Solvatochromism of symmetrical 2,6-distyrylpyridines. An experimental and theoretical study

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General procedure for the preparation of 2,6-bis[2-(4-methoxyphenyl)ethenyl]pyridine

*2,6-Bis[2-(4-methoxyphenyl)ethenyl]pyridine (**1**)* was synthesized using a modified literature procedure [S1]. A mixture of 2,6-lutidine (0.022 mol), anisaldehyde (0.044 mol) and acetic anhydride (20 mL) was refluxed for 72 hours. The reaction mixture was poured into cold water (100 mL) and shook, until the excess of acetic anhydride was completely hydrolyzed. The product was filtered, washed with ethanol and recrystallized from benzene.

*2,6-Bis[2-(4-methoxyphenyl)ethenyl]pyridine (**1**)* White crystalline compound; Yield 42 %; m.p. = 182 °C; FTIR (KBr) ν (cm^{-1}) = 3025, 2959, 2840, 1639, 1604, 1578, 1561, 1510, 1452, 1252, 1177, 1031, 970, 826, 736, 534; ^{1}H NMR (500 MHz, DMSO–d_6) δ: 3.84 (s, 6H, OCH_3), 6.92 (d, J = 9.0 Hz, 4H, Ar), 7.08 (d, J = 16.0 Hz, 2H, CH=CH), 7.21(d, J = 7.5 Hz, 2H, Py), 7.55 (d, J = 9.0 Hz, 4H, Ar), 7.60 (t, J = 7.5 Hz, 1H, Py), 7.65 (d, J = 16.0 Hz, 2H, CH=CH); ^{13}C NMR (125 MHz, DMSO–d_6) δ: 55.3 (OCH_3), 114.1 (Ar), 119.7 (Py), 126.3 (CH=CH), 128.4 (Ar), 129.6 (Ar), 132.3 (CH=CH), 136.8 (Py), 155.7 (Py), 159.8 (Ar).

*General procedure for the preparation of 2,6-bis[2-(4-hydroxyphenyl)ethenyl]pyridine derivatives (**2**–**5**)*

*2,6-Bis[2-(4-hydroxyphenyl)ethenyl]pyridine derivatives (**2**–**5**)* were synthesized using a modified literature procedure [S2]. 3-Ethoxy-4-hydroxy-5-nitrobenzaldehyde and 3-ethoxy-4-hydroxy-5-bromobenzaldehyde were previously synthesized according to the literature procedure [S3,S4]. A mixture of 2,6-lutidine (0.025 mol), corresponding 4-hydroxybenzaldehyde derivative (0.075 mol) and acetic anhydride (25 mL) was refluxed for 24 hours at 155 °C. The reaction mixture was poured into cold water (150 mL) and shaken, until the excess acetic anhydride was completely hydrolyzed. The product was filtered, washed with water and recrystallized repeatedly from ethanol. A mixture of obtained 2,6-bis-[2-(4-ethanoyloxyphenyl)ethenyl]pyridine derivative (0.038 mol) and 0.75 mol dm^{-3} alcoholic potassium hydroxide (15 mL) was refluxed for ninety minutes. Then, the reaction product was precipitated from the clear solution, as a voluminous powder, by a stream of carbon dioxide. 2,6-Bis[2-(4-hydroxyphenyl)ethenyl]pyridine derivatives (**2**–**5**) were recrystallized from ethanol.

*2,6-Bis[2-(4-hydroxyphenyl)ethenyl]pyridine (**2**)* Yellow powder; Yield 34 %; m.p. >300 °C; FTIR (KBr) ν (cm^{-1}) = 3252, 3046, 3026, 1632, 1604, 1582, 1559, 1512, 1458, 1254, 1208, 1173, 1004, 955, 830, 820, 801, 781, 740, 518; ^{1}H NMR (200 MHz, DMSO–d_6): δ 6.82 (d, J = 8.4 Hz, 4H, Ar), 7.08 (d, J = 16.2 Hz, 2H, CH=CH), 7.31(d, J = 7.8 Hz, 2H, Py), 7.52 (d, J = 9.0 Hz, 4H, Ar), 7.66 (d, J = 15.6 Hz, 2H, CH=CH), 7.69 (t, J = 7.5 Hz, 1H, Py), 9.70 (s, 2H, OH); ^{13}C NMR (50 MHz, DMSO–d_6): δ 115.9 (Ar), 120.1 (Py), 125.2 (CH=CH), 127.8 (Ar), 128.8 (Ar), 132.5 (CH=CH), 137.3 (Py), 155.4 (Py), 158.2 (Ar).

*2,6-Bis[2-(3-ethoxy-4-hydroxyphenyl)ethenyl]pyridine (**3**)* Dark goldenrod powder; Yield 32 %; m.p. >300 °C; FTIR (KBr) ν (cm^{-1}) = 3370, 3065, 2977, 2904, 1637, 1596, 1583, 1561, 1515, 1435, 1276, 1121, 1039, 964, 821, 811, 735, 610, 589, 539, 505; ^{1}H NMR (500 MHz, DMSO–d_6): δ 1.37 (t, J = 7.0 Hz, 6H, CH_3), 4.09 (q, J = 7.0 Hz, 4H, CH_2), 6.82 (d, J = 8.0 Hz, 2H, Ar), 7.10 (d, J = 8.0 Hz, 2H, Ar), 7.13 (d, J = 16.0 Hz, 2H, CH=CH), 7.26 (s, 2H, Ar), 7.32(d, J = 7.5 Hz, 2H, Py), 7.67 (d, J = 16.0 Hz, 2H, CH=CH), 7.71 (t, J = 7.5 Hz, 1H, Py), 9.18 (s, 2H, OH); ^{13}C NMR (125 MHz, DMSO–d_6): δ 14.8 (CH_3), 63.8 (CH_2), 111.7 (Ar), 115.7 (Ar), 119.9 (Ar), 120.9 (Py), 125.1 (CH=CH), 128.0 (Ar), 132.6 (CH=CH), 137.2 (Py), 147.0 (Ar), 147.6 (Ar), 155.1 (Py).

*2,6-Bis[2-(3-ethoxy-4-hydroxy-5-nitrophenyl)ethenyl]pyridine (**4**)* Red powder; Yield 35 %; m.p. >300 °C; FTIR (KBr) ν (cm^{-1}) = 3428, 2980, 2937, 2875, 1644, 1614, 1577, 1545, 1472, 1453, 1397, 1340, 1278, 1262, 1136, 1056, 969, 875, 847, 760, 607, 518; ^{1}H NMR (500 MHz, DMSO–d_6): δ 1.42 (t, J = 7.0 Hz, 6H, CH_3), 4.24 (q, J = 7.0 Hz, 4H, CH_2), 7.36 (d, J = 16.0 Hz, 2H, CH=CH), 7.39 (d, J = 7.5 Hz, 2H, Py), 7.63 (s, 2H, Ar), 7.75 (s, 2H, Ar), 7.76 (d, J = 16.0 Hz, 2H, CH=CH), 7.80 (t, J = 7.5 Hz, 1H, Py), 10.47 (s, 2H, OH); ^{13}C NMR (125 MHz, DMSO–d_6): δ 14.5 (CH_3), 65.0 (CH_2), 114.6 (Ar), 115.1 (Ar), 121.2 (Py), 127.5 (Ar), 128.0 (CH=CH), 130.6 (CH=CH), 137.2 (Ar), 137.5 (Py), 142.9 (Ar), 148.9 (Ar), 154.5 (Py).

*2,6-Bis[2-(3-ethoxy-4-hydroxy-5-bromophenyl)ethenyl]pyridine (**5**)* Light goldenrod powder; Yield 36 %; m.p. >300 °C; FTIR (KBr) ν (cm^{-1}) = 3500, 2975, 2933, 1635, 1599, 1579, 1557, 1499, 1457, 1428, 1394, 1282, 1185, 1046, 962, 911, 841, 830; ^{1}H NMR (200 MHz, DMSO–d_6):

δ 1.38 (t, J = 7.0 Hz, 6H, CH_3), 4.14 (q, J = 6.8 Hz, 4H, CH_2), 7.22 (*d*, J = 15.8 Hz, 2H, CH=CH), 7.29 (s, 2H, Ar), 7.32 (*d*, J = 8.0 Hz, 2H, Py), 7.41 (*s*, 2H, Ar), 7.70 (*d*, J = 16.4 Hz, 2H, CH=CH), 7.75 (*t*, J = 8.0 Hz, 1H, Py), 9.52 (*s*, 2H, OH); ^{13}C NMR (50 MHz, DMSO–d_6): δ 14.8 (CH_3), 64.8 (CH_2), 109.9 (Ar), 110.6 (Ar), 121.0 (Py), 123.9 (Ar), 126.9 (CH=CH), 129.3 (Ar), 131.5 (CH=CH), 137.7 (Py), 144.6 (Ar), 147.9 (Ar), 155.0 (Py).

*General procedure for the preparation of 1-methyl-2,6-bis[2-(4-hydroxyphenyl)ethenyl]pyridinium iodide (**6**)*

A mixture of 2,6-lutidine (0.025 mol) and methyl-iodide (0.125 mol) was stirred at room temperature for 24 h. After that, the product was filtered and recrystallized repeatedly from ethanol. A mixture of obtained 1-methyl-2,6-dimethylpiridinium iodide (0.01 mol), 4-hydroxybenzaldehyde (0.023 mol), 2 mL piperidine and 50 mL abs. ethanol was refluxed for 3 days. The reaction mixture was cooled to room temperature and the product was filtered, then recrystallized from dimethylformamide.

*1-Methyl-2,6-bis[2-(4-hydroxyphenyl)ethenyl]pyridinium iodide (**6**)* Red crystalline compound; Yield 31 %; m.p. >300 °C; FTIR (KBr) ν (cm^{-1}) = 3441, 2934, 2805, 1660, 1603, 1591, 1562, 1514, 1483, 1392, 1391, 1324, 1268, 1231, 1171, 1102, 940, 834; ^{1}H NMR (200 MHz, DMSO–d_6): δ 4.24 (s, 3H, CH_3), 6.87 (d, J = 9.0 Hz, 4H, Ar), 7.42 (*d*, J = 15.6 Hz, 2H, CH=CH), 7.66–7.74 (m, 6H, Ar and CH=CH), 8.17 (d, J = 7.2 Hz, 2H, Py), 8.32 (t, J = 7.2 Hz, 1H, Py); ^{13}C NMR (50 MHz, DMSO–d_6): δ 115.4 (CH_3), 116.2 (Ar), 122.6 (CH=CH), 126.2 (Py), 130.8 (Ar), 142.5 (Ar), 142.7 (CH=CH), 153.8 (Py), 160.7 (Py), 162.5 (Ar).

*General procedure for the preparation of 2,6-bis[2-(4-(4-*n*-heptyloxybenzoyloxy)phenyl)ethenyl]pyridine (7)*

*2,6-Bis[2-(4-(4-*n*-heptyloxybenzoyloxy)phenyl)ethenyl]pyridine* (**7**) was prepared by acylation of compound (**2**) with the 4-*n*-heptyloxybenzoyl chloride [S5]. To an ice-cold solution of 2,6-bis[2-(4-hydroxyphenyl)ethenyl]pyridine **(2)** (3.00 mmol) in dry pyridine (30 mL), 4-*n*-heptyloxybenzoyl chloride (6.00 mmol) was added slowly. The reaction was carried out at room

temperature for 2 days. The product was filtered, and recrystallized from acetone and toluene successively.

*2,6-Bis[2-(4-(4-*n*-heptyloxybenzoyloxy)phenyl)ethenyl]pyridine* (**7**) White powder; Crystal 179 °C B1 186 °C Isotropic liquid; Yield 43 %; FTIR (KBr) ν (cm^{-1}) = 2929, 2856, 1727, 1606, 1579, 1562, 1510, 1460, 1257, 1212, 1164, 1073, 762; ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.90 (t, J = 7.0 Hz, 6H, CH_3), 1.32-1.50 (m, 16H, CH_2), 1.82 (qu, J = 7.0 Hz, 4H, CH_2), 4.04 (t, J = 6.5 Hz, 4H, CH_2), 6.97 (d, J = 8.5 Hz, 4H, Ar), 7.18 (d, J = 16.0 Hz, 2H, CH=CH), 7.24 (d, J = 8.5 Hz, 4H, Ar), 7.27 (d, J = 7.5 Hz, 2H, Py), 7.64 (t, J = 7.5 Hz, 1H, Py), 7.66 (d, J = 8.5 Hz, 4H, Ar), 7.73 (d, J = 16.5 Hz, 2H, CH=CH), 8.15 (d, J = 9.0 Hz, 4H, Ar); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.06 (CH_3), 22.58 (CH_2), 25.92 (CH_2), 29.01 (CH_2), 29.08 (CH_2), 31.73 (CH_2), 68.32 (CH_2), 114.3 (Ar), 120.5 (Ar), 121.4 (Py), 122.1 (Ar), 128.1 (Ar), 128.3 (CH=CH), 132.0 (CH=CH), 132.3 (Ar), 134.4 (Ar), 137.0 (Py), 151.0 (Ar), 155.3 (Py), 163.6 (Ar), 164.8 (C=O).

