

# Host–Guest Binding in Two Coordinatocathrates of 1,1'-Binaphthyl-2,2'-dicarboxylic Acid. X-Ray Crystal Structures of the Inclusion Compounds with 1-Propanol (2:1) and *t*-Butanol (1:1)

MÁTYÁS CZUGLER

*Central Research Institute of Chemistry, Hungarian Academy of Sciences, H-1525 Budapest  
POB 17, Hungary*

EDWIN WEBER

*Institut für Organische Chemie und Biochemie der Universität Bonn, Gerhard-Domagk-Strasse 1,  
D-5300 Bonn-1, Germany*

(Received 12 July 1990; in final form 24 October 1990)

**Abstract.** 1,1'-Binaphthyl-2,2'-dicarboxylic acid (**1**) forms crystalline inclusion compounds with 1-PrOH (2:1) and *t*-BuOH (1:1). X-ray crystal structures of the two inclusion compounds are reported. Crystals of 1-1-PrOH (2:1) show triclinic ( $P\bar{1}$ ) symmetry with the unit cell dimensions  $a = 10.160(1)$ ,  $b = 14.050(2)$ ,  $c = 15.167(1)$  Å,  $\alpha = 100.37(1)$ ,  $\beta = 104.40(1)$ , and  $\gamma = 94.82(1)^\circ$ . Crystals of 1-*t*-BuOH (1:1) are monoclinic ( $P2_1/n$ ) with the cell dimensions  $a = 10.603(5)$ ,  $b = 14.377(4)$ ,  $c = 15.664(7)$  Å,  $\beta = 104.24(4)^\circ$ . In both structures, H-bonded loops involving host —COOH functions and guest —OH groups establish the supramolecular association. They relate these coordinatocathrates to previous alcohol inclusions of **1**. Due to the unusual 2:1 (host:guest) stoichiometry, additional dimer-like interactions between —COOH groups of host molecules are found in the 1-PrOH inclusion compound. From the point of view of topology these structures can be referred to as channel inclusion compounds.

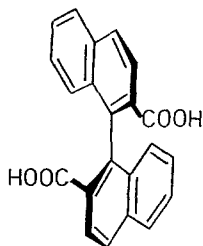
**Key words.** Inclusion compounds, X-ray crystal structure analysis, coordinatocathrates, carboxylic host, guest alcohols, hydrogen bonding.

**Supplementary Data** relating to this article are deposited with the British Library as Supplementary Publication No. SUP. 82105 (36 pages).

## 1. Introduction

Using the concept of coordinatocathrate formation [1] it is possible to engineer specific inclusion of an appropriately chosen guest substance even from a mixture containing other compounds. Selectivity upon crystallization reflects the efficiency of host-guest interactions in terms of steric and electronic fits (compressibility and coordination features match). Representative crystal structures [2–6] indicate that for coordinatocathrates of carboxylic hosts and hydroxylic guests hydrogen bonding [7] plays a dominating role. Certain key hydrogen bonding schemes became apparent [1, 8] depending on the steric and functional conditions of the interacting host–guest systems, as alluded above. Hydrogen bonding loops of 10-, 12-, and 24-membered ring size are being constructed in these lattices. To shed light on the generality of

this observation we report the results of crystal structure determinations of two further examples of this class of lattice inclusion compound. They refer to inclusion compounds of **1** (1,1'-binaphthyl-2,2'-dicarboxylic acid) [2] with 1-propanol (2:1) and *t*-butanol (1:1).

**1**

## 2. Experimental

### 2.1. SAMPLE PREPARATION

Host compound **1** was synthesized as described previously [2]. Crystals of the inclusion compounds were obtained by recrystallization of host **1** from the corresponding guest solution.

