ELECTRONIC SUPPLEMENTARY MATERIAL (ESI)

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Supramolecular Layers and Versatile Packing Modes: the Solid State Behavior of *ortho, ortho*-linked Bisphenols

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Table S1 Reaction conditions for the unsuccessful fission of bisacetal **4**

attempt	catalyst	time	temperature	product
1	HCl (10 %)	5 h	reflux	starting material
2	HCl (10 %)[1]	17.5 h	reflux	starting material
3	HCl (15 %)[2]	7 h	reflux	starting material
4	HCl (37 %)[2]	7 h	reflux	starting material
5	HBr (48 %)[2]	6 h	reflux	starting material
6	HBr (48 %)	16 h	reflux	decomposition
7	H_2SO_4 (10 %)	7 h	reflux	starting material
8	H_2SO_4 (45 %)	24 h	50 °C	decomposition
9	H_2SO_4 (96 %)	12 h	50 °C	decomposition
10	AcOH, Ac_2O, H_2SO_4[3]	2 h	RT	starting material
11	TFA (99 %)	7 d	RT	starting material

[1] R. H. Schlessinger, J. L. Wood, J. Org. Chem. 51 (1986) 2621.
[2] J. de Mendoza, P. M. Nieto, P. Prados, C. Sanchez, Tetrahedron 46 (1990) 671.
[3] R. H. Rich, P. A. Bartlett, J. Org. Chem. 61 (1996) 3916.

Table S2 Distances and angles of hydrogen bond type and other interactions

atoms	symmetry	distances (Å)		angle (°)
		D···A	H···A	D–H···A
1a				
Intramolecular contacts				
C(12)-H(12A)···O(3)	*x*, *y*, *z*	3.114(3)	2.57	116
C(18)-H(18)···O(12)	*x*, *y*, *z*	3.080(3)	2.56	115
Contacts within the host layer				
C(9)-H(9A)···O(11)	1/2-*x*, -1/2+*y*, 1/2-*z*	3.186(3)	2.50	129
C(15)-H(15)···O(1)	3/2-*x*, 1/2+*y*, 1/2-*z*	3.263(3)	2.54	133
Contacts within the solvent layer				
C(2I)-H(2I)···O(1I)	1/2-*x*, 1/2+*y*, 1/2-*z*	3.346(4)	2.47	149
C(1K)-H(1K1)···O(1K)	3/2-*x*, 1/2+*y*, 1/2-*z*	3.267(4)	2.50	135
C(2G)-H(2G1)···O(1G)	*x*, -1+*y*, *z*	3.364(4)	2.53	143
C(1H)-H(1H1)···O(1H)	*x*, -1+*y*, *z*	3.392(4)	2.56	143
Contacts between layers				
O(3)-H(3)···O(1K)	3/2-*x*, 1/2+*y*, 1/2-*z*	2.590(3)	1.76	171
O(12)-H(12)···O(1I)	1/2-*x*, -1/2+*y*, 1/2-*z*	2.579(3)	1.76	163
O(9)-H(9)···O(1H)	*x* , *y*, *z*	2.642(3)	1.80	177
O(6)-H(6)···O(1G)	1/2+*x*, 3/2-*y*, -1/2+*z*	2.646(3)	1.81	179
C(2)-H(2)···O(1K)	3/2-*x*, 1/2+*y*, 1/2-*z*	3.250(4)	2.59	127
C(9)-H(9A)···O(1G)	1/2+*x*, 3/2-*y*, -1/2+*z*	3.250(3)	2.57	129
C(15)-H(15)···O(1H)	*x* , *y*, *z*	3.289(4)	2.62	128
C(1H)-H(1H2)···O(2)	-1/2+*x*, 1/2-*y*, -1/2+*z*	3.309(4)	2.47	143
C(1G)-H(1G3)···O(1G)	*x*, -1+*y*, *z*	3.484(4)	2.65	143
C(1G)-H(1G1)···O(11)	*x*, 2-*y*, 1-*z*	3.560(4)	2.67	151
C(1G)-H(1G2)···O(10)	*x*, *y*, 1+*z*	3.406(4)	2.60	139
C(2G)-H(2G2)···O(10)	*x*, *y*, 1+*z*	3.316(4)	2.49	142
C(1I)-H(1I1)···O(5)	*x*, *y*, *z*	3.435(4)	2.59	144
C(1I)-H(1I2)···O(8)	*x*, 1+*y*, *z*	3.398(4)	2.59	140
C(2K)-H(2K1)···O(7)	*x*, 1+*y*, *z*	3.522(4)	2.67	146
C(2K)-H(2K2)···O(4)	*x*, *y*, *z*	3.471(4)	2.65	141
2a				
Intramolecular contacts				
O(3)-H(3)···Br(1)	*x*, *y*, *z*	3.158(2)	2.64	123
O(6)-H(6)···Br(2)	*x*, *y*, *z*	3.158(2)	2.63	123
C(5)-H(5)···O(6)	*x*, *y*, *z*	3.147(3)	2.65	114
Contacts within the host layer				
C(7)-H(7B)···centroid C(11)-C(12)	1-*x*, 2-*y*, 2-*z*	3.567(2)	2.88	128
Br(1)···O(4)	*x*, -1+*y*, *z*	3.367(2)		
Br(2)···O(1)	*x*, 1+*y*, *z*	3.309(2)		
Contacts within the solvent layer				
C(1G)-H(1G1)···O(1G)	1-*x*, -*y*, 1-*z*	3.388(4)	2.46	162
C(2G)-H(2G1)···O(1H)	*x*, *y*, *z*	3.583(4)	2.66	161
C(1H)-H(1H1)···S(1G)	-*x*, 1-*y*, 1-*z*	3.735(3)	2.90	146
C(1H)-H(1H3)···O(1H)	-*x*, 1-*y*, 1-*z*	3.407(4)	2.49	160
Contacts between layers				
O(3)-H(3)···O(1H)	*x*, *y*, 1+*z*	2.627(3)	1.91	145
O(6)-H(6)···O(1G)	*x*, 1+*y*, *z*	2.596(3)	1.91	141
C(1G)-H(1G3)···O(2)	*x*, -1+*y*, *z*	3.364(4)	2.71	126
C(1G)-H(1G3)···O(4)	-*x*, 1-*y*, 2-*z*	3.412(3)	2.55	150
C(1G)-H(1G2)···O(5)	*x*, -1+*y*, -1+*z*	3.400(4)	2.60	141
C(2G)-H(2G2)···O(5)	*x*, -1+*y*, -1+*z*	3.484(4)	2.66	145
C(1H)-H(1H2)···O(2)	*x*, *y*, *z*	3.476(4)	2.62	148
C(2H)-H(2H1)···O(1)	1-*x*, 1-*y*, 1-*z*	3.385(4)	2.47	159
Br(1)···O(1H)	*x*, *y*, -1+*z*	3.303(2)		