Table S1 Solvent polarity parameter sets of Kamlet-Taft and Catalán, for the selected solvents

Solvent	Kamlet-Taft			Catalán			
	π*	β	α	SA	SB	SP	SdP
1,4-Dioxane	0.55	0.37	0.00	0.000	0.444	0.737	0.312
Acetonitrile	0.75	0.31	0.19	0.044	0.286	0.645	0.974
Anisole	0.73	0.22	0.00	0.084	0.299	0.820	0.543
Diethyl ether	0.27	0.47	0.00	0.000	0.562	0.617	0.385
Dichloromethane	0.82	0.00	0.30	0.040	0.178	0.761	0.769
Diisopropyl ether	0.27	0.49	0.00	0.000	0.657	0.625	0.324
Dimethylacetamide	0.88	0.76	0.00	0.028	0.650	0.763	0.987
Dimethylformamide	0.88	0.69	0.00	0.031	0.613	0.759	0.977
Dimethyl sulfoxide	1.00	0.76	0.00	0.072	0.647	0.830	1.000
Ethyl acetate	0.55	0.45	0.00	0.000	0.542	0.656	0.603
Ethanol	0.54	0.77	0.83	0.400	0.658	0.633	0.783
Ethylene glycol	0.92	0.52	0.90	0.717	0.534	0.777	0.910
Formamide	0.97	0.48	0.71	0.549	0.414	0.814	1.006
Chloroform	0.58	0.00	0.44	0.047	0.071	0.783	0.614
2-Propanol	0.48	0.95	0.76	0.283	0.830	0.633	0.808
Methanol	0.60	0.62	0.93	0.605	0.545	0.608	0.904
1-Butanol	0.47	0.88	0.79	0.341	0.809	0.674	0.655
N-Methylformamide	0.90	0.80	0.62	–	–	–	–
1-Propanol	0.52	0.90	0.78	0.367	0.782	0.658	0.748
Pyridine	0.87	0.64	0.00	0.033	0.581	0.842	0.761
Acetic acid	0.64	0.45	1.12	0.689	0.390	0.651	0.676
tert-Butanol	0.41	1.01	0.68	0.145	0.928	0.632	0.732
Tetrahydrofuran	0.58	0.55	0.00	0.000	0.591	0.714	0.634
Toluene	0.54	0.11	0.00	0.000	0.128	0.782	0.284

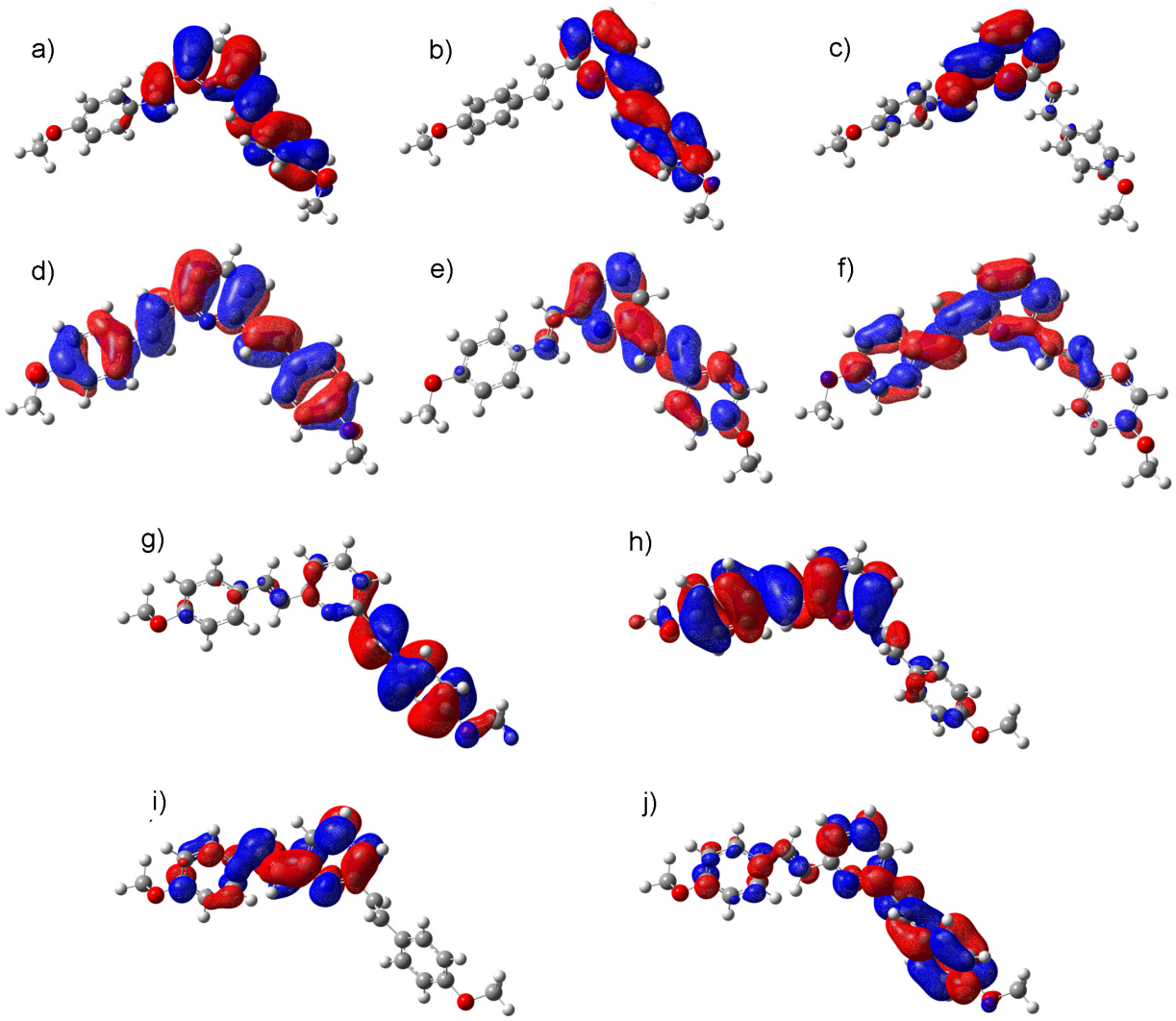


Fig. S1. a) HOMO orbital for conformation **I**; b) LUMO orbital for conformation **I**; c) LUMO+1 orbital for conformation **I**; d) HOMO orbital for conformation **II**; e) LUMO orbital for conformation **II**; f) LUMO+1 orbital for conformation **II**; g) HOMO–1 orbital for conformation **III**; h) HOMO orbital for conformation **III**; i) LUMO orbital for conformation **III**; j) LUMO+2 orbital for conformation **III**. Conformations depicted in Fig. 7 (a–c) in the main text.

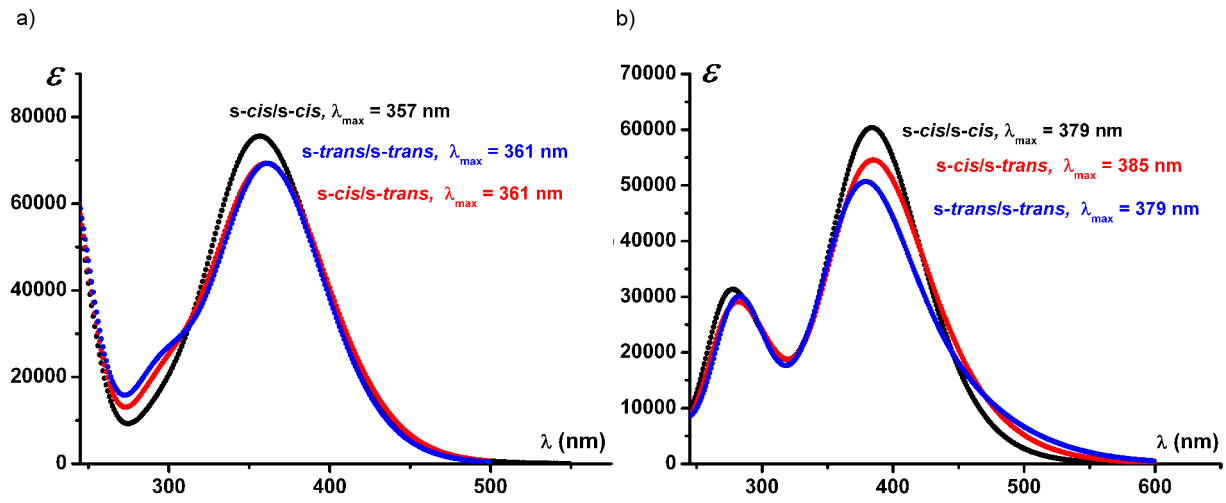


Fig. S2. UV-Vis spectra of compound **1** in s-*cis*/s-*cis*, s-*trans*/s-*trans* and s-*cis*/s-*trans* conformations (Fig. **7** in the main text), as obtained with MP2 method, calculated by: a) semiempirical ZINDO/S method; b) TD-DFT calculations with 6-311G basis set

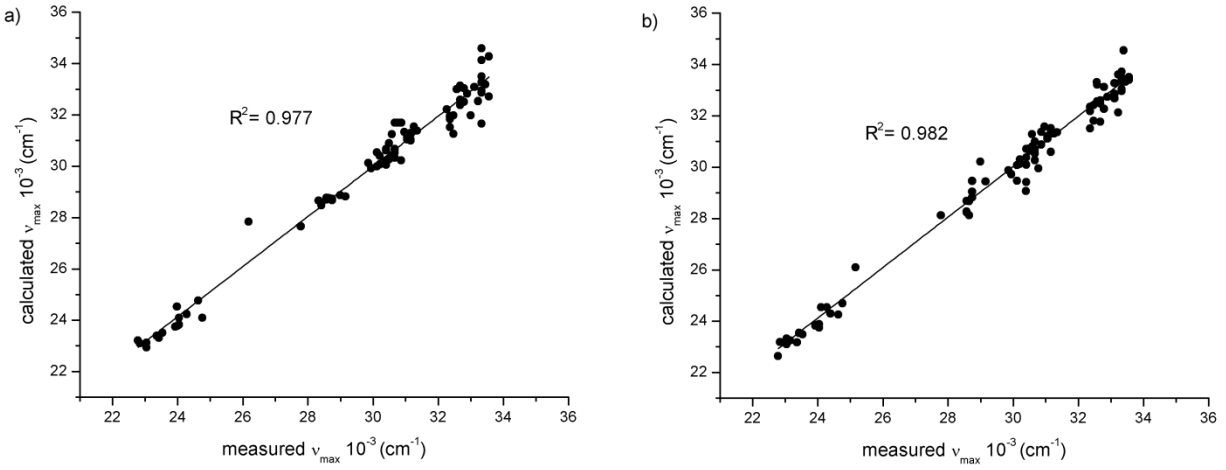
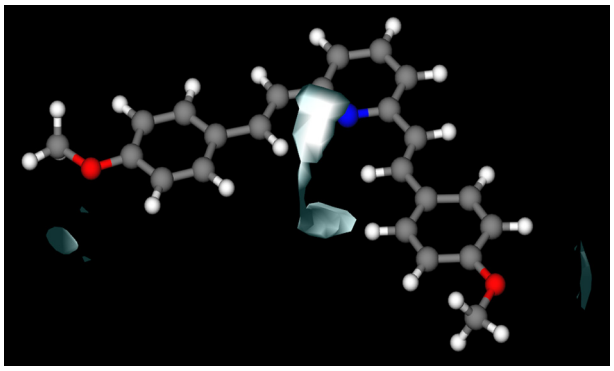
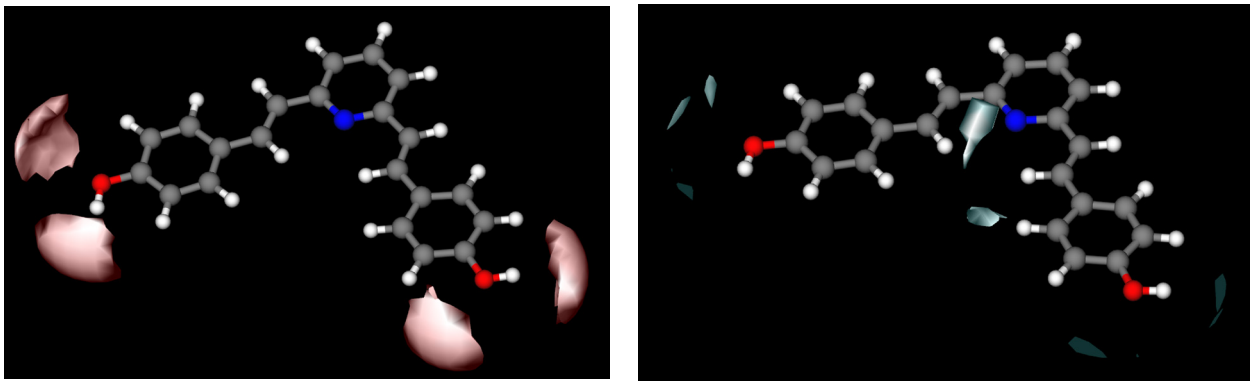