### 2.2. X-RAY DATA COLLECTION AND PROCESSING

**1**·1-PrOH (2:1): A colorless chunky crystal having approximate dimensions of  $0.18 \times 0.20 \times 0.30$  mm was mounted in a glass capillary in a random orientation. Preliminary examination and data collection were performed with MoK $_{\alpha}$  radiation ( $\lambda = 0.71073$  Å) on an Enraf-Nonius CAD4 computer controlled  $\kappa$  axis diffractometer equipped with a graphite-crystal incident beam monochromator. Cell constants (Table I) and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range  $12 > \theta < 17^\circ$ , measured by the computer-controlled diagonal slit method of centering. There were no systematic absences; the space group was determined to be  $P\bar{1}$  (No. 2). Data were collected at a temperature of 296(1) K using the  $\theta$ - $2\theta$ -scan technique with variable scan rate, to a maximum  $2\theta$  of  $50.0^\circ$ ; (minimum  $2\theta = 0.0^\circ$ ). A total of 7162 reflections were collected (Table I) of which 5605 were unique. The intensities of the standards remained constant within experimental error throughout data collection. No decay correction was applied. Lorentz and polarization corrections were applied to the data. No absorption correction was made. An extinction correction was not performed.

**1**·*t*-BuOH (1:1): Data collection was performed in a similar manner as described before (Table I). Scattering of the crystal was relatively weak thus out of the 4061 collected data only 1107 proved to be useful for the analysis.

Table I. Crystal data and experimental details<sup>a</sup>.

Compound	1-1-PrOH (2:1)	1- <i>t</i> -BuOH (1:1)
Formula	C <sub>47</sub> H <sub>36</sub> O <sub>9</sub>	C <sub>26</sub> H <sub>24</sub> O <sub>5</sub>
Formula weight	758.8	416.5
Cell constants:		
<i>a</i> , Å	10.160(1)	10.603(5)
<i>b</i> , Å	14.050(2)	14.377(4)
<i>c</i> , Å	15.167(1)	15.664(7)
$\alpha$ , deg	100.37(1)	90.00
$\beta$ , deg	104.40(1)	104.24(4)
$\gamma$ , deg	94.82(1)	90.00
<i>V</i> <sub>c</sub> , Å <sup>3</sup>	2043.8(8)	2314.4(3.2)
Space group:	<i>P</i> $\bar{1}$	<i>P</i> 2 <sub>1</sub> / <i>n</i>
<i>Z</i>	2	4
<i>D</i> <sub>c</sub> , g cm <sup>-3</sup>	1.21	1.195
$\mu$ (MoK $\alpha$ ), cm <sup>-1</sup>	0.70	0.77
Data collection parameters:		
Radiation	MoK $\alpha$ ( $\lambda = 0.71073$ Å)	MoK $\alpha$ ( $\lambda = 0.71073$ Å)
Scan technique	$\theta/2\theta$	$\theta/2\theta$
No. of refl. collected	7162	4061
with $I > 3*\sigma(I)$	3878	1107
Crystal size, mm	0.18 × 0.20 × 0.30	0.2 × 0.2 × 0.25
Temperature, K	296(1)	296(1)
Least squares parameters:		
No. of atoms	92	56
Variables	505	280
Observations	3832 <sup>c</sup> [ $I > 3.0*\sigma(I)$ ]	1366 [ $I > 1.5*\sigma(I)$ ]
<i>R</i>	0.0612	0.0888
<i>R</i> <sub>w</sub>	0.0986	0.1443
<i>R</i> <sub>tot</sub>	0.0937	0.0888
Largest shift	1.32* its e.s.d.	0.54* its e.s.d.
<i>S</i> <sup>b</sup>	1.77	0.88
Highest residual electron density, e Å <sup>-3</sup>	0.56	0.26

<sup>a</sup>E.s.ds, where given, are in parentheses.

<sup>b</sup> $S = [\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$

<sup>c</sup>38 reflections which were suspected to be in error due to either measurement errors or suffering from uncorrected secondary extinction effects were suppressed from the final refinement.

The structure was refined in full-matrix least-squares where the function minimized was  $\sum w(|F_o| - |F_c|)^2$  and the weight *w* is defined as  $4F_o^2/\text{sig}(F_o^2)^2$ . The standard deviation  $\text{sig}(F_o^2)$  is defined as follows:  $\text{sig}(F_o^2) = (S^2(C + R^{2B}) + (pF_o^2)^2/Lp)^{1/2}$ , where *S* is the scan rate, *C* is the total integrated peak count, *R* is the ratio of scan time to background counting time, *B* is the total background count, *Lp* is the Lorentz polarization factor, and the parameter *p* is a factor introduced to downweight intense reflections. Here *p* was set to 0.10 and 0.045 for the 1-PrOH and the *t*-BuOH inclusions, respectively.

