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Crystalline Diol Hosts Featuring a Bulky Biphenyl Framework – Host Synthesis and Formation of Inclusion Compounds

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Abstract. Biphenyls having two hydroxy containing benzo condensed oligocyclic substituents in positions 2,2' were synthesized to yield the crowded diols **5** – **10**. These compounds proved successful clathrate hosts. Crystalline inclusion formation is reported and discussed with reference to structural parameters of the host molecules covering the bis-fluorenol analogous host compounds **1–4**.

1 Introduction

Inclusion compounds [1] and molecular recognition [2] have emerged as important and challenging frontiers in chemistry [3] owing to potential uses such as chiral separation, nonlinear optics or sensing [4]. Clathrates are particular compounds of this sort typical of a co-crystalline structure coming from the crystal lattice association of host and guest components [5]. Molecules which contain the fluorene moiety have proved to be successful hosts capable of forming clathrates with a variety of guests [6]. These hosts are often functionalized by a hydroxy group at position 9 of the fluorene unit [7], and are sometimes dimerized *via* an aromatic moiety such as biphenyl [4], to yield a crowded diol host molecule of which 2,2'-bis(9-hydroxy-9-fluorenyl)biphenyl (**1**) is the parent compound [8]. Substituting the fluorene moieties at the 2- and 7-positions with spacer groups including *t*-butyl and halogens (**2–4**), also yields successful host compounds which are often stabilized by hydrogen bonds between host and guest in the clathrates formed [9]. These compounds were defined as 'coordinato-clathrates' [10]. Moreover, the addition of chloro and bromo substituents has the added potential for a number of electrostatic interactions [11].

Previously, we have elucidated a dozen of clathrate structures of these host compounds (**1–4**) involving diethyl ether, 1,3-dioxolane, 1,4-dioxane, di-*n*-propylamine, acetonitrile, butyronitrile, dimethylformamide, cyclopentanol, cyclohexanone and (–)-fenchone as the guest [8a, 9, 12]. We have also found that these hosts not only accommodate selected guests from solution but also from the vapour phase to offer chemical sensor developments [13]. Another observation is the atropisomeric behaviour of host molecule **1** thus being a promising structure for enantio discrimination of organic compounds [8]. To this end, a challenge appears for making more fundamental structural modifications at the fluorenyl groups of **1** than lateral substitution. This is met with the modified structures **5–10** that have dibenzosuberyl (**5**), dibenzosuberenyl (**6**), tribenzocycloheptatrienyl (**7**), epoxydihydro-tribenzocycloheptatrienyl (**8**), xanthenyl (**9**) or thioxanthenyl (**10**) characteristic groups flanking the biphenyl centre.

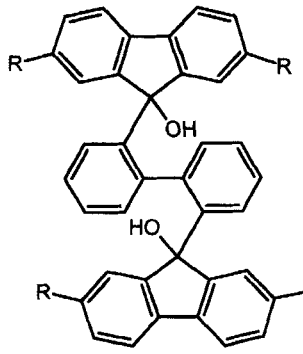
Here we report the synthesis of compounds **5–10** and present the extensive results of crystalline inclusion formation covering host molecules **1–10**.

2 Synthesis

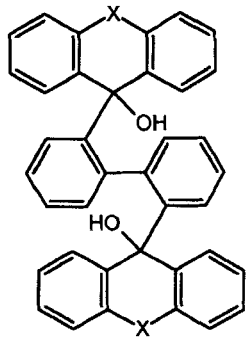
Compounds **5–10** were synthesized [14] in 15–73% yield by lithiation (*n*-BuLi/Et_2O) of 2,2'-dibromobiphenyl (**11**) and subsequent reaction with the respective ketone **12–17**. The starting ketones were prepared according to literature procedures [7a]. Inclusion compounds were obtained by recrystallization of host compounds from the respective guest solvent.