Table S2 Continued

atoms	symmetry	distances (Å)		angle (°)
		D···A	H···A	D–H···A
4				
C(7)-H(7A)···O(7)	1/2-*x*, -1/2+*y*, 3/2-*z*	3.147(1)	2.43	128
C(14)-H(14A)···O(8)	- *x*, 2-*y*, 2-*z*	3.424(1)	2.54	149
C(14)-H(14B)···O(5)	1-*x*, 2-*y*, 2-*z*	3.074(1)	2.43	122
C(15)-H(15B)···O(1)	1+*x*, *y*, *z*	3.444(1)	2.47	169
C(16)-H(16B)···O(2)	1/2+*x*, 3/2-*y*, 1/2+*z*	3.360(2)	2.47	147
5				
C(7)-H(7B)···O(4)	-*x*, 2-*y*, -*z*	3.260(2)	2.54	130
C(14)-H(14C)···O(4)	1-*x*, 2-*y*, -*z*	3.439(2)	2.51	158
C(15)-H(15B)···O(3)	*x*, -1+*y*, *z*	3.247(2)	2.44	139
C(14)-H(14A)···centroid (B)	1+*x*, *y*, *z*	3.668(2)	2.92	134
C(15)-H(15A)···centroid (B)	-*x*, 1-*y*, 1-*z*	3.607(2)	2.78	143
6				
O(1)-H(1)···O(2)	¾-*x*, ¼+*y*, -¼+ *z*	2.695(1)	1.89	161
C(9)-H(9A)···O(1)	¾-*x*, -¼ +*y*, -¾ +*z*	3.465(2)	2.50	169
7				
O(1)-H(1)···O(2)	*x*, *y*, *z*	2.618(2)	1.89	147
O(5)-H(5)···O(6)	*x*, *y*, *z*	2.627(2)	1.90	147
O(5)-H(5)···O(5)	*x*, 1-*y*, -*z*	2.766(2)	2.42	106
C(3)-H(3)···O(4)	*x*, *y*, *z*	2.749(3)	2.43	100
C(5)-H(5A)···O(6)	*x*, 1-*y*, -*z*	3.350(3)	2.44	167
C(10)-H(10)···O(2)	1-*x*, 1/2+*y*, 1/2-*z*	3.432(3)	2.55	159
C(12)-H(12)···O(8)	*x*, *y*, *z*	2.725(3)	2.39	101
C(14)-H(14)···O(7)	1-*x*, -1/2+*y*, 1/2-*z*	3.437(3)	2.54	163
C(16)-H(16B)···O(8)	1+*x*, 1/2-*y*, -1/2+*z*	3.382(3)	2.55	145
8				
O(1)-H(1A)···O(1)	-*x*, 1-*y*, 2-*z*	2.695(2)	1.92(5)	176(6)
O(1)-H(1B)···O(2)	*x*, *y*, *z*	2.776(3)	2.02(7)	157(5)
O(2)-H(2B)···O(2)	-*x*, 1-*y*, 1-*z*	2.798(2)	1.98(5)	172(4)
O(2)-H(2A)···O(1)	*x*, *y*, *z*	2.776(3)	1.92(7)	156(5)
C(4)-H(4)···centroid (B)	*x*, 1/2-*y*, 1/2+*z*	3.564(2)	2.68	156
C(7)-H(7A)···centroid C(9)-C(10)	1-*x*, 1-*y*, 1-*z*	3.795(2)	2.95	144
C(7)-H(7B)···centroid C(1)-C(2)	1+*x*, *y*, *z*	3.582(2)	2.63	161
C(10)-H(10)···centroid (A)	*x*, *y*, -1+*z*	3.548(2)	2.69	151

[a] Centroid means the centre of gravity of the respective aromatic ring, centroid (A) = C(1)-C(2)-C(3)-C(4)-C(5)-C(6), centroid (B) = C(8)-C(9)-C(10)-C(11)-C(12)-C(13)