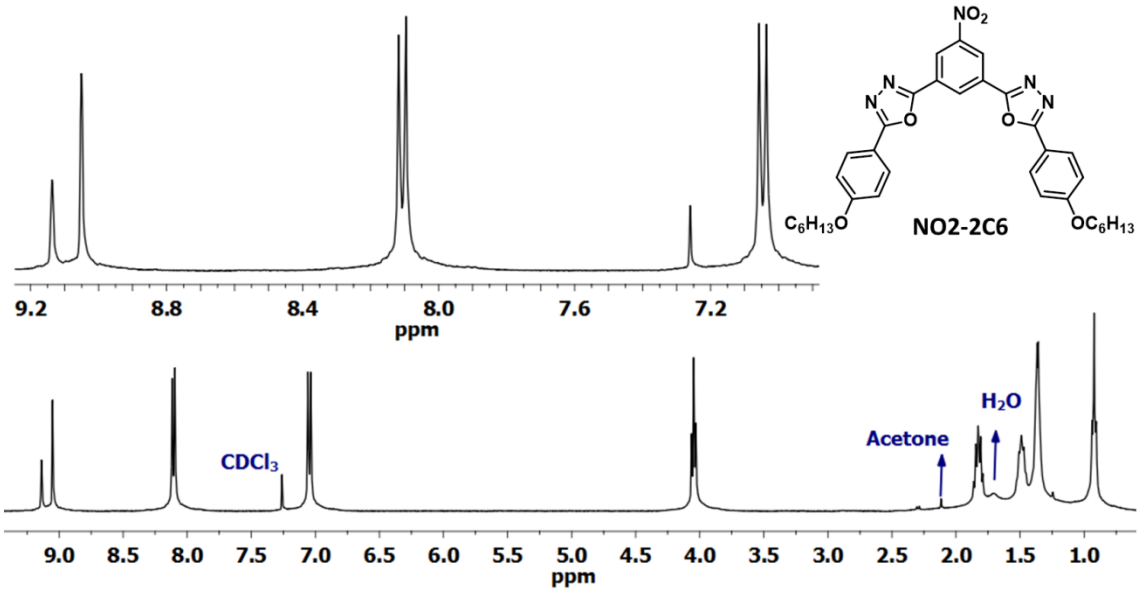

Figure S18.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **NO2-2C6**.

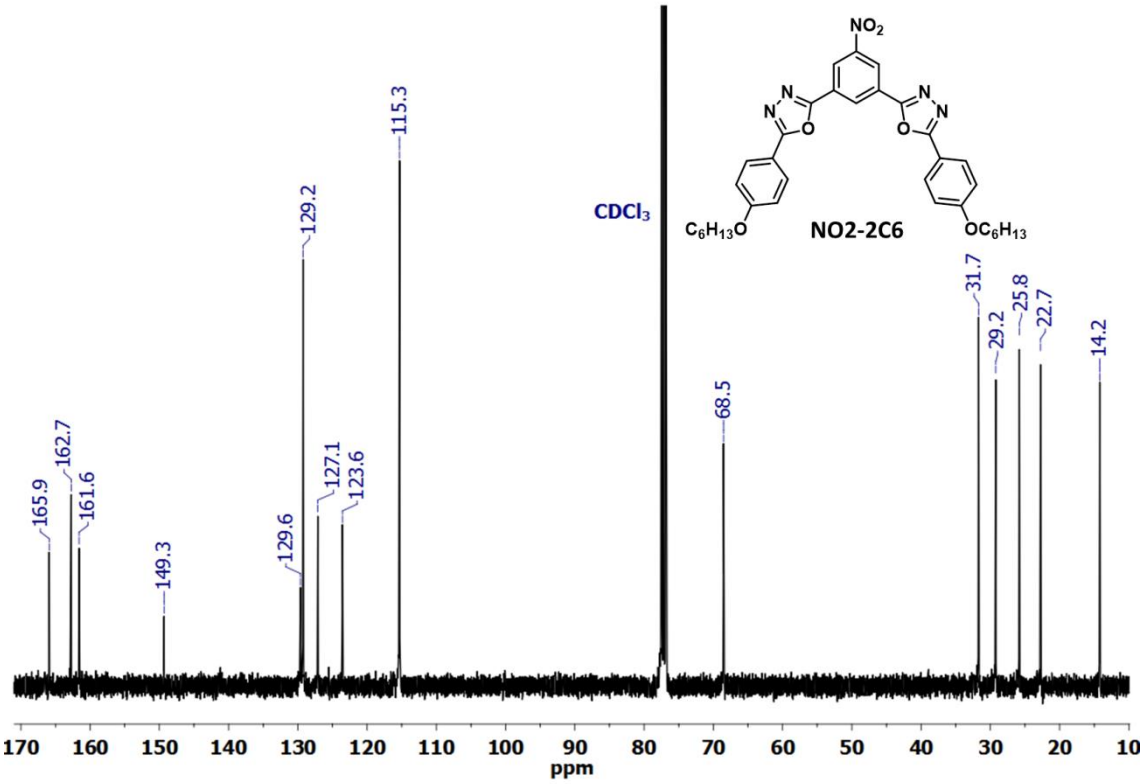

Figure S19.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **NO2-2C6**.