3 Inclusion Properties

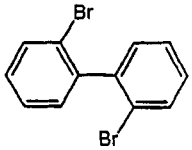
The basic molecule **1** has the qualification required of a good host compound [6, 15]. It is bulky, stiff and con-



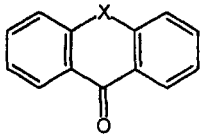
1 R = H
2 R = C(CH$_3$)$_3$
3 R = Cl
4 R = Br



5 X = CH_2CH_2
6 X = CH=CH
7 X = 
8 X = 
9 X = O
10 X = S



11



12 X = CH_2CH_2
13 X = CH=CH
14 X = 
15 X = 
16 X = O
17 X = S

tains the hydroxyl groups which act as hydrogen-bond donors in host guest interaction. Logically host compound **1** forms a great number of crystalline inclusions with a variety of solvents (38 different species; Table 1). These include amines and alcohols of different sizes and degrees of ramification, dipolar aprotic compounds of different polarities, heterocycles of different ring sizes and with different numbers and types of hetero atoms unlike aromatic hydrocarbons which are less efficient as guests. Compared to **1** the *t*-butyl-substituted host molecule **2** has rather similar inclusion behaviour, although there are some differences (Tabel 1). Compounds **3** (chloro derivative) and **4** (bromo derivative) are too insoluble under the given conditions, therefore they are not found in Table 1. However, crystal structures of respective inclusion compounds [9, 12c] show their ability to act as hosts although they are not very useful for co-crystallization.

A different situation is found for dibenzosuberyl analogous host molecule **5** and even more pronounced for the dibenzosuberenyl, tribenzocycloheptatrienyl and epoxydihydro-tribenzocycloheptatrienyl derivatives **6**–**8** which show moderate to poor host properties. In particular compound **6** yields three crystalline inclusions from the solvents given in Table 1. Most distinctly, alcohols are being excluded from inclusion formation here, except for 1-propanol and cyclopentanol in the case of compound **5**.

The change in the linkage unit X (*cf.* formula) from carbon bridging elements (**5**–**8**) to hetero atoms (O, S) restores inclusion ability to an extent almost comparable to **1** and **2**. This is evident in Table 1 for compounds **9** and **10.** Nevertheless, the alcohols are still rather unsuitable guests compared with **1** and **2**. To a smaller degree this is also true for the dipolar aprotic solvents, while aromatic hydrocarbons fail completely to act as guest molecules.

A wide range of stoichiometric host:guest ratios was found for the different inclusion compounds including 2:1, 3:2, 1:1, 2:3, 1:2, 1:3 and 1:4 (Table 1). None of the hosts exhibit one single stoichiometric ratio regardless of the guest. But there is a pattern of preference emerging from these data: most of the inclusion compounds of **1** have 1:2 host:guest ratio followed by 1:1 stoichiometric inclusions, while host compound **2** prefers 1:1 ratio. This is perhaps a result of the bulky *t*-butyl groups effectively filling lattice space not available for a second guest molecule. Hence compound **1**, according to its number of functional groups, mainly behaves as a bivalent host, whereas for host compound **2** monovalency is more likely. In a sense, this property is also seen for the xanthenyl and the thioxanthenyl derivatives **9** and **10**. Compound **10** is clearly in favour of the 1:1 host:guest stoichiometric ratio in its complexes, whereas **9** shows no such clear distinction between 1:1 and 1:2 ratio (or monovalent *vs.* bivalent binding behaviour). This latter host also exhibits a comparatively high number of the odd stoichiometric ratios 1:3, 2:3 and 3:2 probably due to the xanthenyl oxygen which is an additional binding site for a potential guest. Other things noticeable in Table 1 are the unusual stoichiometric host:guest ratio of 1:4 in case of the complex **7**·piperidine and that inclusions with DMF and DMSO are formed by all of these hosts.