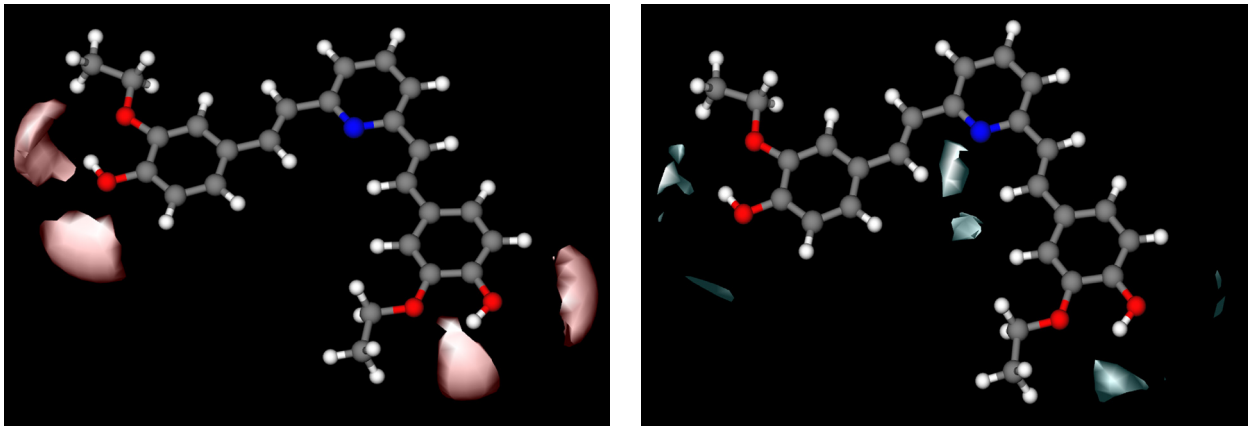
Fig. S3. Relationship between calculated and measured ν_{max} values according to a) Kamlet-Taft and b) Catalán models, described in the main text



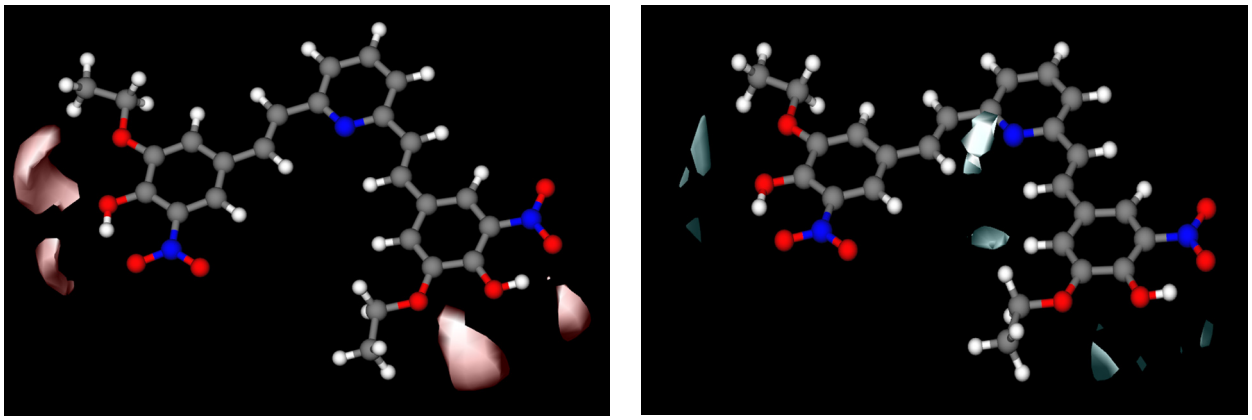
Compound **1**



Compound **2**



Compound **3**



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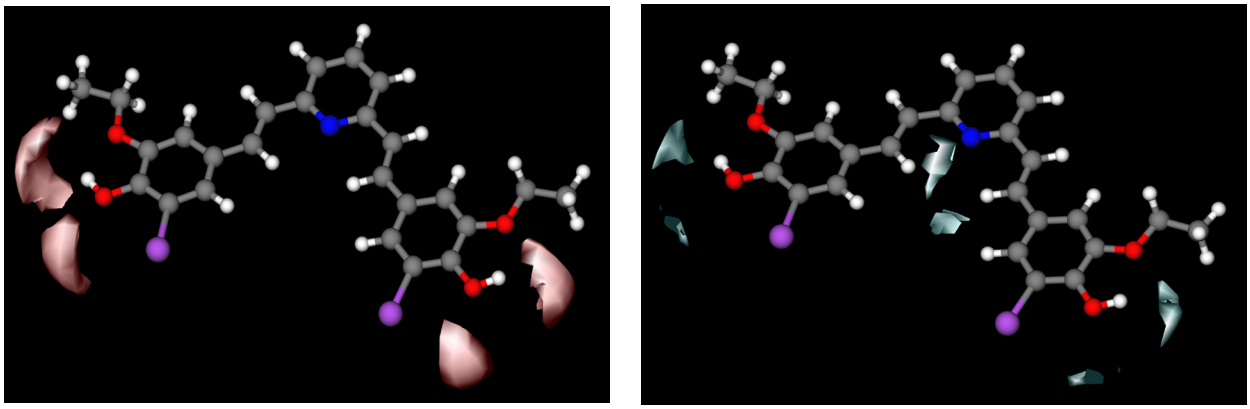
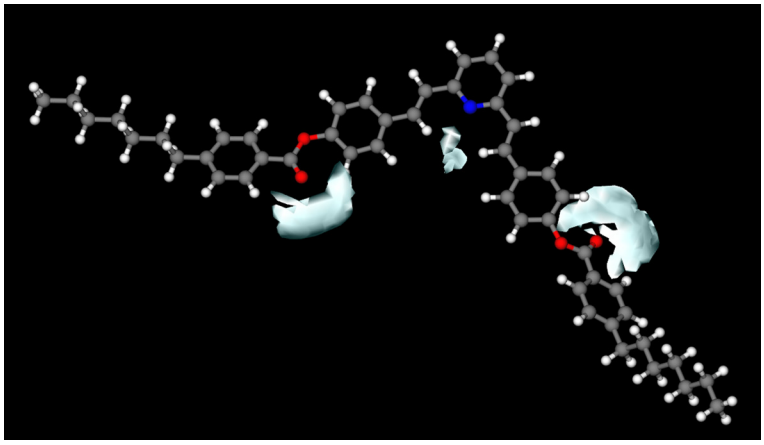


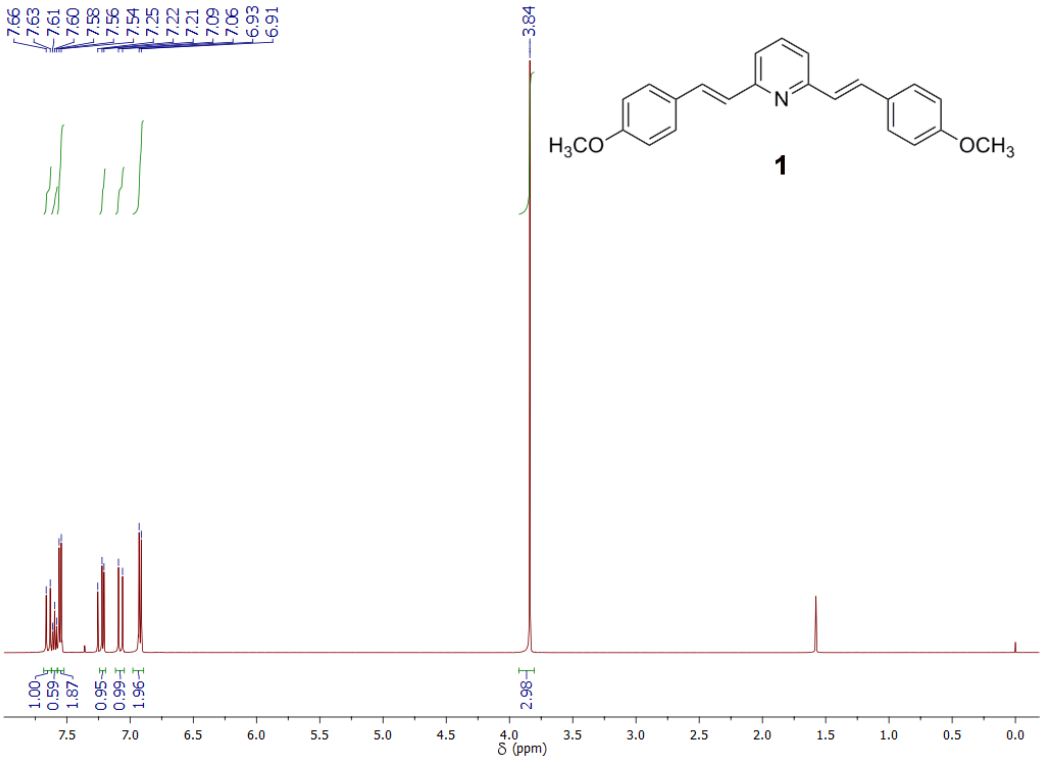
Compound **5**



Compound **7**

Fig. S4. Molecular interaction fields, obtained with the GRID HBD probe (O1), light blue, depicted on isocontour level of –4.5 (–3.8 for compound **2**) kcal/mol; and HBA probe (OC2), red, depicted on isocontour level of –2.0 kcal/mol