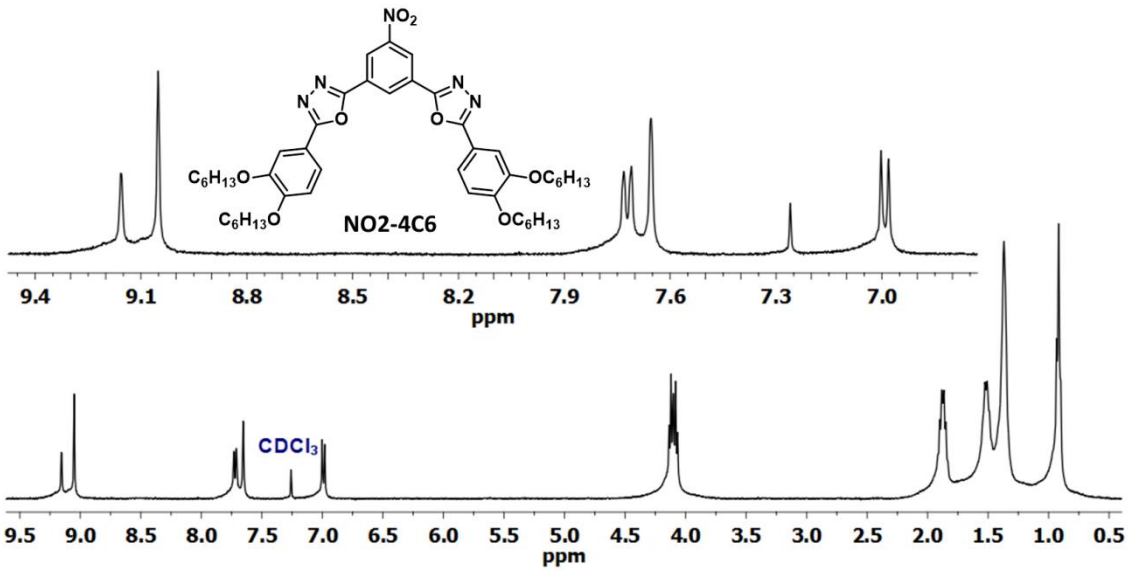

Figure S20.[1]H NMR spectrum ($CDCl_3$, 400MHz) of compound **NO2-4C6**.

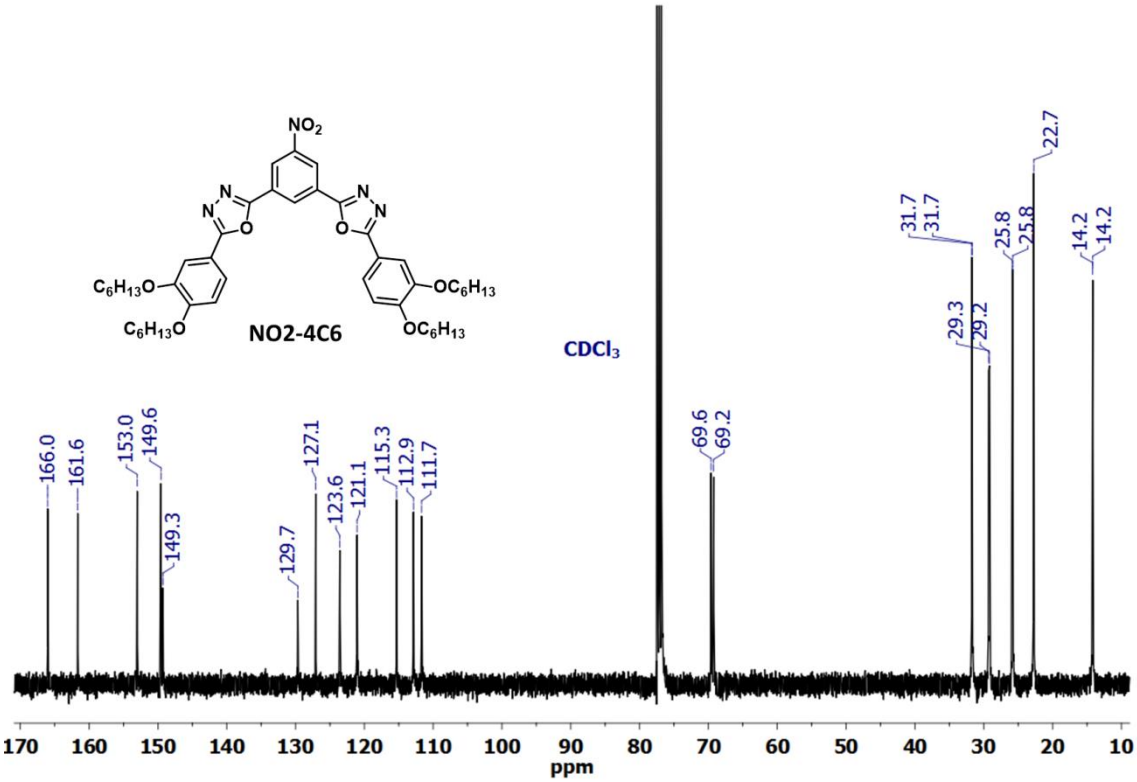

Figure S21.[13]C NMR spectrum ($CDCl_3$, 100MHz) of compound **NO2-4C6**.

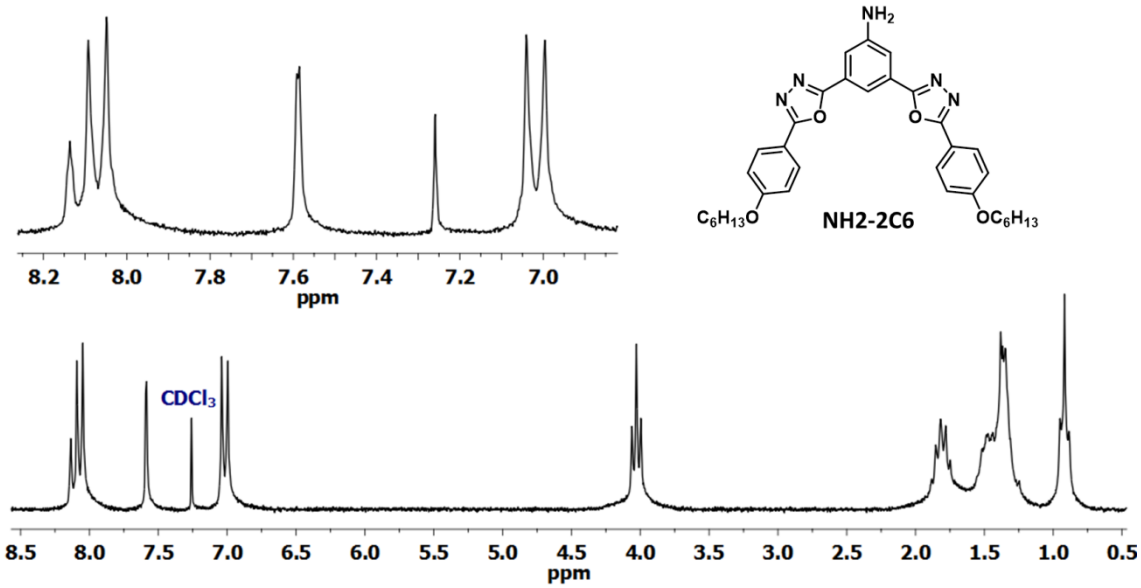

Figure S22. ^{1}H NMR spectrum ($CDCl_3$, 200MHz) of compound **NH2-2C6**.