In conclusion, 2,2'-bis(9-hydroxy-9-fluorenyl)biphenyl and its analogues have proved to be a rich source of crystalline inclusion hosts. They form inclusions with a variety of uncharged organic molecules ranging from protic dipolar to apolar compounds (158 different spe-

Table 1 Crystalline Inclusion Compounds (Host:guest stoichiometric ratios)[a]

Guest solvent	Host compound							
	1	**2**	**5**	**6**	**7**	**8**	**9**	**10**
1-Propylamine	1:1	[b]	1:1	–	1:1	1:2	1:2	–
2-Methyl-1-propylamine	1:2	[b]	2:1	–	1:3	1:3	1:2	1:2
2-Butylamine	1:2	[b]	[b]	–	–	–	1:2	1:2
Cyclohexylamine	1:2	1:3	1:3	[b]	–	–	1:3	–
Diethylamine	1:1	1:1	2:1	[b]	[b]	[b]	1:2	1:1
Di-1-propylamine	1:1	1:1	2:1	–	1:1	1:1	1:2	1:1
Di-1-butylamine	1:1	1:1	[b]	[b]	[b]	[b]	1:1	1:1
Piperidine	1:2	1:2	–	–	1:4	[b]	1:2	1:1
2-Methylpiperidine	1:2	1:1	–	–	[b]	[b]	–	1:1
Morpholine	1:2	1:1	1:2	1:2	[b]	[b]	1:3	1:3
Triethylamine	1:1	1:1	2:1	–	–	–	1:2	1:1
Tri-1-propylamine	1:1	1:1	[b]	[b]	1:1	1:1	1:1	1:1
Tri-1-butylamine	1:1	1:1	2:1	–	–	–	1:1	1:1
Methanol	1:2	1:2	–	–	–	–	1:1	–
Ethanol	1:2	1:2	–	–	–	–	2,3	–
1-Propanol	1:2	1:2	1:1	–	–	–	1:2	–
2-Methyl-1-propanol	1:1	1:1	–	–	–	–	–	1:2
1-Butanol	1:2	1:1	–	–	–	–	1:2	2:3
t-Butanol	1:2	1:1	–	–	–	–	–	3:2
Cyclopentanol	[b]	1:1	1:1	–	–	–	–	–
Cyclohexanol	[b]	1:1	[b]	–	–	[b]	1:1	–
Acetone	1:2	1:1	1:1	–	1:1	–	1:1	–
Cyclopentanone	1:2	2:3	1:1	[b]	[b]	[b]	[b]	–
Cyclohexanone	1:2	1:2	–	–	–	–	–	–
Dimethylformamide	1:2	2:3	1:2	1:1	2:1	1:1	1:1	1:1
Dimethyl sulfoxide	1:2	1:2	1:2	1:2	1:2	1:2	2:1	1:1
Nitromethane	1:2	1:1	–	–	–	–	–	–
Nitroethane	1:2	1:1	–	–	–	–	–	–
Acetonitrile	1:2	1:2	–	–	–	–	3:1	–
Butyronitrile	1:2	1:1	–	–	–	–	3:2	–
Tetrahydrofuran	1:2	1:1	1:1	–	–	–	1:3	1:1
1,4-Dioxane	1:2	1:1	1:3	–	–	–	–	–
Toluene	[b]	1:1	1:2	[b]	–	–	–	–
Xylene	1:1	–	[b]	[b]	–	–	–	–

[a] Crystalline inclusion compounds (host:guest stoichiometric ratio) are also formed between: **1** and 3-methylpiperidine (1:2), 2-propanol (1:2), 2-methylcyclopentanone (1:2), (–)-fenchone (1:2), propionitrile (1:2), dichloromethane (1:2), diethylether (1:1); **2** and 2-methylcyclohexylamine (1:3), 3-methylcyclohexylamine (1:2), benzylamine (1:2), *N*-methylbenzylamine (1:2), pyridine (1:1), 2-butanol (1:1), 2-methylcyclohexanol (1:1), 3-methylcyclohexanol (1:1), 2-methylcyclopentenone (1:2), 3-methylcyclopentanone (1:1), 2-methylcyclohexanone (1:1), 3-methylcyclohexanone (2:1), 4-methylcyclohexanone (1:1), (–)-fenchone (1:2), γ-butyrolactone (1:1), propionitrile (1:1).