NMR spectra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7.56
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6.91
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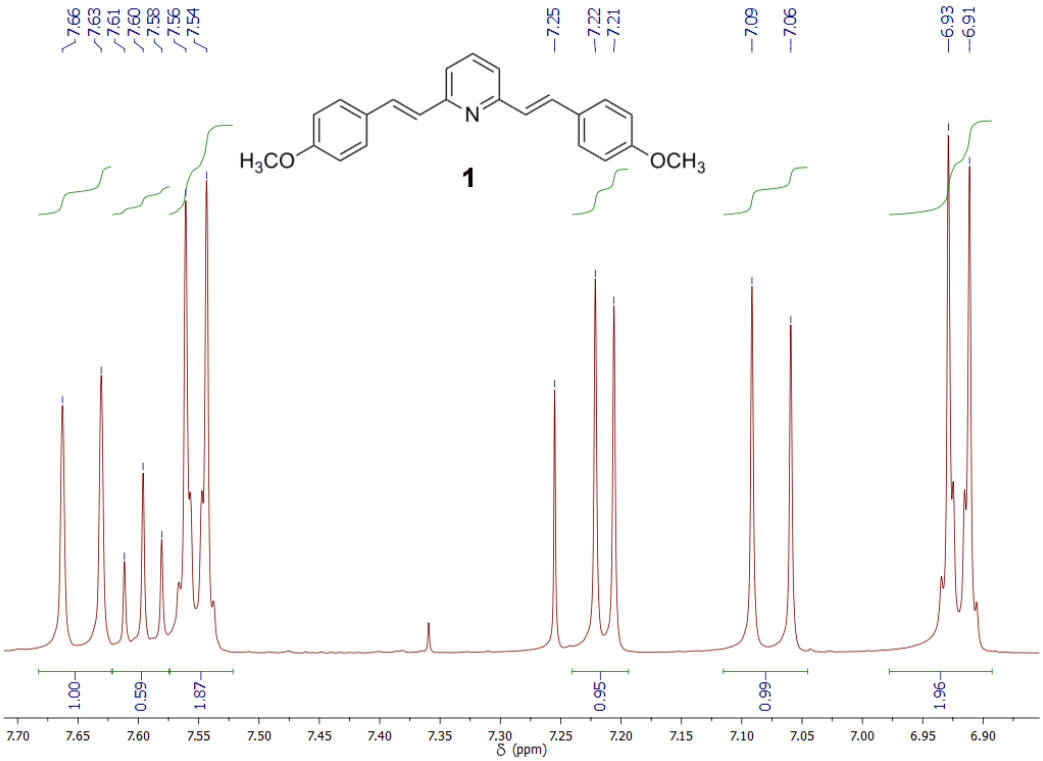
N
1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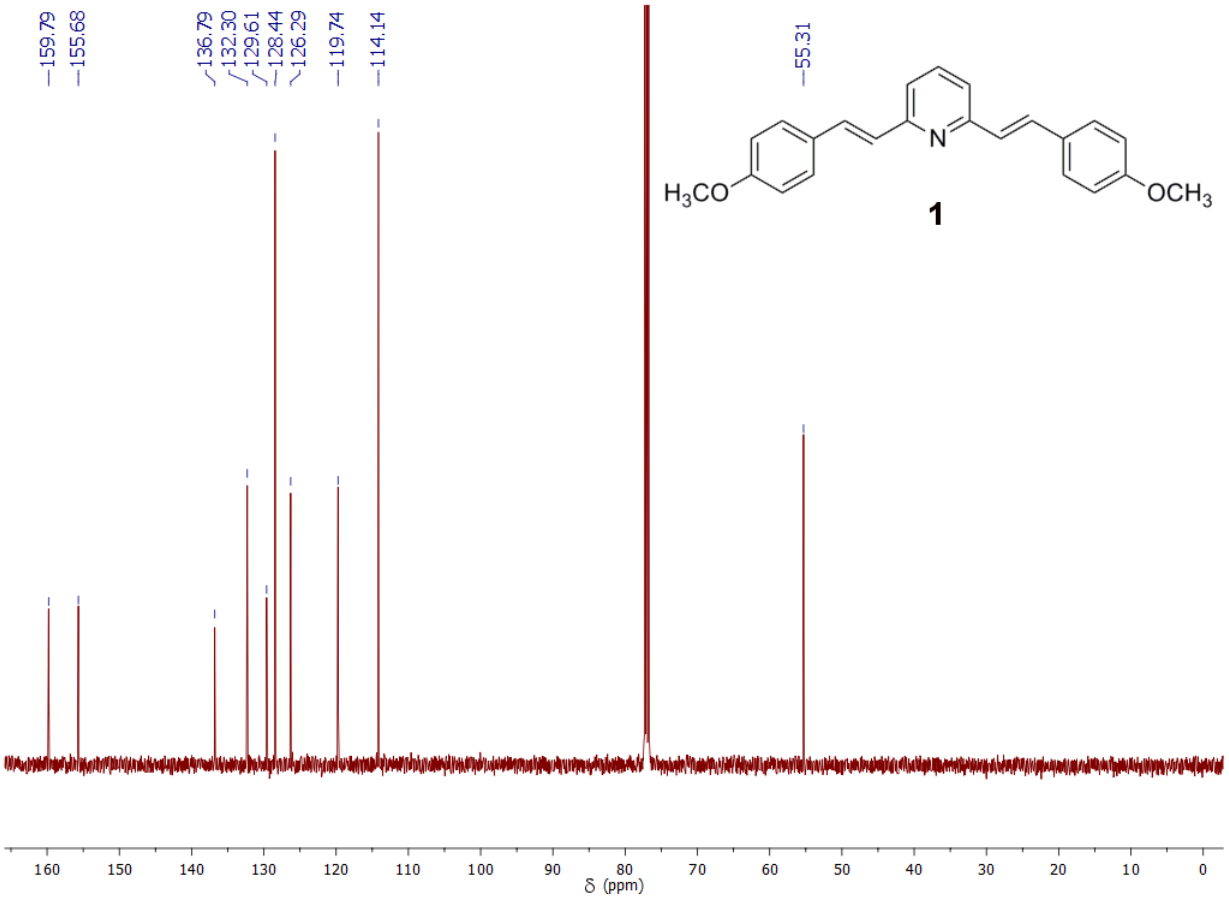
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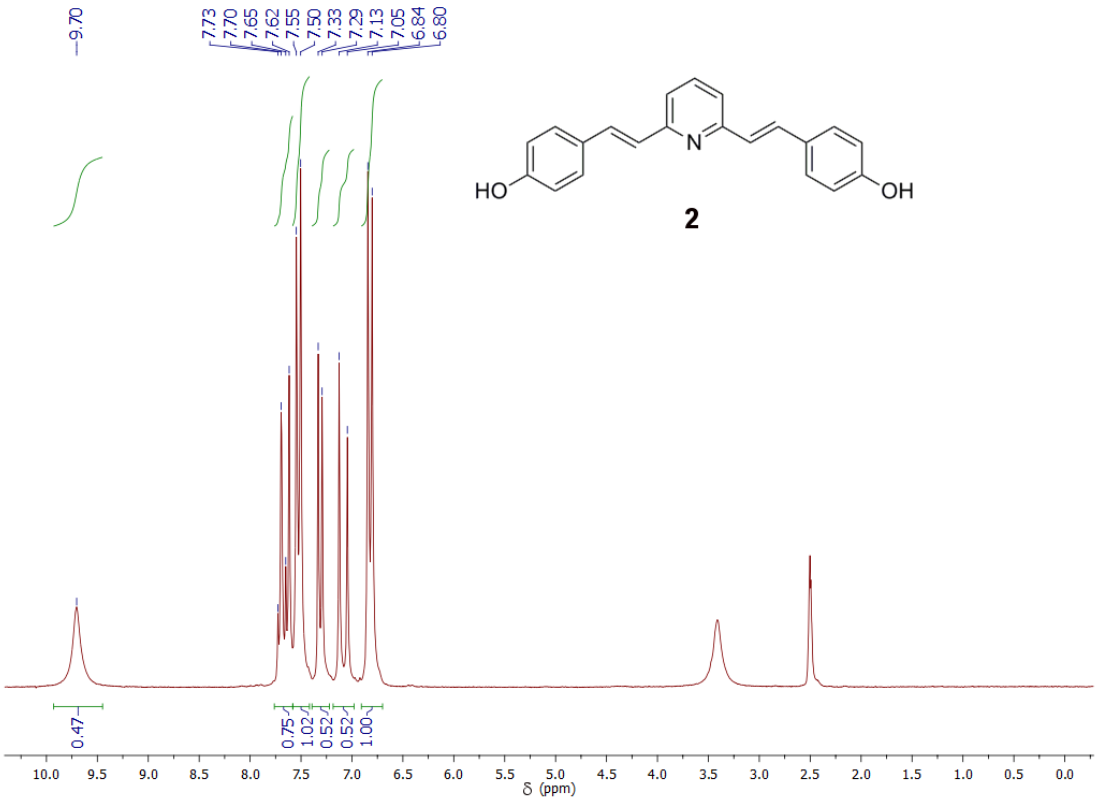
δ (ppm)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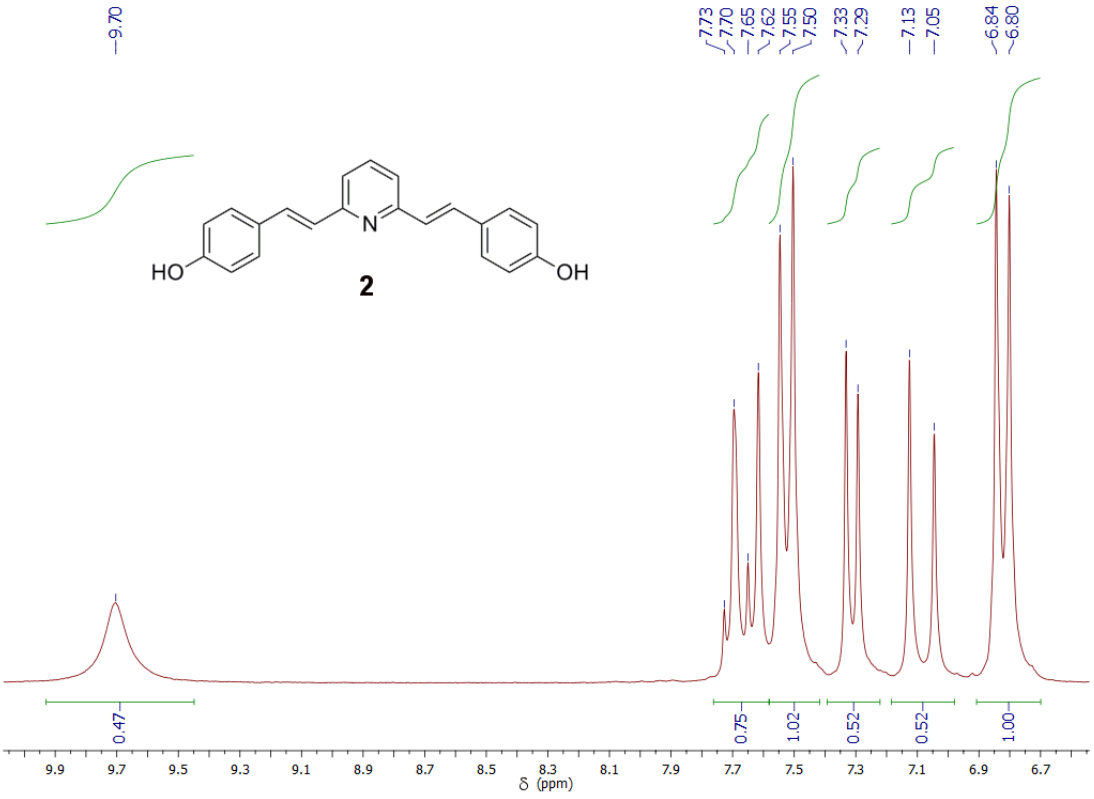