### 2.3. STRUCTURE DETERMINATION AND REFINEMENT

The structure of 1:1-PrOH (2:1) was solved by direct methods. A total of 44 atoms were located from an E-map prepared from the best phase set. The remaining atoms were located in succeeding difference-weighted Fourier syntheses. Hydrogen atoms were positioned on the basis of geometric evidence and added to the structure factor calculations but their positions were not refined. Positions for the putative H atoms at the carboxylic and alcoholic functions were deduced from difference electron density syntheses calculated near to the full-matrix least-squares refinement termination. Scattering factors were taken from Cromer and Waber [9]. Anomalous dispersion effects were included in  $F_c$  [10]; the values of  $f'$  and  $f''$  were those of Cromer [11]. The final cycle of refinement included 505 variable parameters and converged with unweighted and weighted agreement factors of 6.1 and 9.9%.

An initial structure model for 1:*t*-BuOH (1:1) was also yielded by direct methods and gave a plausible starting model which was refined in the same way as described for the 1-PrOH case. The relatively high final *R* values of 8.8 and 14.4% for the appropriate unweighted and weighted residuals are explained by the poor crystal quality yielding only about a quarter of the data set as observed (cf. Table I). Hydrogen atomic positions for this structure were deduced from geometric evidence, where appropriate. These H atoms were included in the structure factor calculations, but their parameters were not treated. Attempts at locating the H atoms of —OH functions from difference electron density syntheses yielded rather dubious positions. These are only given (cf. Table IX, deposited) as indicative of a possible position in hydrogen bonding, and no such site could be proposed for one of the —COOH functions.

Other pertinent details of the refinements are summarized in Table I. All calculations were performed on a PDP-11/34 minicomputer using SDP-PLUS and local programs [12].

## 3. Results and Discussion

The final atomic coordinates of the non-hydrogen atoms of the two inclusion compounds are given in Tables II and III. Some relevant interplanar angles are shown in Table IV. Hydrogen bonding parameters are listed in Table V. Shape descriptors in the form of bond distances and angles (Tables VI and VII), lists of coordinates of the H atoms (Tables VIII and IX), anisotropic thermal parameters (Tables X and XI), and structure factors (Tables XII and XIII) for both complexes have been deposited. The molecular structures of the two inclusion compounds are shown in Figures 1 and 2, and packing diagrams as well as H bond excerpts are illustrated in Figures 3 and 4.

### 3.1. MOLECULAR STRUCTURES

The guest molecules (1-PrOH and *t*-BuOH) show signs of apparent disorder as indicated by the unreasonable bonding distances (Tables VI and VII) and high temperature factors for the pertinent atoms, particularly for 1-PrOH. This observation applies in general to inclusion compounds, where some parts of the molecules composing the crystal structure are less rigidly held, thus disorder readily develops.

Table II. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters for the non-hydrogen atoms of 1:1-PrOH (2:1) (e.s.ds are in parentheses).