[b] Difficult to crystallize.

cies; Table 1). Inclusion formation obviously depends on structural parameters of the hosts such as size and nature of substituents (R) and of the bridging groups (X). Compared with the parent molecule **1** and its laterally substituted derivative **2**, the analogues **5–10** structurally modified at the bridge are certainly less efficient regarding the extent of inclusion formation. However, they are more selective hosts since they capture a smaller variety of guest molecules. This demonstrates different structural behaviour of a plain fluorene unit compared to the twisted and folded conformations of the X-bridged analogues (*cf.* **5–10**). Whilst a great number of structural results involving fluoreno hosts have furnished proof of the preference for π-stacking interaction in a crystal lattice [7–9, 12, 16], the non-planar bridged analogues such as here are more likely to act by the virtue of their van-der-Waals bulk. This corresponds to several inclusion structures of other host compounds that are characteristic of the discussed non-planar units but are more simple in constitution [17].

In the future, host molecules of this type that differ in the 2,2'-biphenyl construction element or have altered

functional groups are a promising target of further investigations as well as for testing of these compounds concerning their application as chemical sensors [13, 18].

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Experimental

Starting compounds were purchased from Janssen (Nettetal). M.p.s (uncorrected) were determined with a Reichert hot-stage apparatus. IR spectra (cm^{-1}) were recorded on a Pye-Unicam SP-1100 spectrometer. Proton and ^{13}C NMR spectra were measured for solutions (Me_4Si as internal standard, ppm) with Varian EM-360 (60 MHz) and Bruker WM 250 (250 MHz) spectrometers, respectively. Mass spectra were obtained using an A.E.I. MS-50 instrument or a Kratos Concept 1H (FAB-MS). Microanalyses were carried out by the Microanalytical Laboratory of the Institut für Organische Chemie und Biochemie, Universität Bonn.

2,2'-Dibromobiphenyl (**11**) and *ketones* **12** – **17** were prepared according to literature procedures (**11** [19], **14** [20], **15** [20], or purchased (**12**, **13**, **16**, **17**).

Host Compounds **1**–**4** were obtained as described [8a, 9].

Host Compounds 5 –10 (General Procedure)

A solution of 32.0 ml (51.2 mmol, 1.6 N in *n*-hexane) *N*-BuLi was added slowly under argon at 0 °C to 8.0 g (25.6 mmol) 2,2'-dibromobiphenyl (**11**) in 70 ml dry diethyl ether. After the reaction mixture had been stirred for another 2 h at the same temperatur, 51.2 mmol of the respective ketone **12**–**17** were added in portions as a solid within 0.5 h. The mixture was stirred for a further 2 h at room temperature, heated at reflux for 15 h, and subsequently hydrolyzed (NH_4Cl solution). The precipitate which formed was collected, washed with diethyl ether, and purified. Specific details for each compound are given below.

2,2'-Bis(1-hydroxy-4,5-dihydro-2:3,6:7-dibenzocycloheptatrien-1-yl)biphenyl (**5**)