δ (ppm)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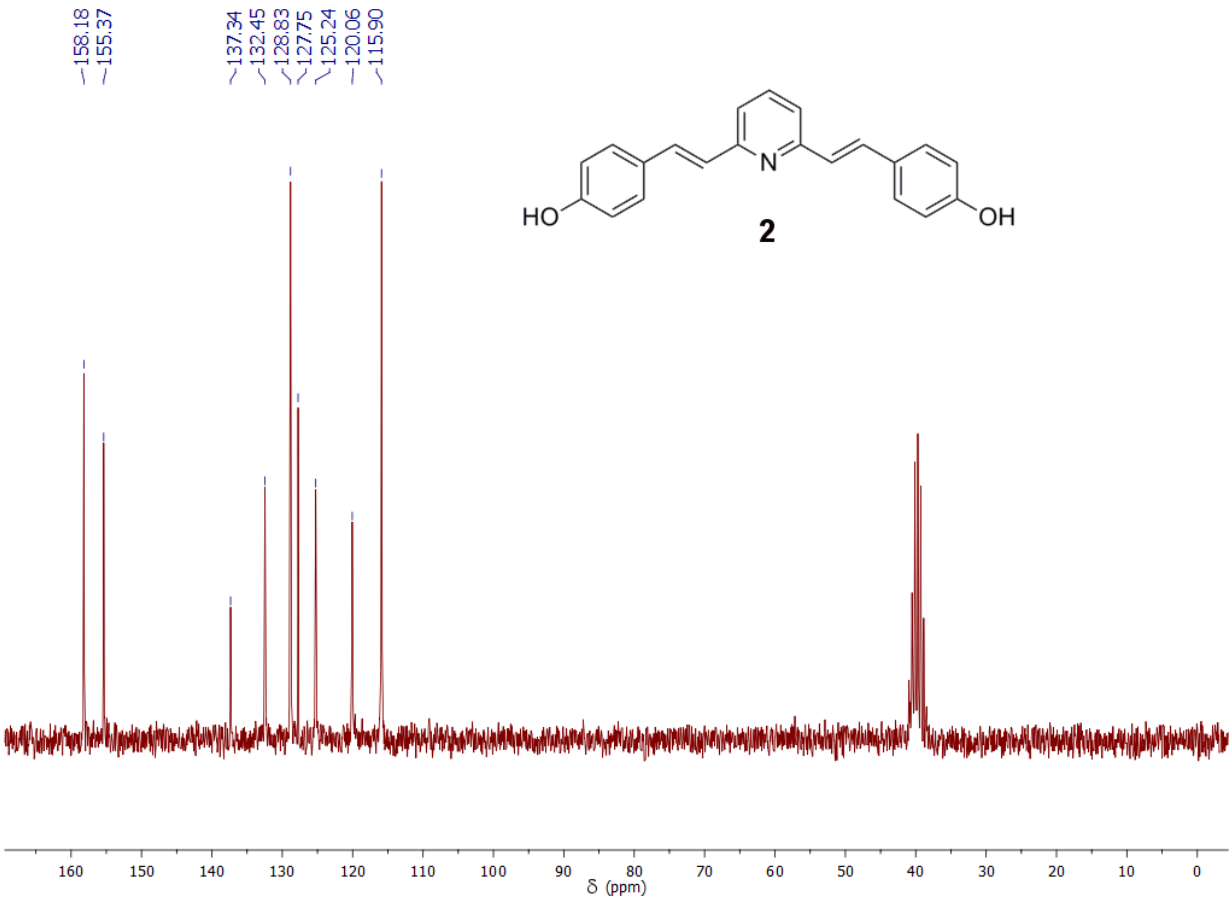
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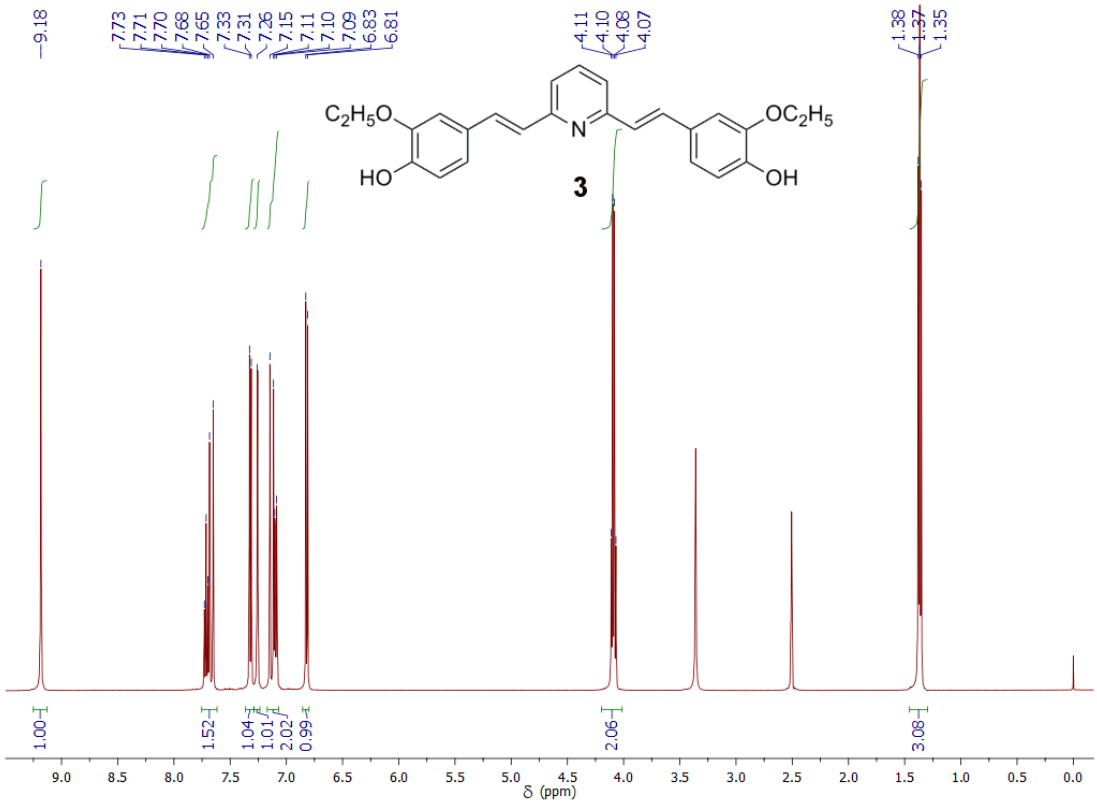
δ (ppm)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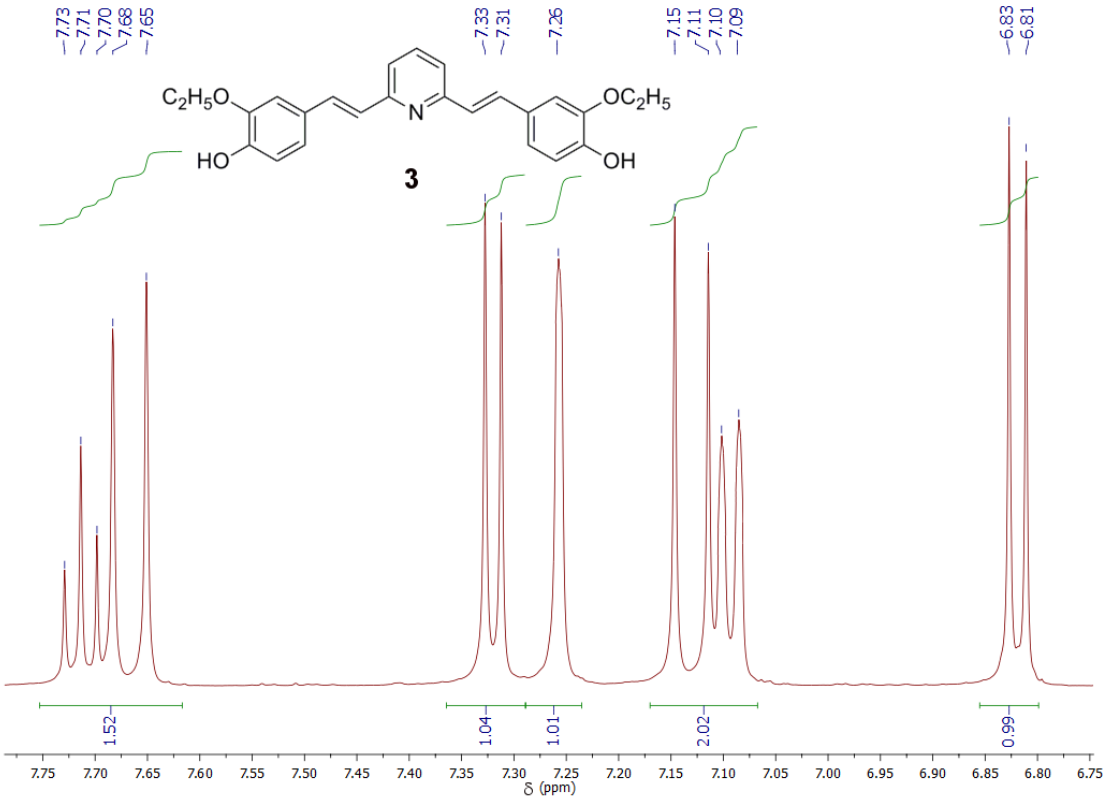
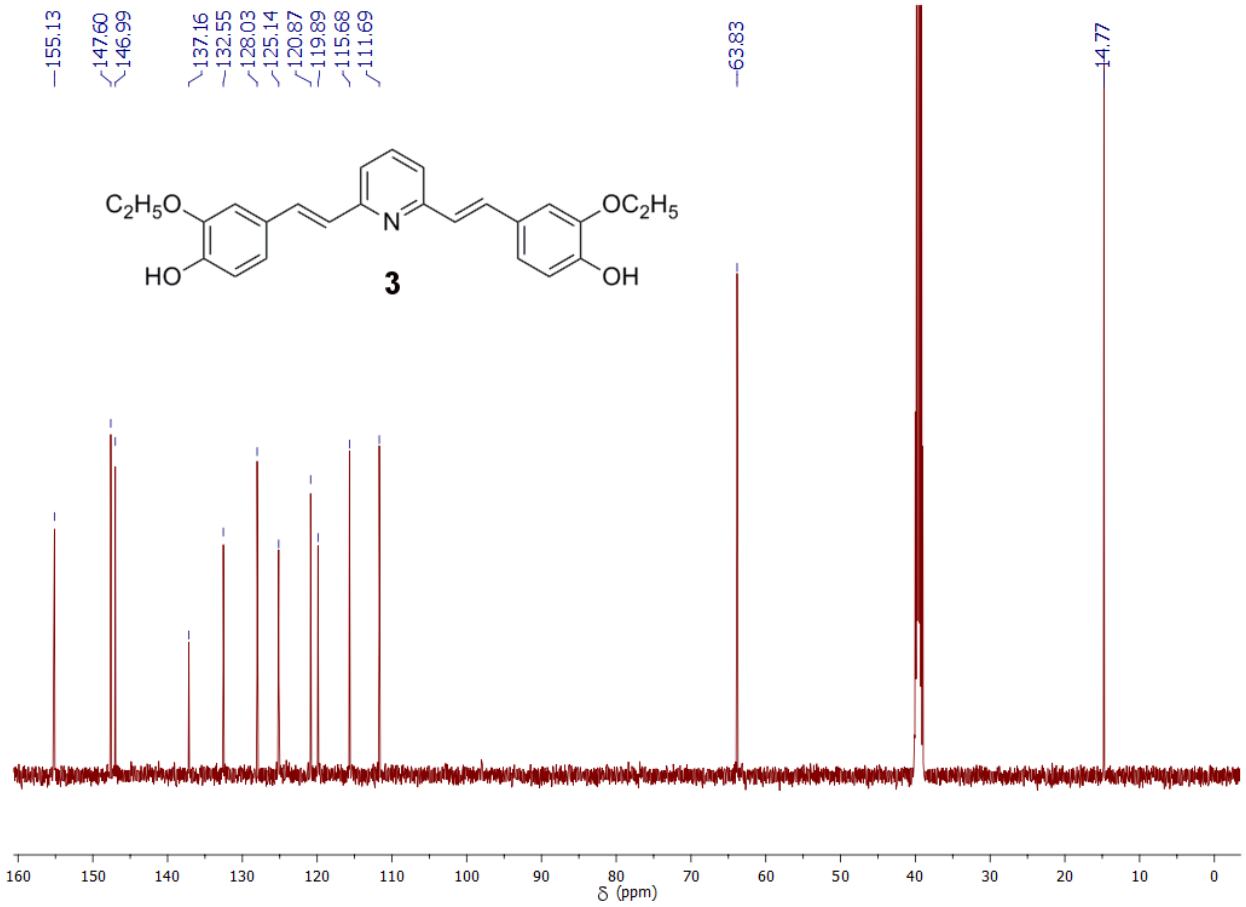
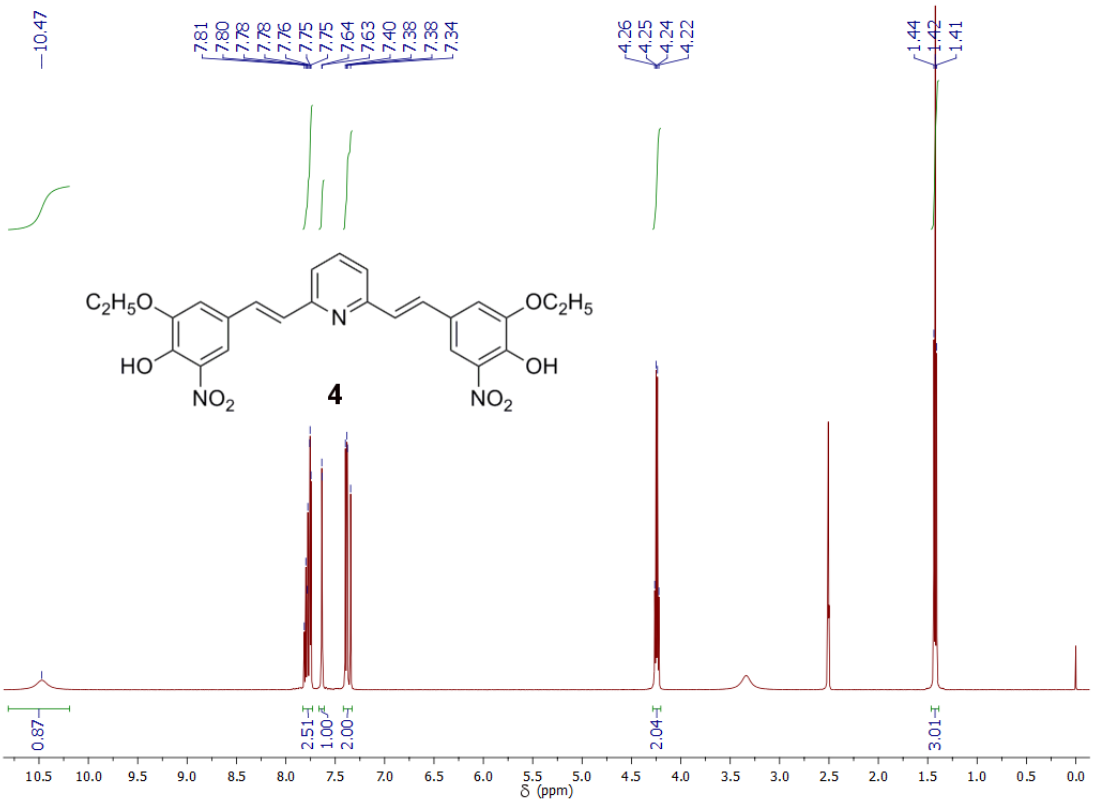
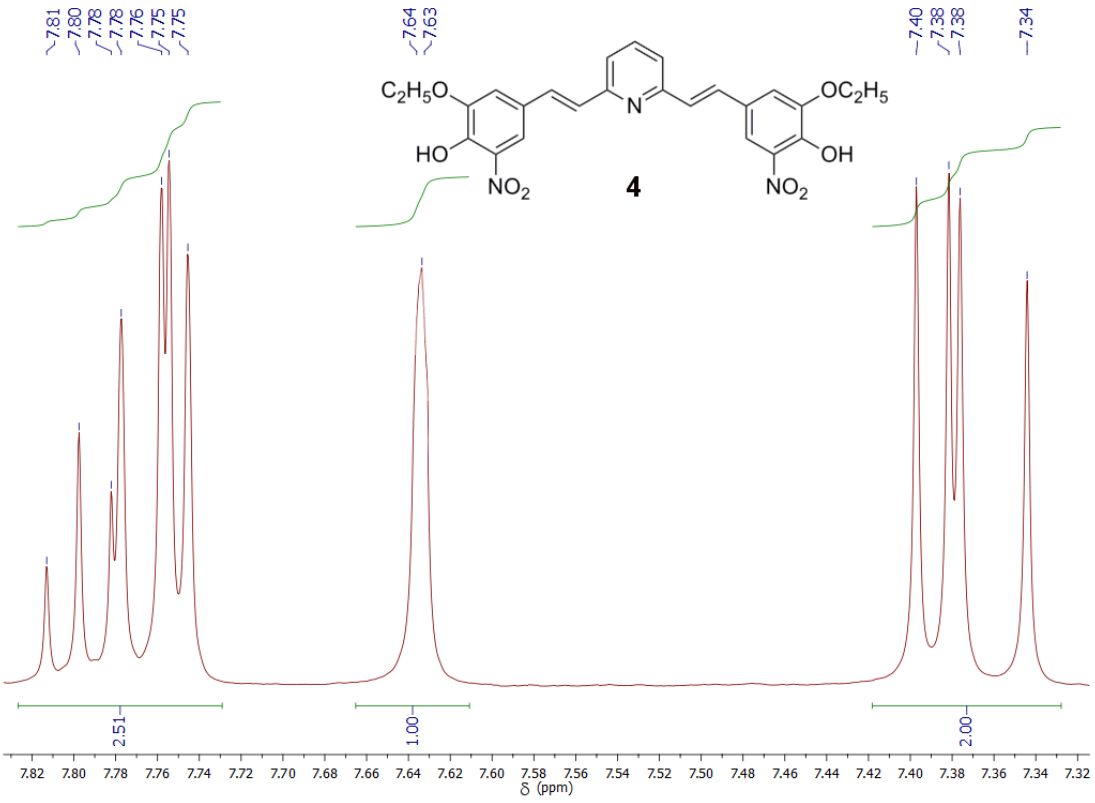
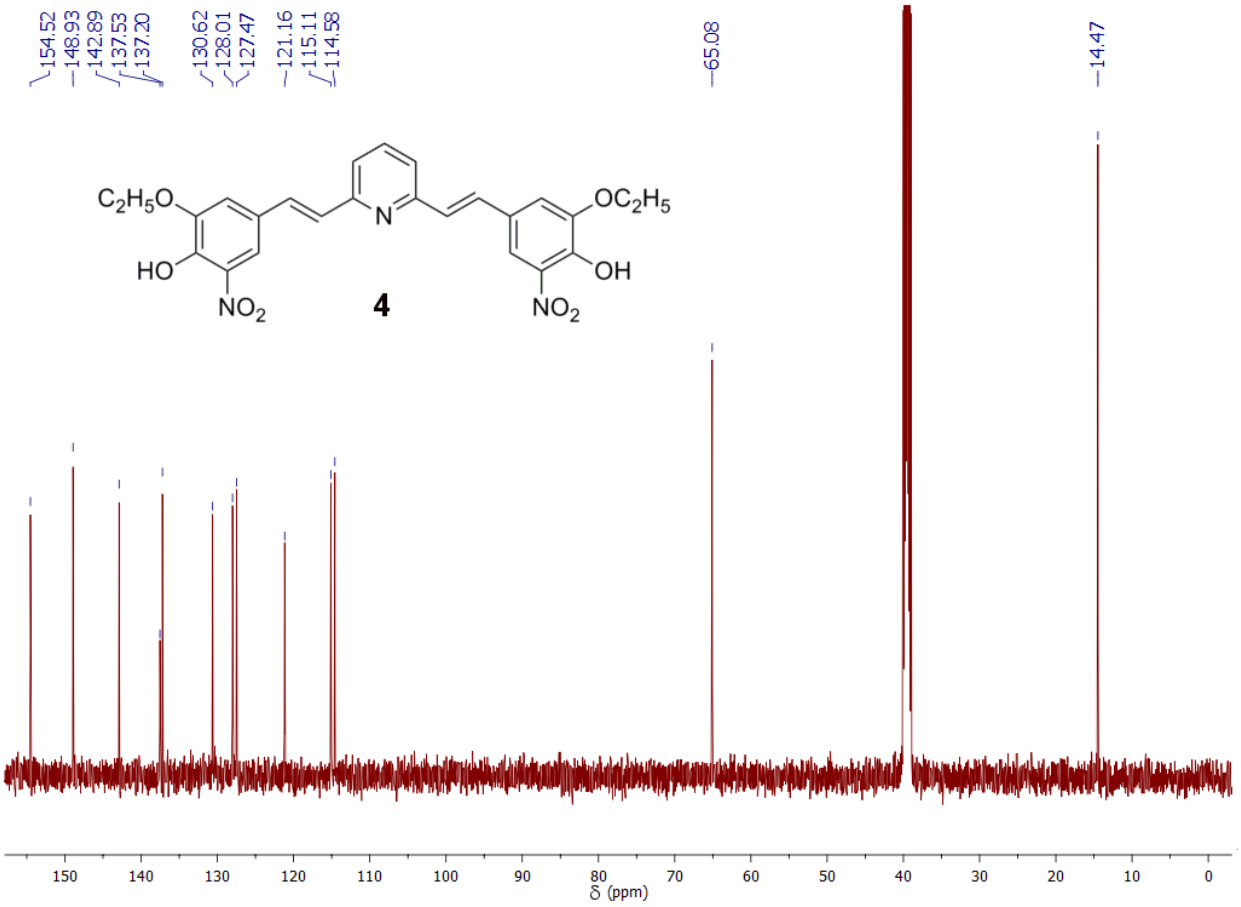
1.37
1.35
C_2H_5O
HO
N
OC_2H_5
OH
3
1.00
1.52
1.04
1.01
2.02
0.99
2.06
3.08
δ (ppm)