Atom	$x/a$	$y/b$	$z/c$	$B_{eq}/B$
C(11)	4048(2)	2901(2)	5328(1)	2.78(5)
C(21)	5234(2)	3260(2)	6032(1)	3.02(5)
C(31)	6338(3)	3796(2)	5850(2)	3.87(6)
C(41)	6279(3)	3937(2)	4981(2)	4.08(6)
C(4a1)	5108(3)	3586(2)	4248(2)	3.54(6)
C(51)	5024(3)	3727(3)	3335(2)	4.99(8)
C(61)	3874(4)	3396(3)	2642(2)	6.7(1)
C(71)	2728(4)	2888(3)	2807(2)	7.0(1)
C(81)	2770(3)	2731(2)	3680(2)	4.93(7)
C(8a1)	3956(2)	3070(2)	4420(2)	3.23(5)
C(91)	5419(2)	3047(2)	6965(2)	3.73(5)
O(101)	4588(2)	2517(2)	7174(1)	5.72(5)
O(111)	6540(2)	3463(2)	7566(1)	7.94(6)
C(11')	2813(2)	2390(2)	5497(1)	2.89(5)
C(21')	2585(2)	1396(2)	5432(1)	3.20(5)
C(31')	1403(3)	967(2)	5614(2)	3.79(6)
C(41')	463(3)	1521(2)	5855(2)	4.04(6)
C(4a1')	658(2)	2541(2)	5932(2)	3.41(5)
C(51')	-291(3)	3148(2)	6196(2)	4.10(6)
C(61')	-61(3)	4125(2)	6277(2)	4.94(7)
C(71')	1094(3)	4557(2)	6086(2)	4.62(7)
C(81')	2022(3)	3999(2)	5830(2)	3.79(6)
C(8a1')	1837(2)	2978(2)	5747(1)	3.01(5)
C(91')	3611(3)	778(2)	5211(2)	3.42(5)
O(101')	4659(2)	1089(1)	5045(1)	5.13(5)
O(111')	3326(2)	-142(1)	5204(2)	5.29(5)
C(12)	7315(3)	2577(2)	11022(2)	3.67(6)
C(22)	6208(3)	2142(2)	10293(2)	4.34(6)
C(32)	5200(4)	1434(3)	10413(2)	5.63(8)
C(42)	5329(4)	1185(3)	11260(2)	6.14(9)
C(4a2)	6443(3)	1621(2)	12027(2)	4.60(7)
C(52)	6599(4)	1383(2)	12913(2)	5.58(9)
C(62)	7679(4)	1828(3)	13656(2)	5.96(9)
C(72)	8668(4)	2514(2)	13547(2)	5.45(8)
C(82)	8566(3)	2754(2)	12704(2)	4.51(7)
C(8a2)	7447(3)	2322(2)	11915(2)	3.80(6)
C(92)	6016(3)	2379(2)	9361(2)	4.61(7)
O(102)	6818(2)	2960(2)	9180(1)	7.03(6)
O(112)	4910(2)	1917(2)	8744(1)	7.09(6)
C(12')	8356(3)	3362(2)	10973(1)	3.86(6)
C(22')	9599(3)	3187(2)	10807(2)	3.73(6)
C(32')	10609(3)	3972(2)	10872(2)	4.88(7)
C(42')	10364(4)	4918(2)	11095(2)	5.70(8)
C(4a2')	9100(4)	5137(2)	11223(2)	5.00(7)
C(52')	8818(5)	6106(2)	11395(3)	6.48(9)
C(62')	7597(6)	6294(3)	11521(3)	8.3(1)
C(72')	6582(5)	5534(3)	11479(3)	7.2(1)
C(82')	6822(4)	4592(3)	11321(2)	5.50(8)
C(8a2')	8088(3)	4357(2)	11182(2)	4.46(7)
C(92')	9864(3)	2171(2)	10512(2)	4.33(7)
O(102')	9106(2)	1450(1)	10470(2)	6.19(6)
O(112')	11032(2)	2127(2)	10299(2)	6.91(6)
O(P1)	11403(3)	450(2)	9423(2)	7.52(7)
C(P1)	11174(6)	391(4)	8399(3)	10.0(1)
C(P2)	958(10)	50(7)	7842(5)	19.1(3)
C(P3)	816(10)	620(9)	7706(6)	23.5(4)

Table III. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters for the non-hydrogen atoms of 1-*t*-BuOH (1:1) (e.s.ds are in parentheses).