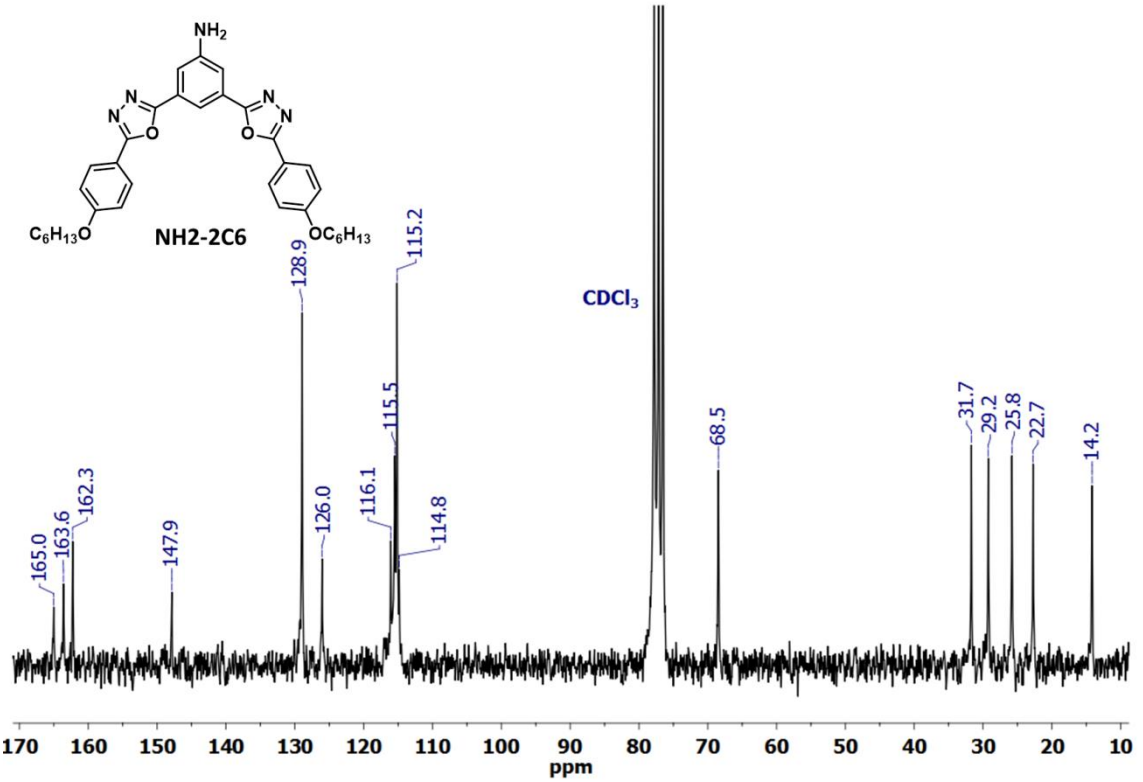

Figure S23. ^{13}C NMR spectrum ($CDCl_3$, 50MHz) of compound **NH2-2C6**.

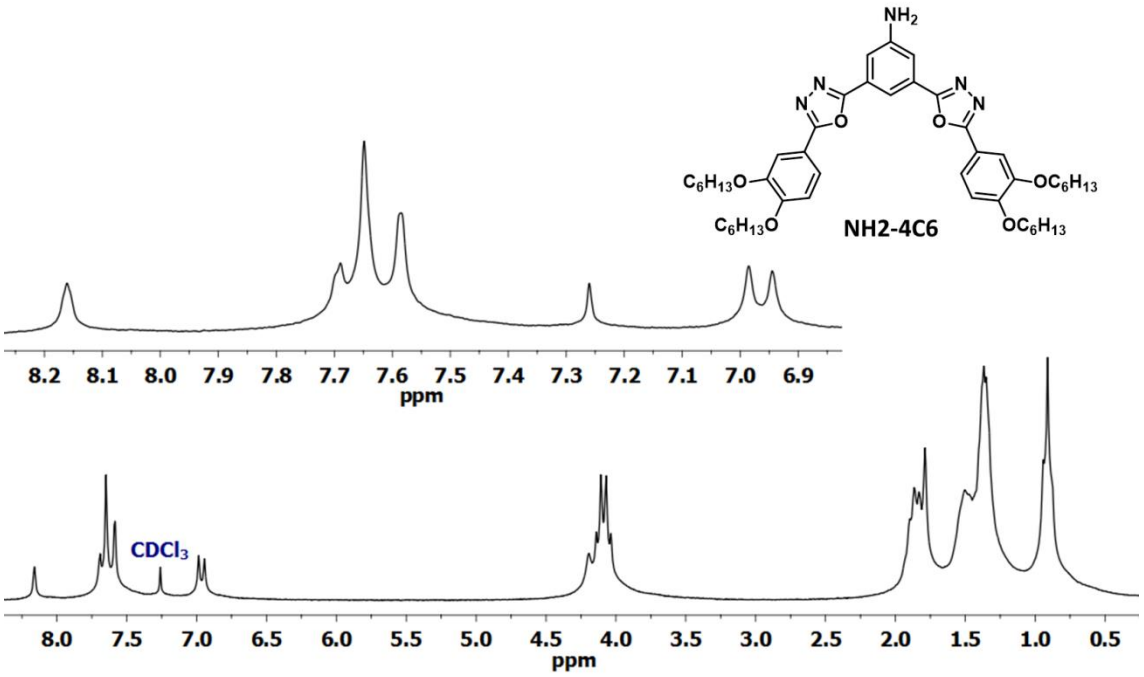

Figure S24.[1]H NMR spectrum ($CDCl_3$, 200MHz) of compound **NH2-4C6**.