Dibenzosuberone (**12**) was used; purification of the crude product by treatment with hot methanol and recrystallization from dimethylformamide.
White powder, m.p. >300 °C, yield 73%. – IR (KBr): υ = 3600 (OH), 3100 (C–H, Ar), 2980, 2960 (C-H, aliph.), 1500 (Ar), 1280, 1160 (C–O), 1120, 1040, 960, 920, 640. – ^1H NMR ($CDCl_3$): δ = 2.35–2.50 (m, 2 H, CH_2), 2.54–2.75 (m, 2 H, CH_2), 2.82–3.14 (m, 4 H, CH_2), 5.68 (s, 2 H, OH), 5.94–6.03 (dd, 2 Ar-H), 6.40–6.62 (m, 4 Ar-H), 6.85–7.09 (m, 12 Ar-H), 7.13–7.28 (m, 4 Ar-H), 8.12-8.23 (m, 2 Ar-H). – ^{13}C NMR ($CDCl_3$): δ = 147.03, 144.28, 141.46, 138.32, 136.93, 136.12, 131.59, 130.75, 128.12, 127.96, 126.57, 126.15, 125.24, 124.95, 124.14, 79.12, 32.93, 30.02.

$C_{42}H_{34}O_2$	Calcd. C 88.39	H 6.00
(570.3)	Found C 87.94	H 6.31
	Molecular mass 570.2 (FAB-MS, M^+)	

2,2'-Bis(1-hydroxy-2:3,6:7-dibenzocycloheptatrien-1-yl)biphenyl (**6**)

Dibenzosuberenone (**13**) was used; treatment of the solid with hot methanol, then with hot toluene and recrystallization from dimethyl sulfoxide.
White powder, m.p. >300 °C, yield 23 %. – IR (KBr): υ = 3400 (OH), 3100–3000 (C-H, Ar), 3000–2900 (C-H, aliph.), 1467, 1433, 1370, 1265, 1150 (C–O), 1025, 1005, 950, 915, 850, 790, 755. – ^1H NMR ($CDCl_3$): δ = 5.0 (s, 2 H, OH), 5.90-6.10 (d, 2 H, CH=CH), 6.23–6.46 (d, 2 H, CH=CH), 6.47–7.10 (m, 12 Ar-H), 7.13–7.56 (m, 7 Ar-H), 7.93–8.16 (d, 3 Ar-H). – ^{13}C NMR ($CDCl_3$): δ = 144.44, 143.09, 138.95, 138.58, 133.49, 132.65, 132.02, 131.20, 130.54, 127.69, 127.41, 126.79, 126.17, 125.52, 125.40, 124.57, 123.96, 123.54, 78.59.

$C_{43}H_{30}O_2$	Calcd. C 89.02	H 5.34
(566,2)	Found C 88.92	H 5.33
	Molecular mass 699.1 (FAB-MS, $M^+ + Cs^+$)	

2,2'-Bis(1-hydroxy-2:3,4:5,6:7-tribenzocycloheptatrien-1-yl)biphenyl (**7**)

Ketone **14** was used; treatment with refluxing diethyl ether.
White powder, m.p. >300 °C, yield 22%. – IR (KBr): υ = 3365 (OH), 3100–3000 (C-H, Ar), 1631 (C=C), 1470, 1433, 1400, 1160, 1125, 1060 (C-O), 1023, 913, 740. – ^1H NMR (DMSO-d_6): δ = 5.72–5.86 (dd, 2 Ar-H), 6.0–6.14 (dt, 2 Ar-H), 6.30–6.50 (m, 3 Ar-H), 6.56–6.71 (dt, 2 Ar-H), 6.8–7.53 (m, 21 Ar-H), 7.99–8.12 (d, 2 Ar-H). – ^{13}C NMR (DMSO-d_6): δ= 148.13, 147.25, 139.10, 138.25, 137.93, 137.70, 136.25, 134.47, 130.32, 129.77, 129.58, 128.80, 128.38, 128.16, 127.80, 127.57, 127.35, 127.15, 126.83, 125.79, 124.79, 124.53, 123.79, 122.26, 78.54.