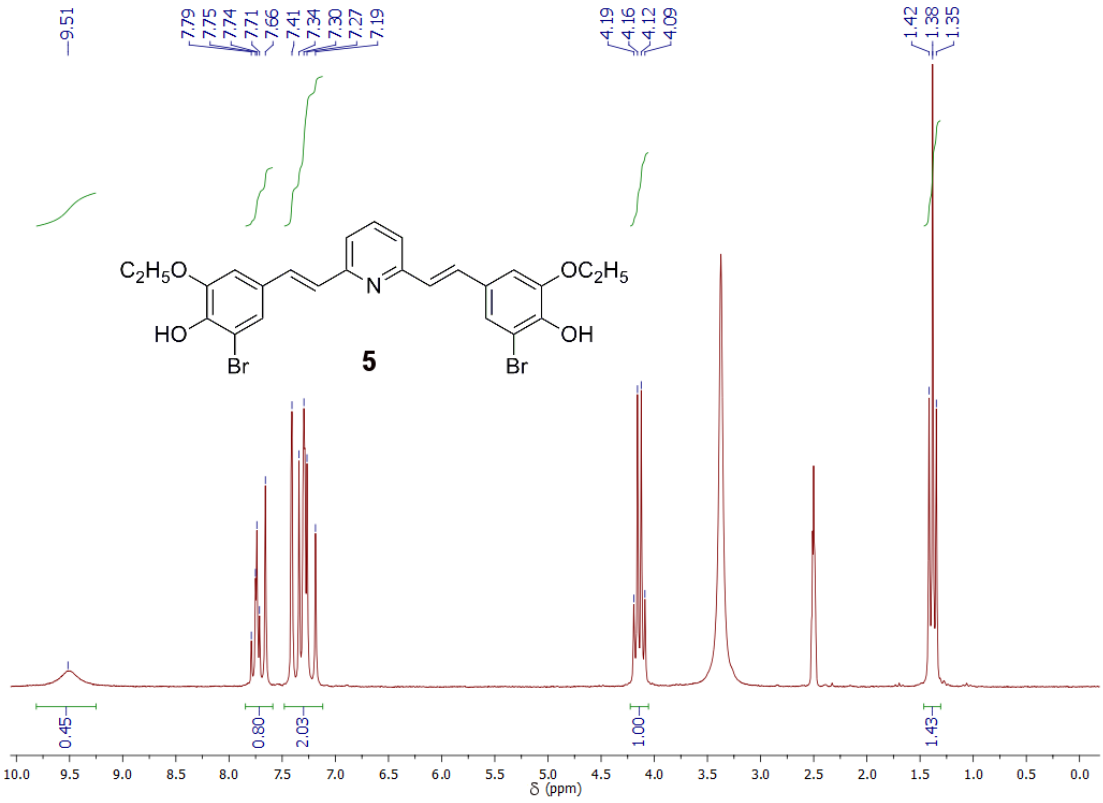

7.73
7.71
7.70
7.68
7.65
7.33
7.31
7.26
7.15
7.11
7.10
7.09
6.83
6.81
C_2H_5O
HO
N
OC_2H_5
OH
3
1.52
1.04
1.01
2.02
0.99
δ (ppm)

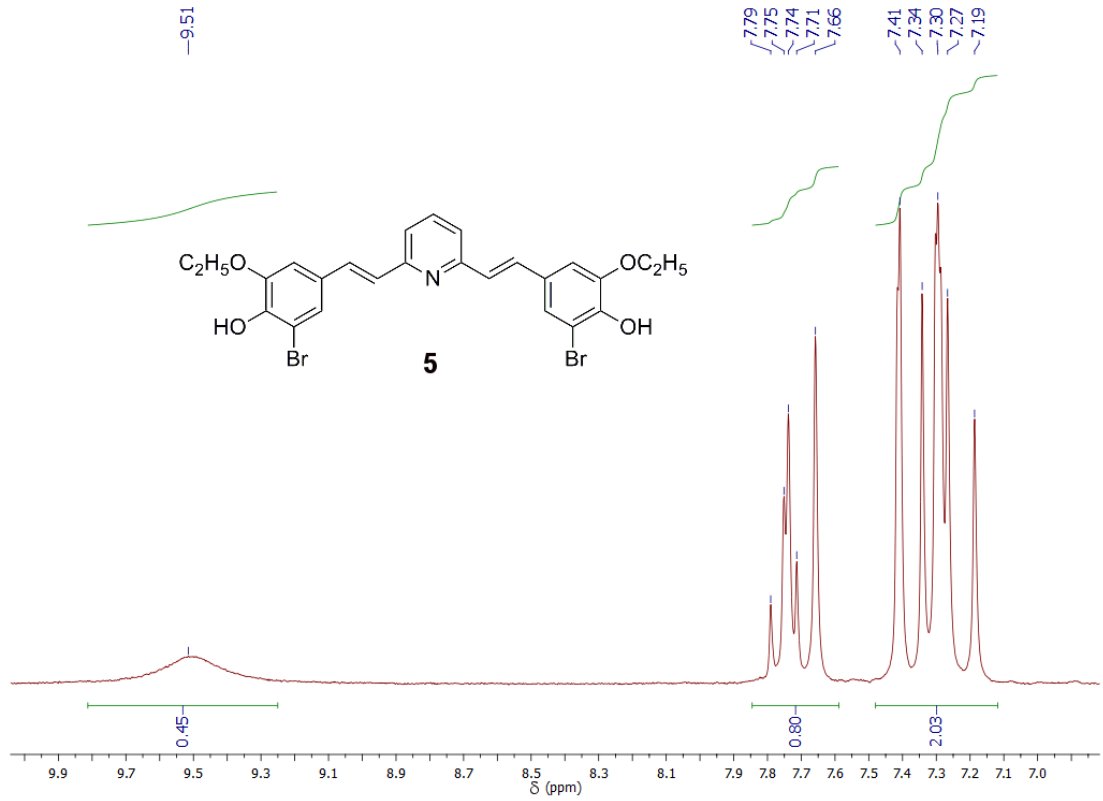

155.13
147.60
146.99
137.16
132.55
128.03
125.14
120.87
119.89
115.68
111.69
63.83
14.77
C2H5O
HO
N
OC2H5
OH
3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