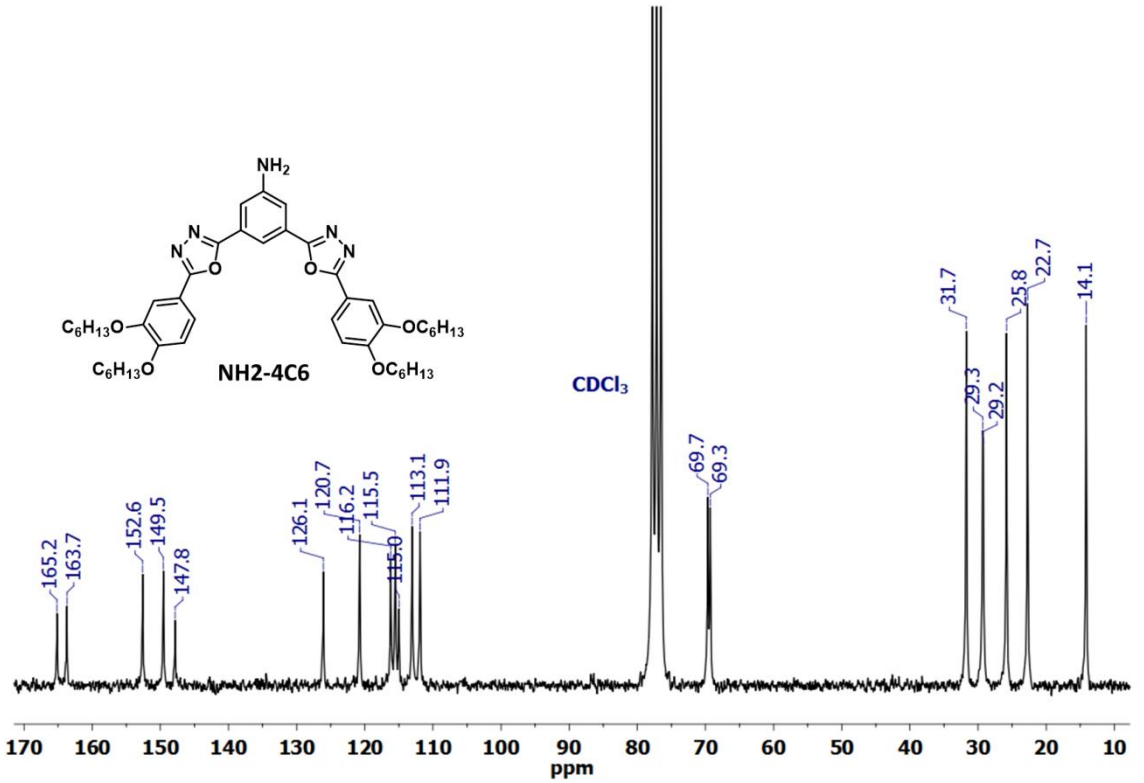

Figure S25.[13]C NMR spectrum ($CDCl_3$, 50MHz) of compound **NH2-4C6**.

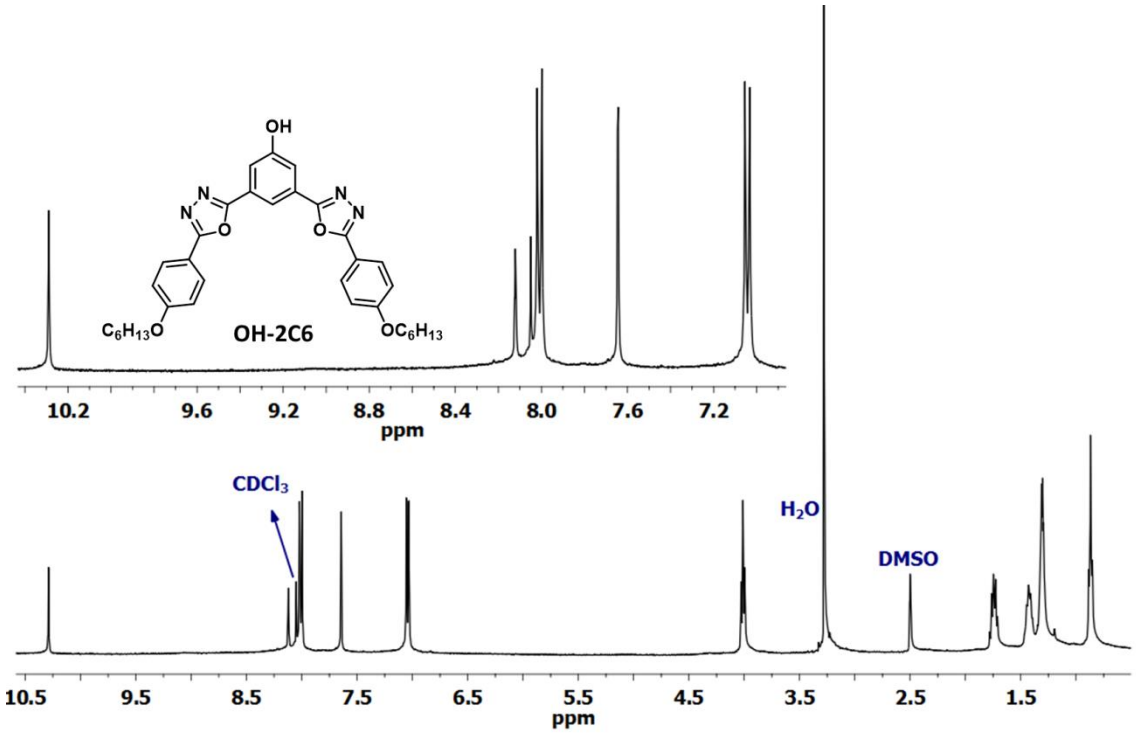

Figure S26.[1]H NMR spectrum (DMSO[D6]/$CDCl_3$, 200MHz) of compound **OH-2C6**.