$C_{50}H_{34}O_2$	Calcd. 666.2550
(666.8)	Found 666.2543 (MS, M^+)

2,2'-Bis(1-hydroxy-3",6"-epoxy-3",6"-dihydro-2:3,4:5,6:7-tribenzocycloheptatrien-1-yl)biphenyl (**8**)

Ketone **15** was used; recrystallization from toluene.
White powder, m.p. > 300 °C, yield 15%. – IR (KBr): υ = 3340 (OH), 3130, 3050 (C-H, Ar), 1629 (C=C), 1402, 1272, 1160, 1125, 1025 (C-O), 882, 834, 754. – ^1H NMR (DMSO-d_6): δ = 4.52–5.15 (m, 6 H, 2 OH, 4 Epoxy-H), 5.16–7.65 (m, 28 Ar-H). – ^{13}C NMR (DMSO-d_6): δ = 148.46, 148.00, 141.84, 141.49, 140.42, 138.75, 138.65, 138.28, 133.06, 131.65, 130.39, 127.92, 127.73, 126.62, 126.39, 125.63, 125.42, 124.42, 123.88, 120.57, 120.46, 84.30, 84.10, 79.16.

$C_{50}H_{34}O_2$	Calcd. 698.2448
(698.8)	Found 698.2491 (MS, M^+)

2,2'-Bis(9-hydroxy-9-xanthenyl)biphenyl (**9**)

Xanthone (**16**) was used; recrystallization from ethanol.
White powder, m.p. 217–219 °C, yield 49%. – IR (KBr): υ = 3460 (OH), 3100–3000 (C-H, Ar,) 1610, 1550 (C=C), 1450, 1320, 1250 (OH), 1200, 1120 (C-O), 940, 890, 760, 635, 600, 530. – ^1H NMR ($CDCl_3$): δ = 4.13 (s, 2 H, OH), 6.46–7.56 (m, 24 Ar-H). – ^{13}C NMR ($CDCl_3$): δ = 150.10, 149.13, 144.40, 141,15, 132.88, 130.78, 129.34, 129.08, 128.90, 128.79, 128.55, 126.39, 125.06, 123.80, 122.87, 116.10, 115.73, 72.76.

$C_{38}H_{26}O_4$	Calcd. C 82.23	H 5.13
(546.6)	Found C 82.42	H 5.57
	Molecular mass 569 (FAB-MS, $M^+ + Na^+$)	

2,2'-Bis(9-hydroxy-9-thioxanthenyl)biphenyl (**10**)

Thioxanthone (**17**) was used; recrystallization from toluene, then from ethanol.

Colourless crystals, m.p. 187–189 °C, yield 37%. – IR (KBr): υ = 3550 (OH), 3135, 3130 (C-H, Ar), 1570 (C=C), 1440 (C-H), 1265 (OH), 1175, 1150 (C-O), 1020, 900, 755, 625. – ^{1}H NMR (DMSO-d_6): δ = 5.23 (s, 2 H, OH), 5.80–6.06 (m, 3 Ar-H), 6.20–7.25 (m, 18 Ar-H), 7.73–8.30 (m, 3 Ar-H). – ^{13}C NMR (DMSO-d_6): δ = 142.47, 141.15, 140.71, 138.26, 133.12, 132.10, 130.43, 128.93, 128.24, 127.43, 127.13, 126.88, 126.63, 126.54, 126.38, 126.04, 125.43, 125.35, 78.03.

$C_{38}H_{26}O_2S_2$	Calcd. C 77.84	H 4.86
(578.7)	Found C 77.77	H 4.53
	Molecular mass Calcd. 578.1366	
	Found 578.1359 (MS, M^+)	

Crystalline Inclusion Compounds

The corresponding host compound was dissolved under heating in a minimum amount of the respective guest solvent. After storage for 12 h at room temperature, the crystals which formed were collected, washed with diethyl ether or methanol, and dried (1 h, 15 Torr, room temperature). Host:guest stoichiometric ratios were determined by ^{1}H NMR integration. Data for each compound are given in Table 1.

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