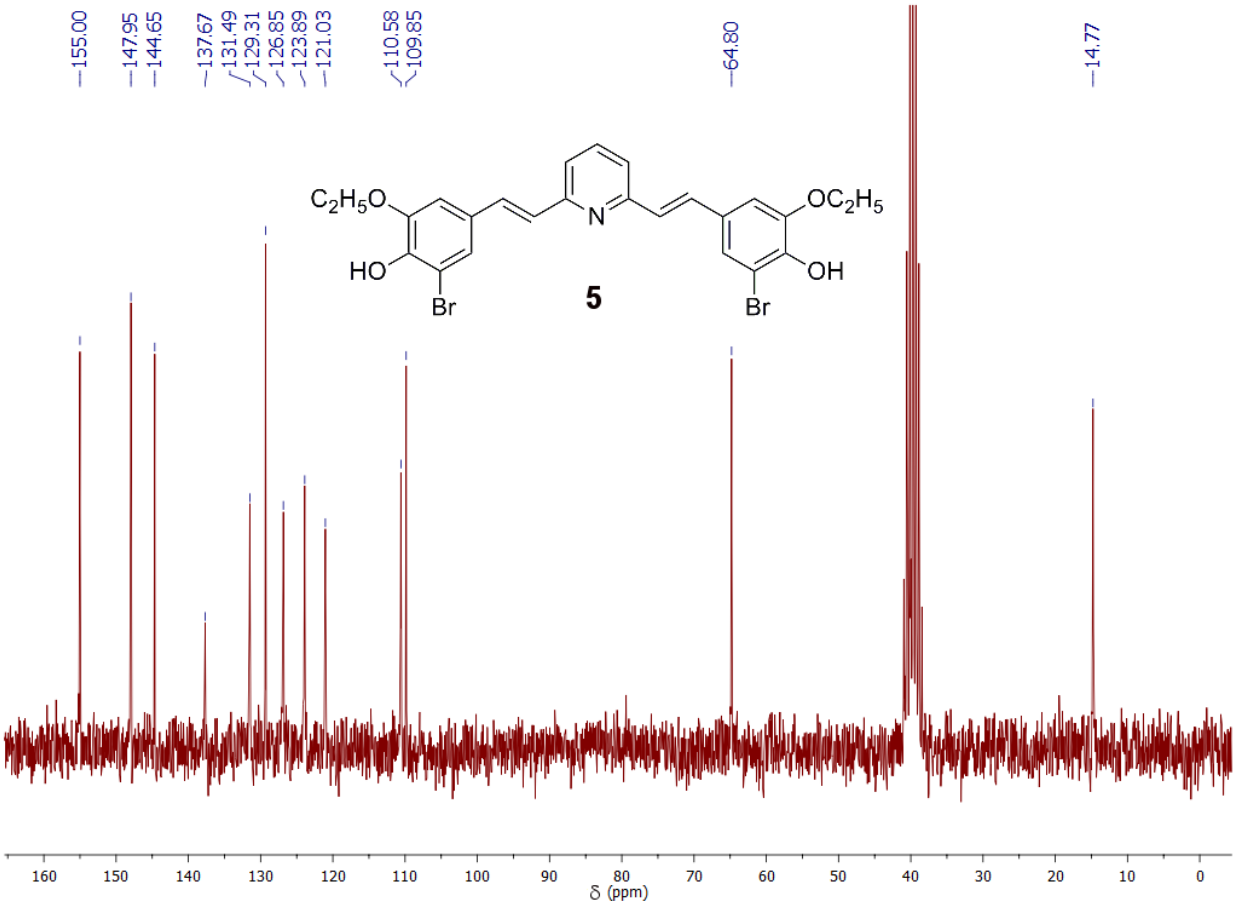

10.47
7.81
7.80
7.78
7.78
7.76
7.75
7.75
7.64
7.63
7.40
7.38
7.38
7.34
4.26
4.25
4.24
4.22
1.44
1.42
1.41
C_2H_5O
HO
NO_2
N
OC_2H_5
OH
NO_2
4
0.87
2.51
1.00
2.00
2.04
3.01
δ (ppm)

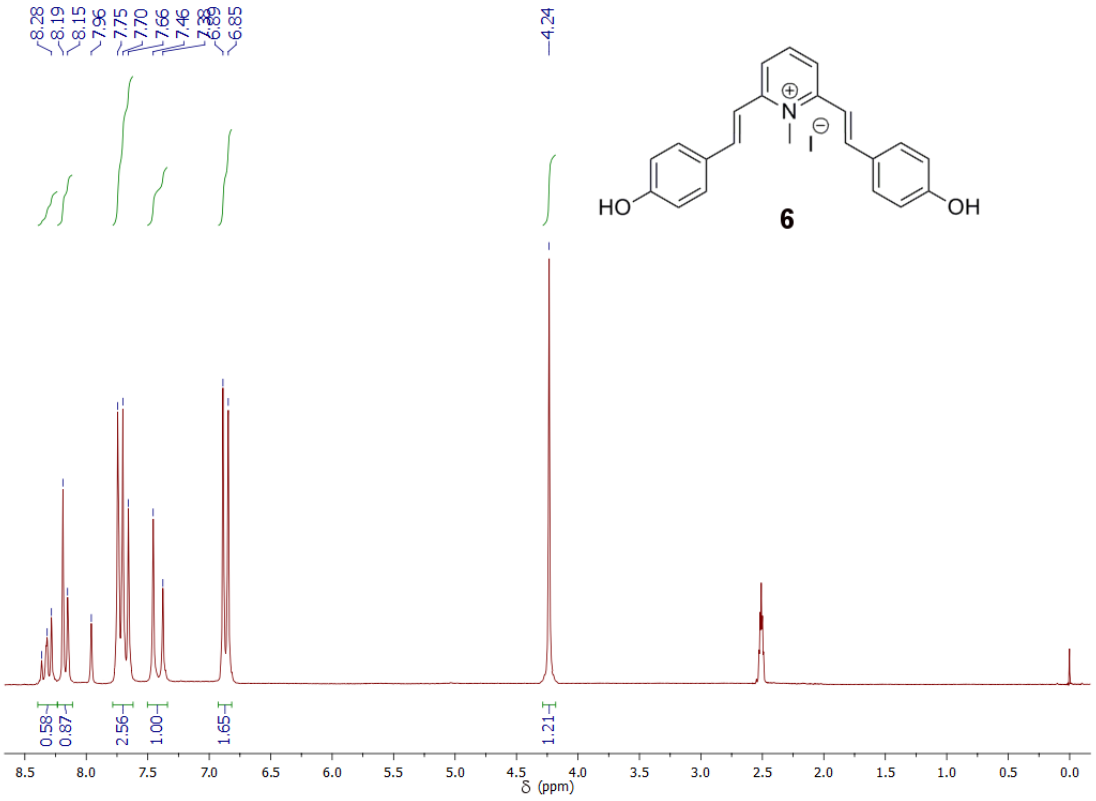

7.81
7.80
7.78
7.78
7.76
7.75
7.75
7.64
7.63
7.40
7.38
7.38
7.34
C_2H_5O
HO
NO_2
N
OC_2H_5
OH
NO_2
4
2.51
1.00
2.00
δ (ppm)

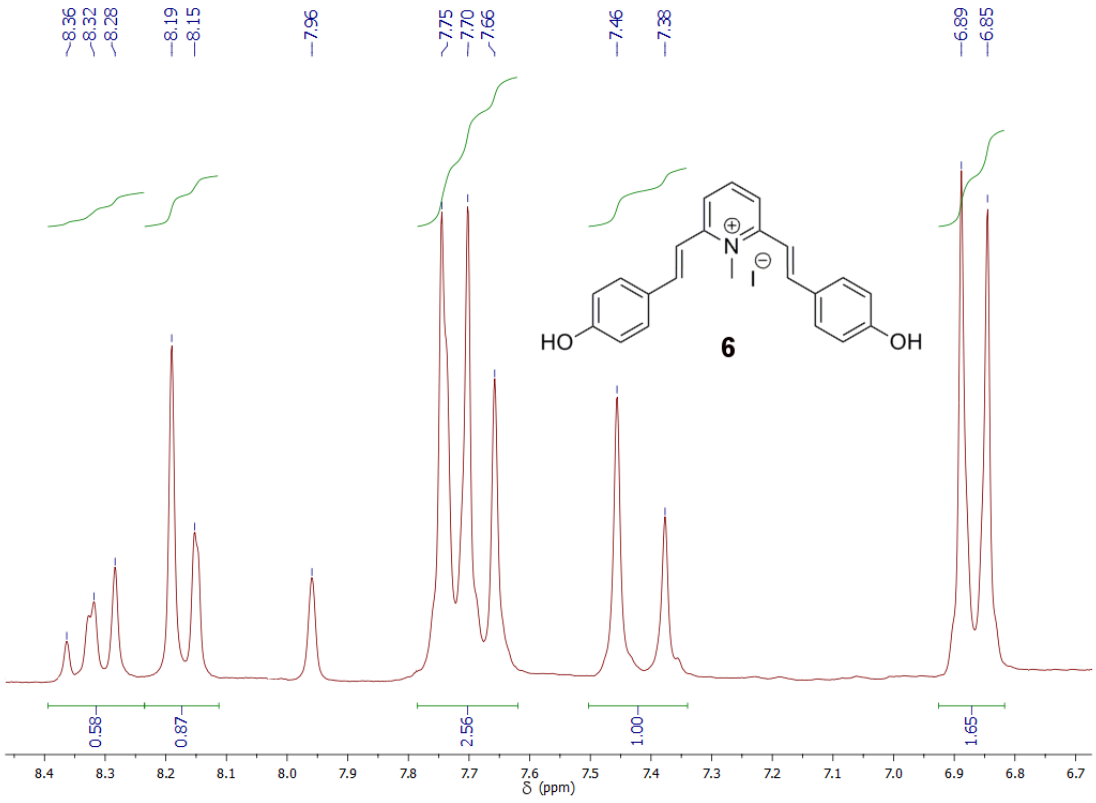

154.52
148.93
142.89
137.53
137.20
130.62
128.01
127.47
121.16
115.11
114.58
65.08
14.47
C_2H_5O
HO
NO_2
N
OC_2H_5
OH
NO_2
4
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

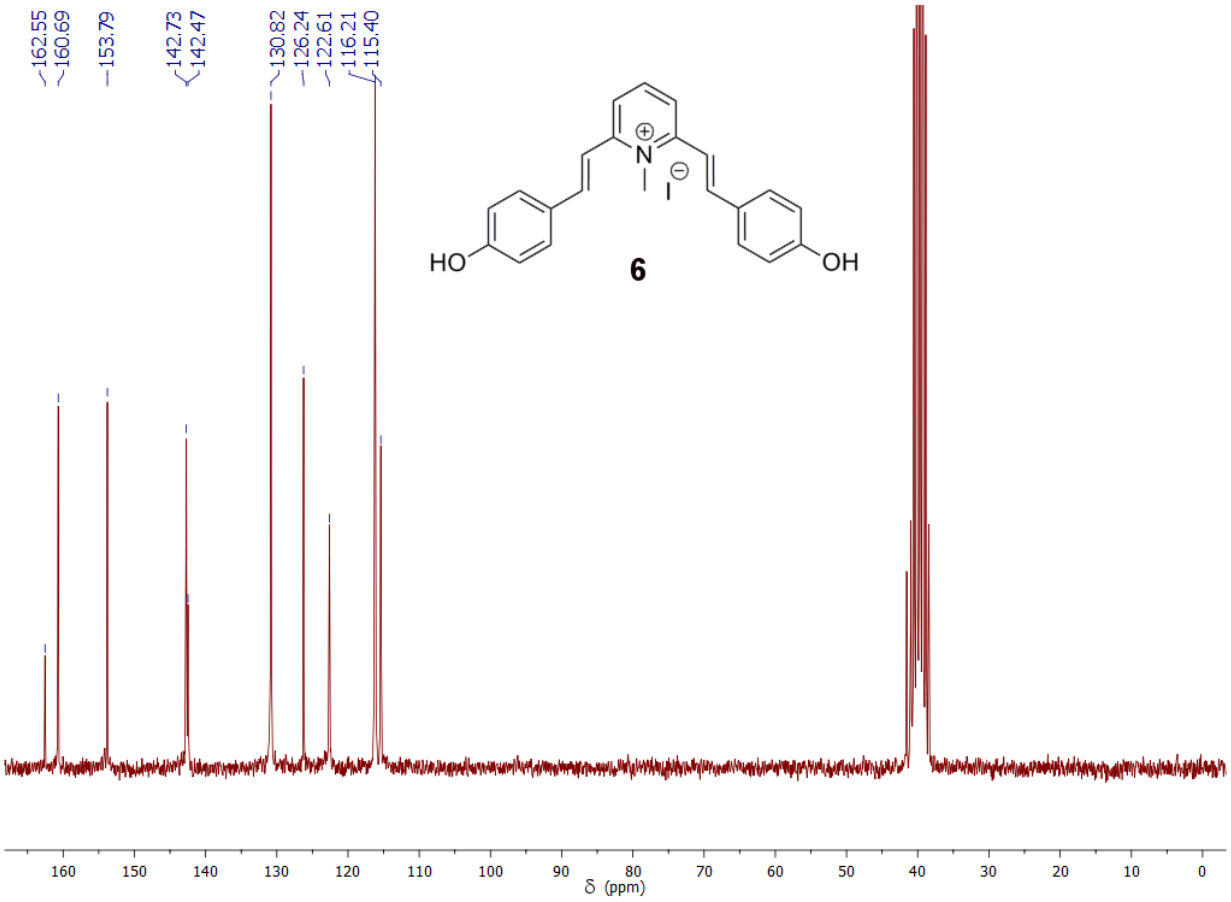

9.51
7.79
7.75
7.74
7.71
7.66
7.41
7.34
7.30
7.27
7.19
4.19
4.16
4.12
4.09
1.42
1.38
1.35
C_2H_5O
HO
Br
N
OC_2H_5
OH
Br
5
0.45
0.80
2.03
1.00
1.43
δ (ppm)