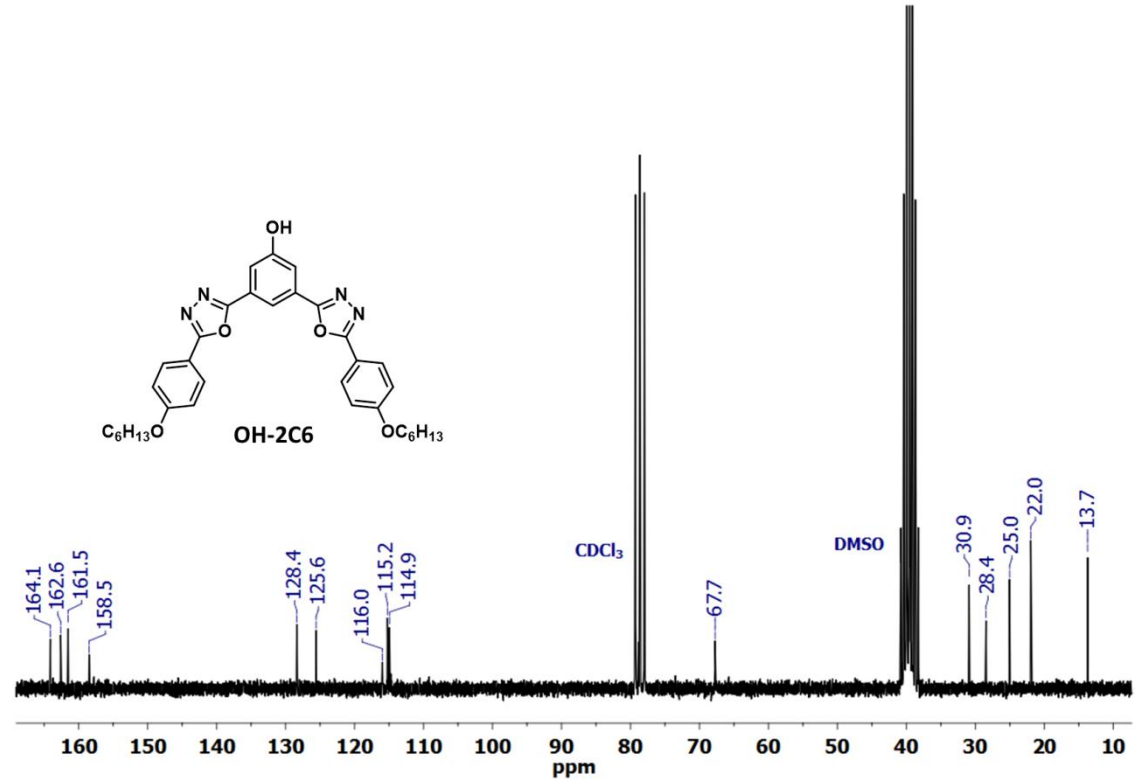

Figure S27.[13]C NMR spectrum (DMSO[D6]/$CDCl_3$, 50MHz) of compound **OH-2C6**.

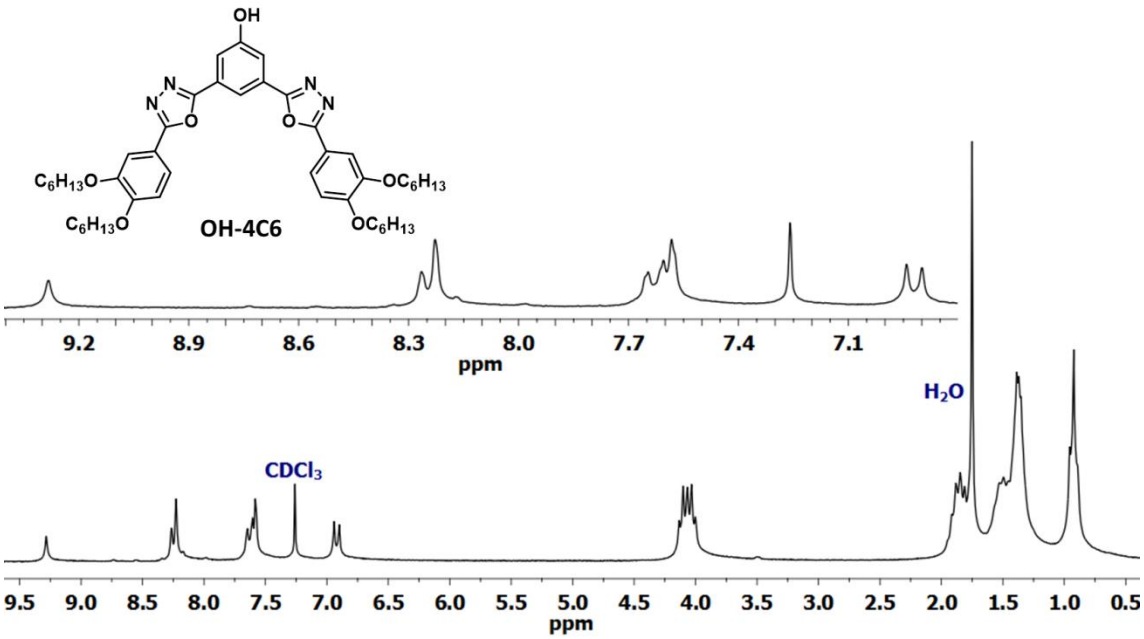

Figure S28.[1]H NMR spectrum ($CDCl_3$, 200MHz) of compound **OH-4C6**.

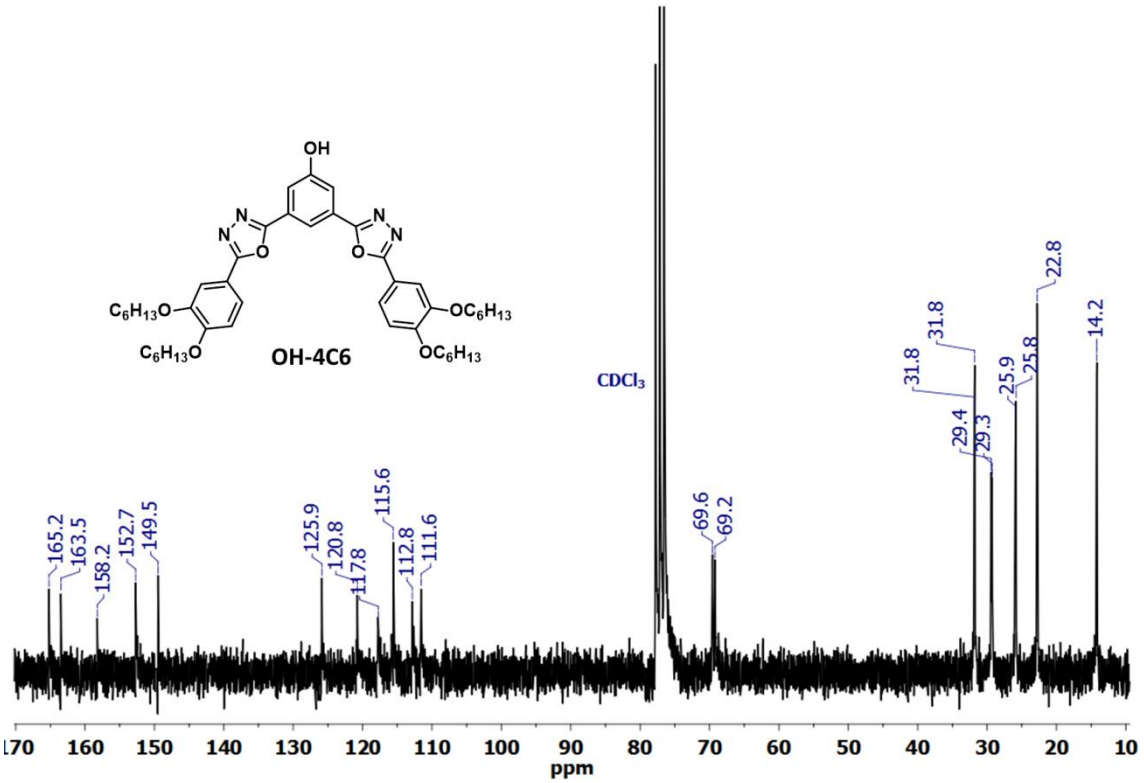

Figure S29.[13]C NMR spectrum ($CDCl_3$, 50MHz) of compound **OH-4C6**.

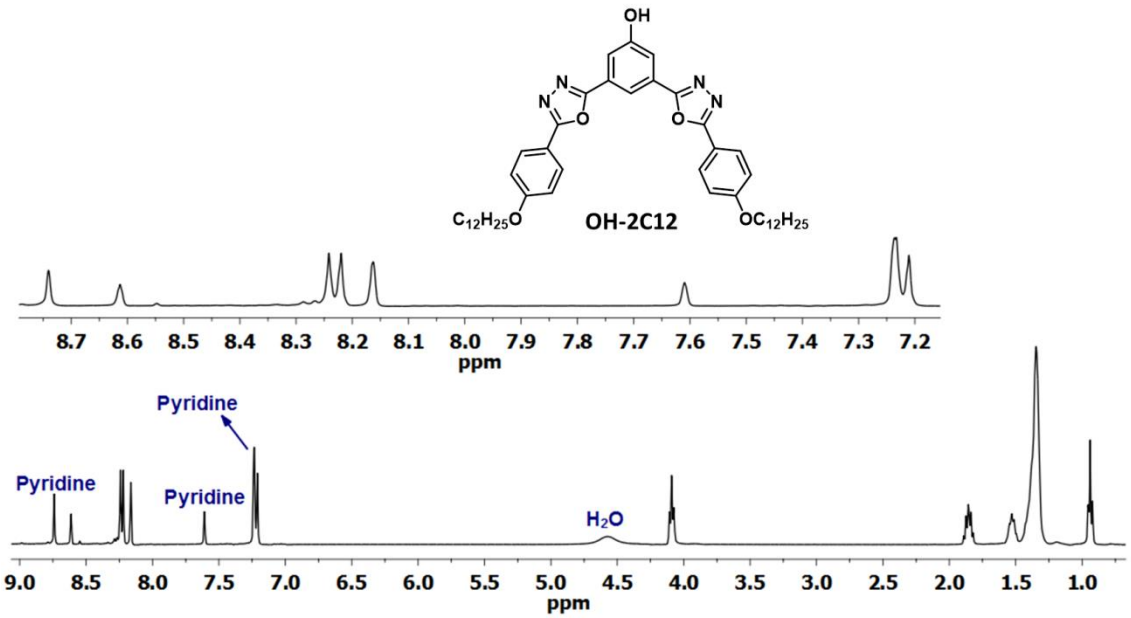

Figure S30.[1]H NMR spectrum (Pyridine[D5], 200MHz) of compound **OH-2C12**.

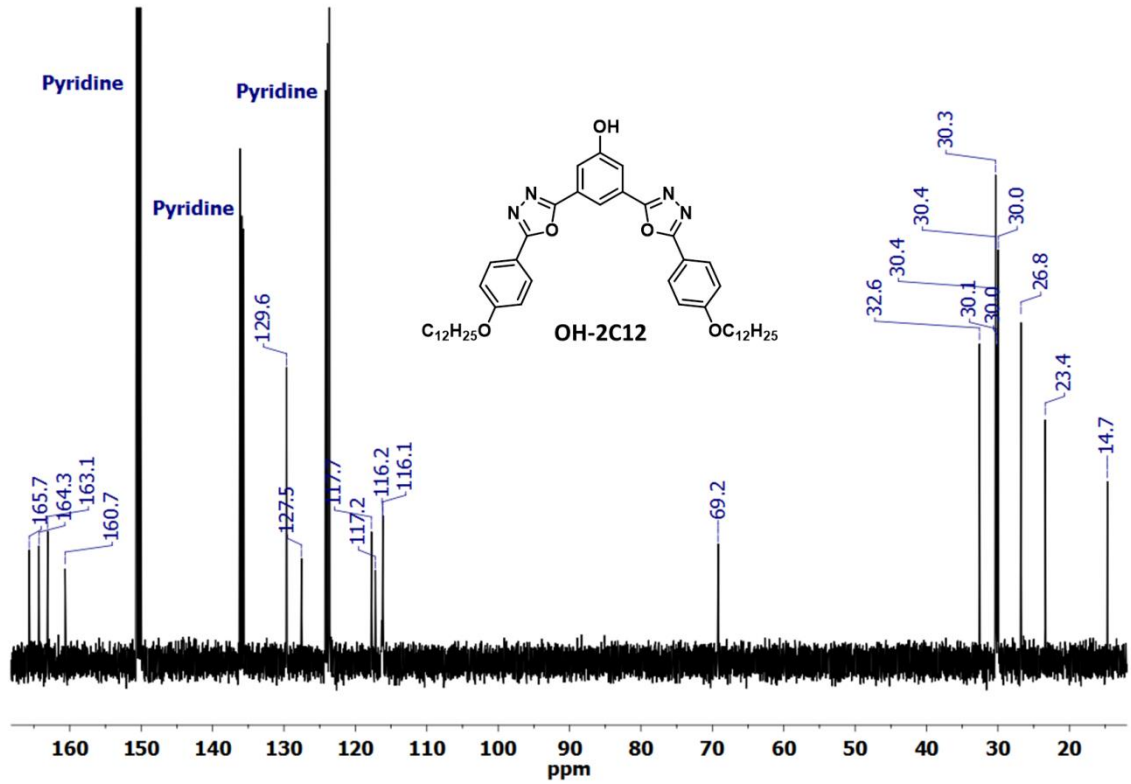

Figure S31.[13]C NMR spectrum (Pyridine[D5], 100MHz) of compound **OH-2C12**.