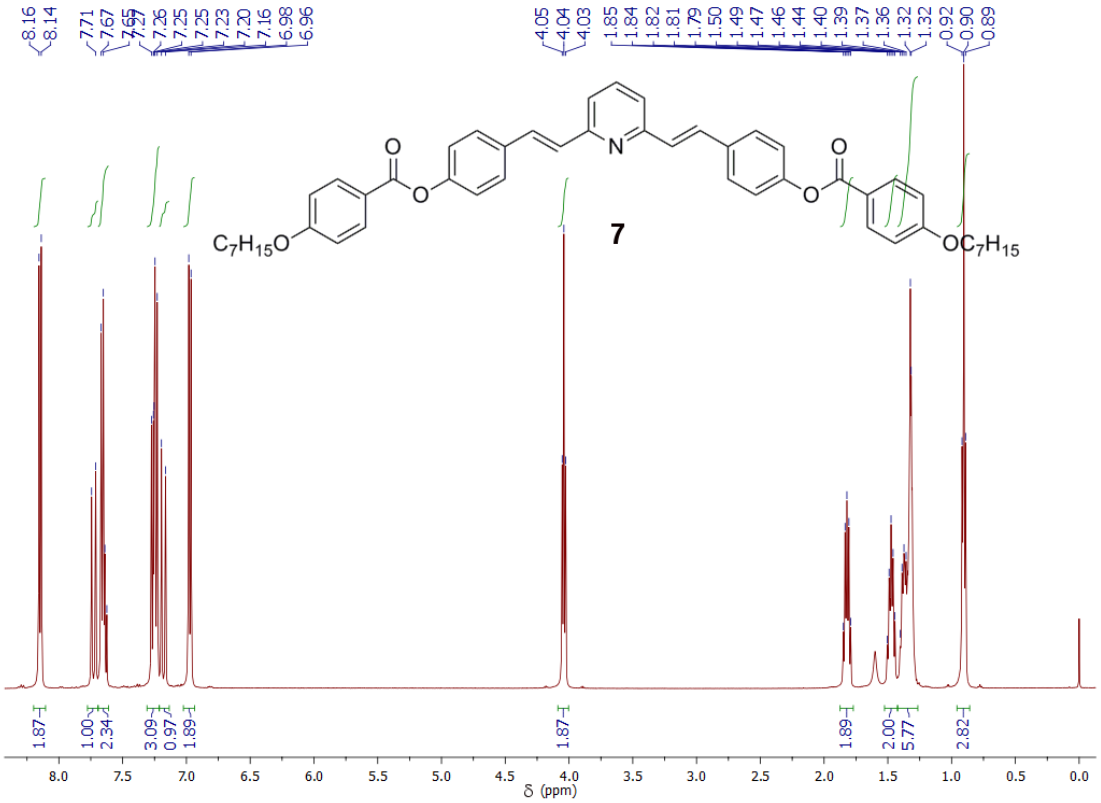

9.51
7.79
7.75
7.74
7.71
7.66
7.41
7.34
7.30
7.27
7.19
C_2H_5O
HO
Br
N
OC_2H_5
OH
Br
5
0.45
0.80
2.03
δ (ppm)

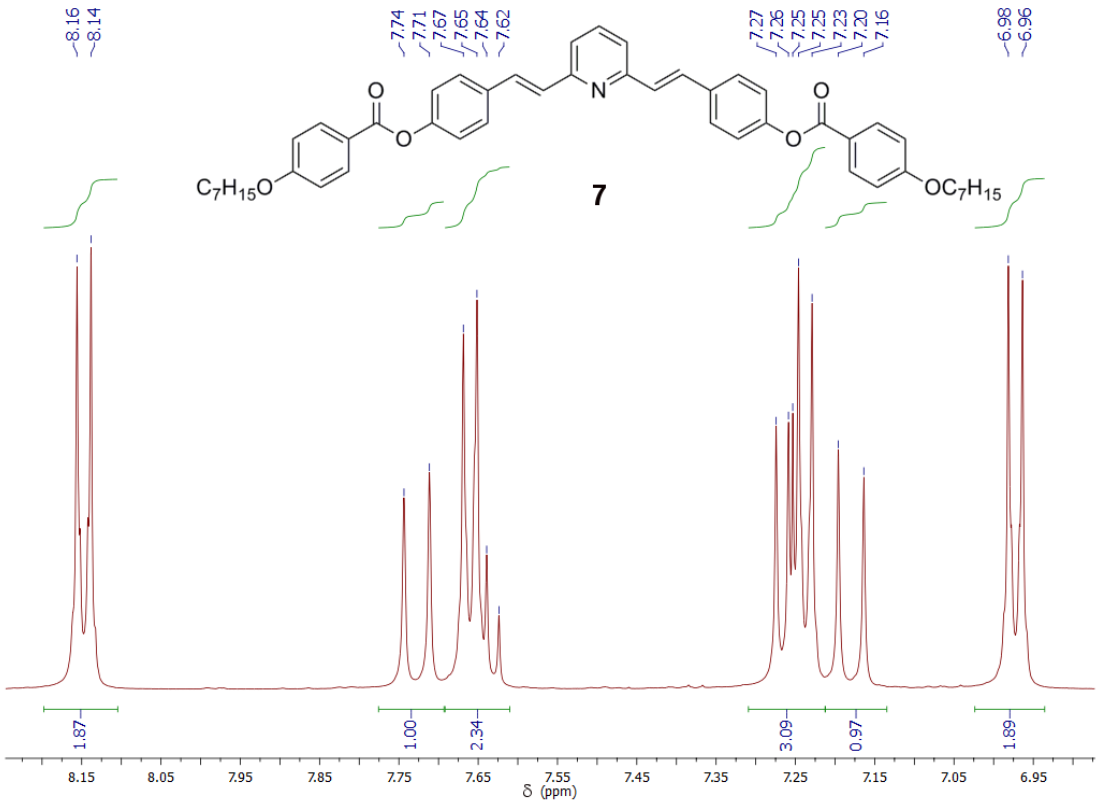

155.00
147.95
144.65
137.67
131.49
129.31
126.85
123.89
121.03
110.58
109.85
64.80
14.77
C2H5O
OC2H5
HO
OH
N
Br
Br
5
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

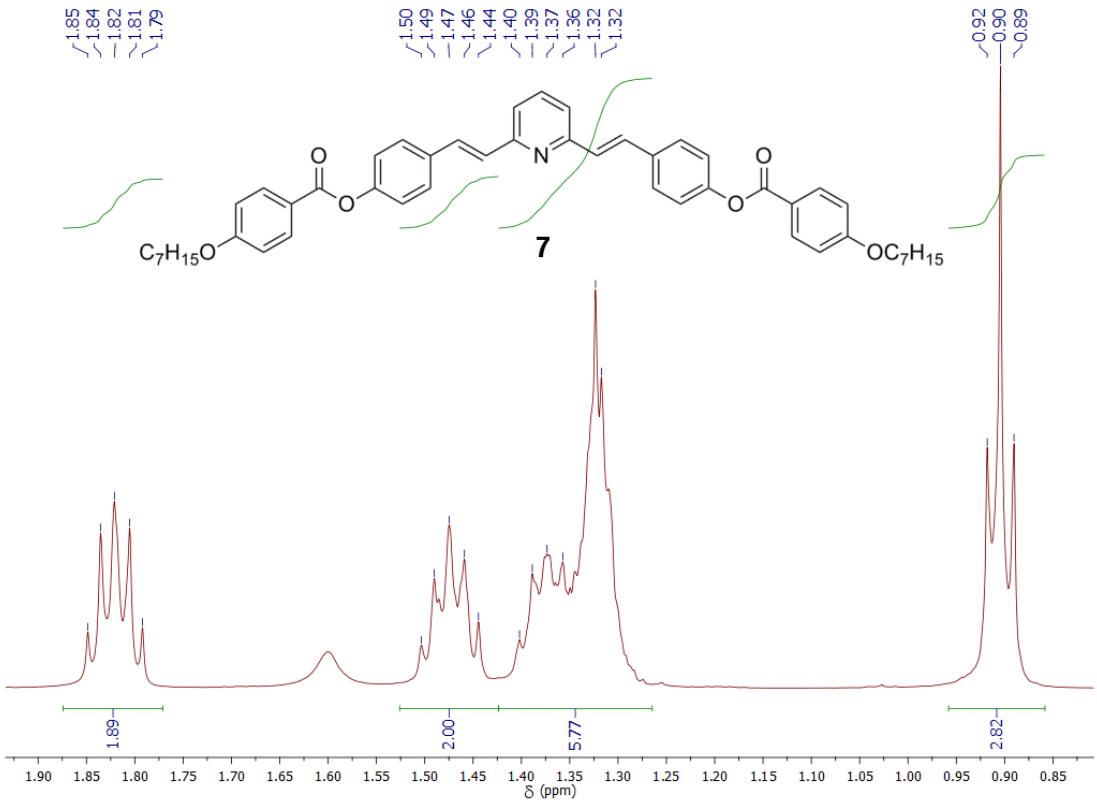

8.28
8.19
8.15
7.96
7.75
7.70
7.66
7.46
7.38
6.89
6.85
4.24
HO
OH
6
0.58
0.87
2.56
1.00
1.65
1.21
δ (ppm)

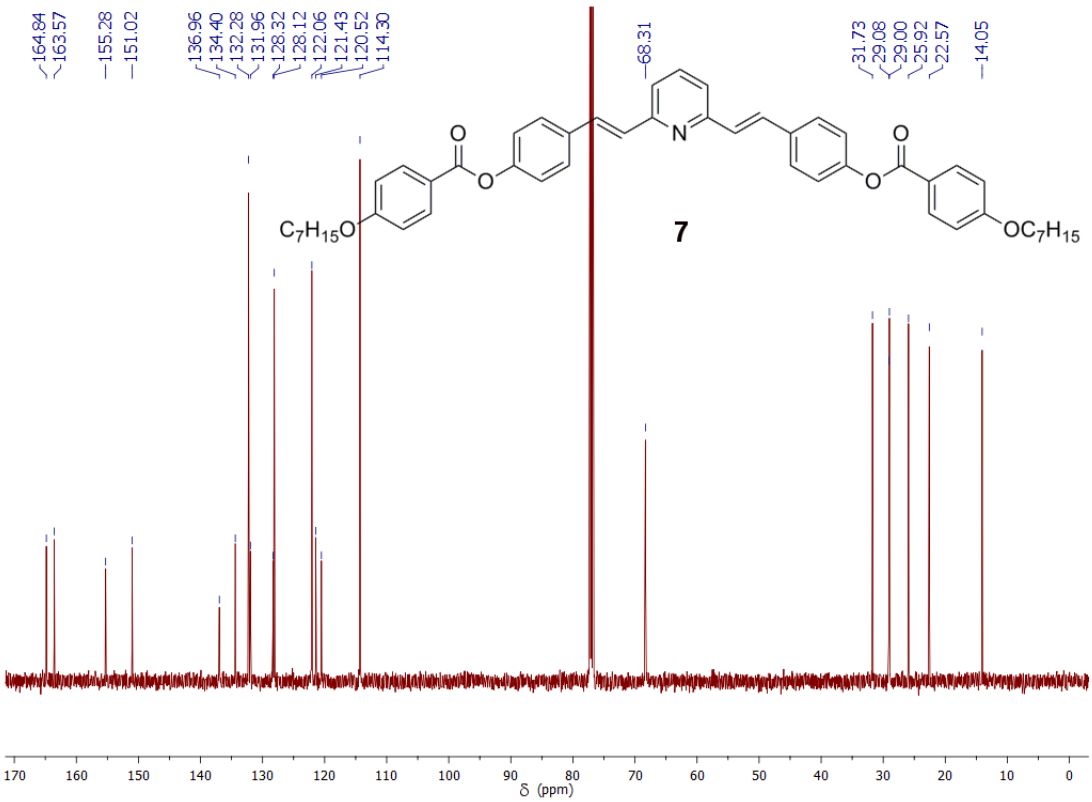

8.36
8.32
8.28
8.19
8.15
7.96
7.75
7.70
7.66
7.46
7.38
6.89
6.85
HO
OH
6
0.58
0.87
2.56
1.00
1.65
δ (ppm)


162.55
160.69
153.79
142.73
142.47
130.82
126.24
122.61
116.21
115.40
HO
OH
N
I
6
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)


8.16
8.14
7.71
7.67
7.27
7.26
7.25
7.25
7.23
7.20
7.16
6.98
6.96
4.05
4.04
4.03
1.85
1.84
1.82
1.81
1.79
1.50
1.49
1.47
1.46
1.44
1.40
1.39
1.37
1.36
1.32
1.32
0.92
0.90
0.89
O
O
N
O
O
C7H15O
OC7H15
7
1.87
1.00
2.34
3.09
0.97
1.89
1.87
1.89
2.00
5.77
2.82
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ (ppm)


8.16
8.14
7.74
7.71
7.67
7.65
7.64
7.62
7.27
7.26
7.25
7.25
7.23
7.20
7.16
6.98
6.96
O
O
N
O
O
C7H15O
OC7H15
7
1.87
1.00
2.34
3.09
0.97
1.89
8.15
8.05
7.95
7.85
7.75
7.65
7.55
7.45
7.35
7.25
7.15
7.05
6.95
δ (ppm)


1.85
1.84
1.82
1.81
1.79
1.50
1.49
1.47
1.46
1.44
1.40
1.39
1.37
1.36
1.32
1.32
0.92
0.90
0.89
C7H15O
O
N
OC7H15
7
1.89
2.00
5.77
2.82
δ (ppm)


164.84
163.57
155.28
151.02
136.96
134.40
132.28
131.96
128.32
128.12
122.06
121.43
120.52
114.30
68.31
31.73
29.08
29.00
25.92
22.57
14.05
C7H15O
O
N
OC7H15
7
δ (ppm)

References

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[S2] E.D. Bergmann, S. Pinchas, J. Org. Chem. 15 (1950) 1184–1190.

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