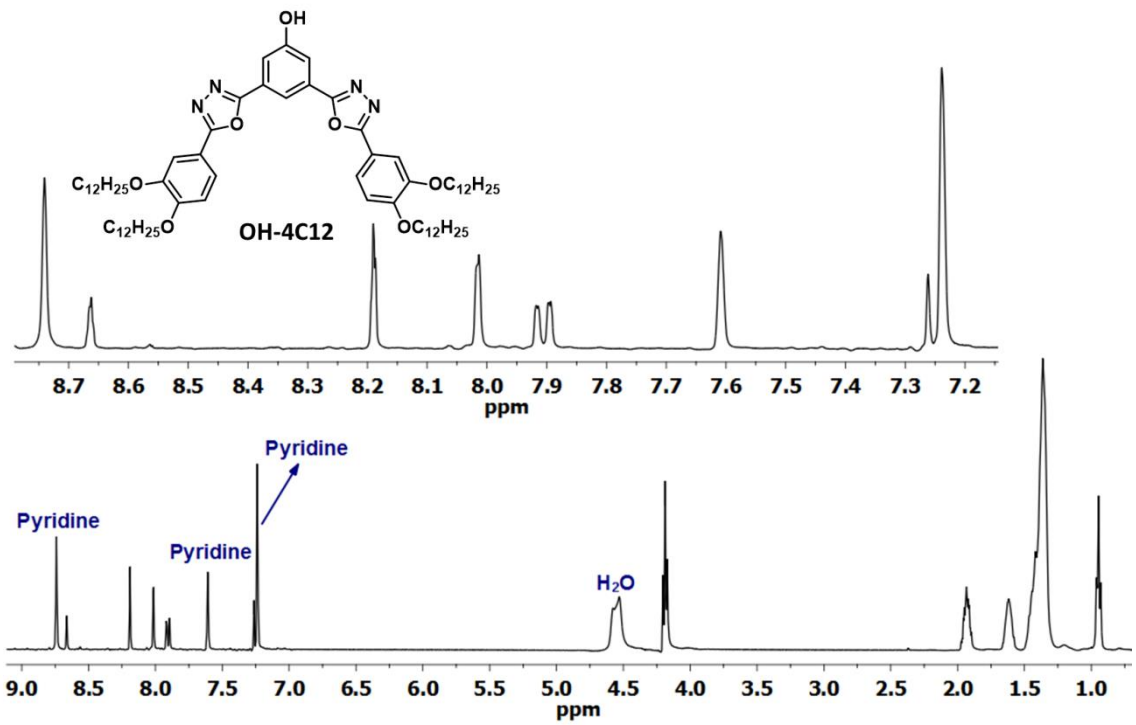

Figure S32.[1]H NMR spectrum (Pyridine[D5], 200MHz) of compound **OH-4C12**.

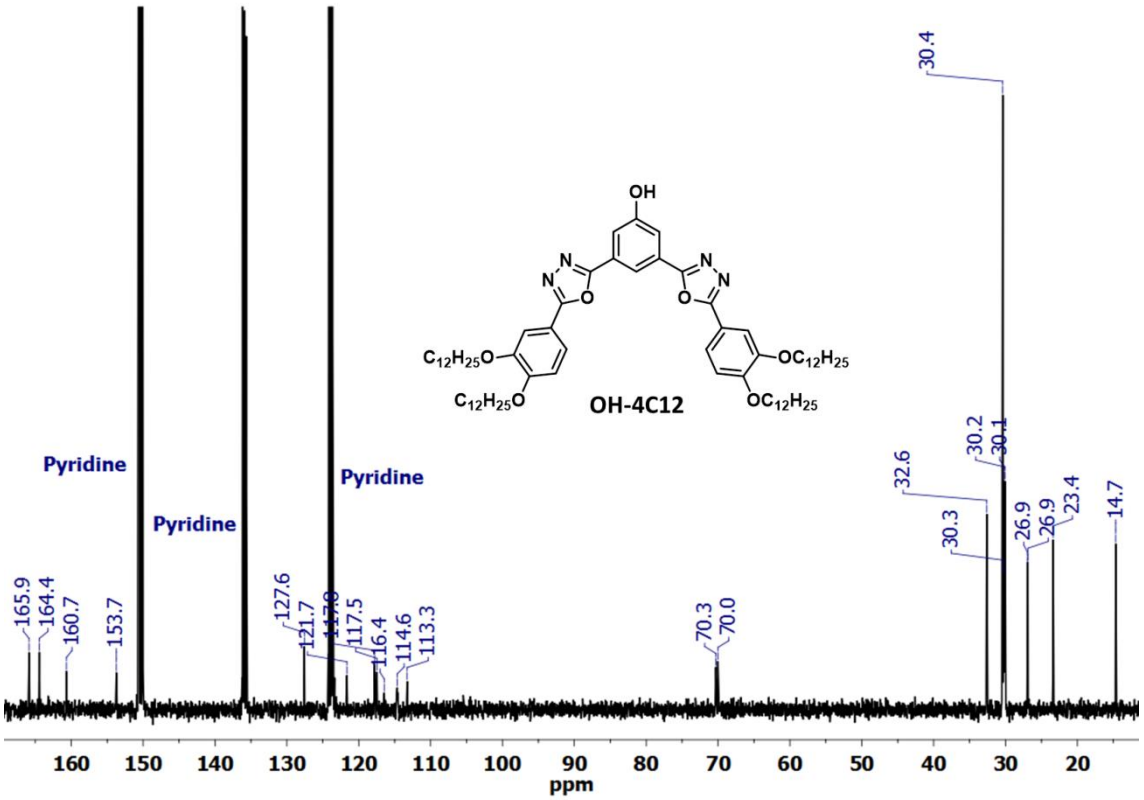

Figure S33.[13]C NMR spectrum (Pyridine[D5], 100MHz) of compound **OH-4C12**.

X-Ray Crystallography

A prismatic yellow crystal was selected from the crystalline sample and fixedat the end of a glass wire for X-ray analysis. The intensity data were collected with an Enraf-Nonius CAD4 diffractometer, at room temperature, with graphite-monochromated Mo Kα radiation. Cell parameters were determined from 25 carefully centered reflections using a standard procedure.[8] The collected data were corrected for Lorentz and polarization effects.[9] The structure was solved by direct methods and refined by full-matrix least-squares methods using SIR97[10] and SHELXL2012[11] programs, respectively. All non-hydrogen atoms were refined anisotropically. H atoms were placed at their calculated positions and included in the structure factor calculations. C-H distances were fixed at 0.93Å for H_{Ar} and 0.97 Å for H_{CH2} with $U_{iso}(H) = 1.2U_{eq}(C)$, and 0.97 Å with $1.5U_{eq}(C)$ for methyl groups. ORTEP was drawn with PLATON software.[12]

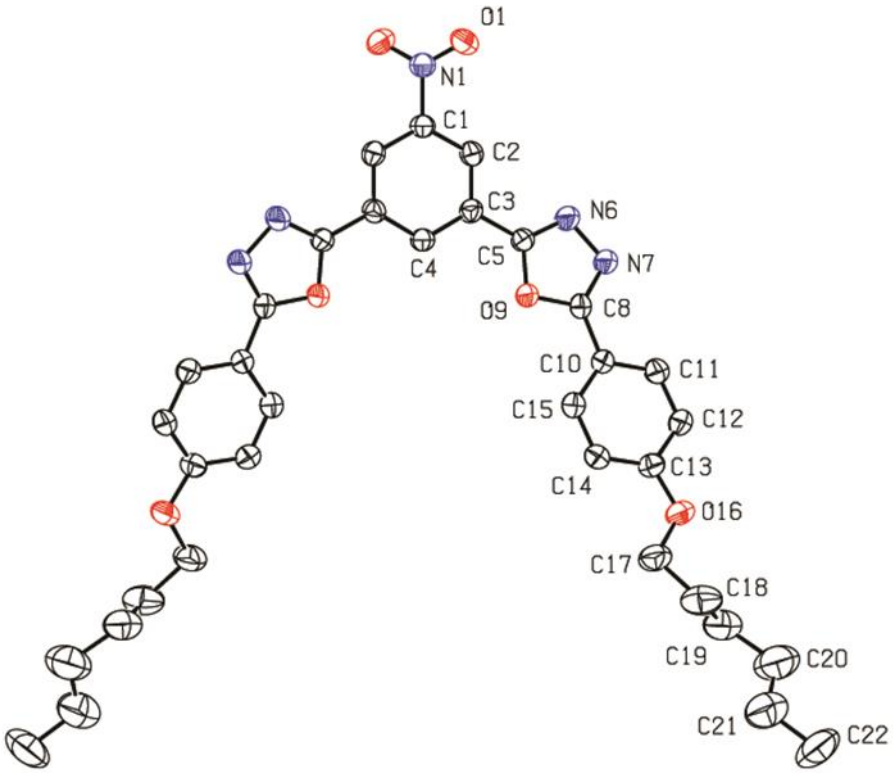

Figure S34. Ortep plot of **NO2-2C6** with labelling scheme. Ellipsoids are drawn at 40% probability level. H atoms were omitted for clarity.Symmetry code: -x+1, y, -z+1/2

Table S1. Crystal data and structure refinement for **NO2-2C6**.

Empirical formula	$C_{34}H_{37}N_5O_6$
Formula weight	611.68
Temperature	293(2) K
Wavelength	0.71069 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 20.583(2) Å
	b = 10.9650(10) Å
	c = 14.503(2) Å
	= 103.007(9)°
Volume	3189.2(6) $Å^3$
Z	4
Density (calculated)	1.274 Mg/m^3
Absorption coefficient	0.089 mm^{-1}
F(000)	1296
Crystal size	0.50 x 0.46 x 0.20 mm^3
Theta range for data collection	2.031 to 25.969°.
Index ranges	$-24 \le h \le 25$, $-13 \le k \le 0$, $-17 \le l \le 0$
Reflections collected	3258
Independent reflections	3125 (R_{int} = 0.0149)
Completeness to theta = 25.240°	99.8 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3125 / 0 / 205
Goodness-of-fit on F^2	1.066
Final R indices [I>2sigma(I)]	R1 = 0.0702, wR2 = 0.1984
R indices (all data)	R1 = 0.1125, wR2 = 0.2269
Largest diff. peak and hole	0.546 and -0.376 $e.Å^{-3}$

Table S2. Bond lengths [Å] and angles [°] for **NO2-2C6**.

N1-O1^{i}	1.219(3)	C8-C10	1.447(4)
N1-O1	1.219(3)	C10-C15	1.384(4)
N1-C1	1.474(5)	C10-C11	1.402(4)
C1-C2	1.386(3)	C11-C12	1.376(4)
C1-C2^{i}	1.386(3)	C12-C13	1.389(4)
C2-C3	1.387(4)	C13-O16	1.354(4)
C3-C4	1.394(3)	C13-C14	1.396(4)
C3-C5	1.467(4)	C14-C15	1.383(4)
C4-C3^{i}	1.394(3)	O16-C17	1.448(4)
C5-N6	1.279(4)	C17-C18	1.548(6)
C5-O9	1.362(3)	C18-C19	1.454(7)
N6-N7	1.409(3)	C19-C20	1.618(8)
N7-C8	1.292(4)	C20-C21	1.359(8)
C8-O9	1.374(3)	C21-C22	1.546(8)
O1^{i}-N1-O1	124.5(4)	C11-C10-C8	118.5(3)
O1^{i}-N1-C1	117.77(19)	C12-C11-C10	120.4(3)
O1-N1-C1	117.76(19)	C11-C12-C13	120.5(3)
C2-C1-C2^{i}	122.6(4)	O16-C13-C12	115.5(3)
C2-C1-N1	118.68(19)	O16-C13-C14	124.9(3)
C2^{i}-C1-N1	118.68(19)	C12-C13-C14	119.6(3)
C1-C2-C3	118.3(3)	C15-C14-C13	119.4(3)
C2-C3-C4	120.5(3)	C14-C15-C10	121.4(3)
C2-C3-C5	118.1(3)	C13-O16-C17	118.9(3)
C4-C3-C5	121.4(3)	O16-C17-C18	106.6(3)
C3^{i}-C4-C3	119.8(4)	C19-C18-C17	111.9(4)
N6-C5-O9	112.9(2)	C18-C19-C20	112.2(5)
N6-C5-C3	126.9(3)	C21-C20-C19	114.1(6)
O9-C5-C3	120.3(2)	C20-C21-C22	110.9(6)
C5-N6-N7	106.5(2)		
C8-N7-N6	106.3(2)		
N7-C8-O9	111.9(3)		
N7-C8-C10	127.8(3)		
O9-C8-C10	120.3(2)		
C5-O9-C8	102.5(2)		
C15-C10-C11	118.6(3)		
C15-C10-C8	122.9(3)		

Symmetry code: (i) -x+1, y, -z+1/2

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