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B<sub>eq</sub>/B</i>
C(1)	4866(6)	1780(4)	1018(4)	3.7(2)
C(2)	5671(6)	1135(5)	779(4)	3.9(2)
C(3)	6041(8)	1214(6)	-30(4)	5.3(2)
C(4)	5574(8)	1917(6)	-613(4)	5.9(3)
C(4a)	4746(7)	2594(6)	-402(5)	5.4(2)
C(5)	4284(9)	3332(7)	-999(5)	7.6(4)
C(6)	350(1)	3987(8)	-764(7)	11.0(5)
C(7)	315(1)	3940(8)	36(7)	9.7(4)
C(8)	3602(8)	3252(6)	626(5)	6.6(3)
C(8a)	4396(7)	2539(6)	411(4)	4.7(2)
C(9)	6203(7)	371(5)	1368(5)	4.6(2)
O(10)	5879(6)	157(4)	2023(3)	6.3(2)
O(11)	7138(5)	-102(4)	1117(3)	7.0(2)
C(1')	4418(5)	1766(4)	1844(3)	3.5(2)
C(2')	5041(6)	2256(5)	2583(4)	4.5(2)
C(3')	4575(8)	2243(6)	3337(4)	5.9(3)
C(4')	3483(7)	1728(6)	3380(5)	6.1(3)
C(4a')	2814(6)	1203(6)	2621(4)	5.1(2)
C(5')	1709(8)	670(6)	2639(6)	7.6(3)
C(6')	1079(8)	190(7)	1886(8)	9.1(4)
C(7')	1535(7)	230(7)	1134(6)	7.5(3)
C(8')	2582(7)	754(5)	1093(5)	5.7(2)
C(8a')	3259(6)	1251(5)	1850(4)	4.4(2)
C(9')	6231(7)	2836(5)	2605(5)	5.0(2)
O(10')	6495(6)	3486(4)	3114(4)	8.6(2)
O(11')	6969(4)	2564(4)	2148(3)	7.4(2)
O(B1)	6022(4)	-1472(4)	2870(3)	7.2(2)
C(B1)	5364(7)	-1526(5)	3552(4)	5.1(2)
C(B2)	556(1)	-2455(8)	3948(7)	9.6(4)
C(B3)	573(2)	-767(9)	4177(9)	16.0(7)
C(B4)	397(1)	-134(1)	307(1)	14.3(7)

Table IV. Characteristic dihedral angle (deg) of main planes in the host molecules (e.s.ds are in parentheses).

(a) 1:1-PrOH (2:1) <sup>a</sup>								
Naphthyl/--COOH			Naphthyl/Naphthyl			--COOH/--COOH		
Plane/Plane	Angle		Plane/Plane	Angle		Plane/Plane	Angle	
1	2	-7.6(2)	1	3	89.4(7)	2	4	-85.0(4)
3	4	3.2(3)	5	7	87.4(1)	6	8	-87.2(3)
5	6	-1.3(3)						
7	8	-7.0(3)						
(b) 1- <i>t</i> -BuOH (1:1) <sup>b</sup>								
Naphthyl/--COOH			Naphthyl/Naphthyl			--COOH/--COOH		
Plane/Plane	Angle		Plane/Plane	Angle		Plane/Plane	Angle	
1	2	-10.8(3)	1	3	86.0(2)	2	4	-80.8(7)
3	4	29.5(2)						

<sup>a</sup>Planes are numbered as: 1 = Naphthyl-1; 2 = --COOH-1; 3 = Naphthyl-2; 4 = --COOH-2; 5 = Naphthyl-3; 6 = --COOH-3; 7 = Naphthyl-4; 8 = --COOH-4.

<sup>b</sup>Plane numbers apply for one molecule of the asymmetric unit.

Table V. Hydrogen bonding parameters (distance, Å; Angle deg.)<sup>a</sup>

Atoms involved	Symmetry	Donor...Acceptor	Donor—H	H...Acceptor	∠ Donor—H...Acceptor
<b>(a) 1:1-PrOH (2:1)</b>					
O(111)—H(111)...O(102)	(x, y, z)	2.62(1)	0.88	1.75	173
O(112)—H(112)...O(101)	(x, y, z)	2.62(1)	0.98	1.66	169
O(112)—H(112')...O(P1)	(x, y, z)	2.59(1)	1.00	1.61	168
O(111')—H(111')...O(101')	(-x, -y, -z)	2.60(1)	0.97	1.65	166
O(P1)—H(OP1)...O(102')	(1-x, -y, 1-z)	2.72(1)	1.04	1.72	160
<b>(b) 1:1-BuOH (1:1)<sup>b,c</sup></b>					
O(11)—H(11)...O(10')	(1.5-x, 0.5+y, 0.5-z)	2.61(1)	0.98	2.06	114
O(B1)—H(OB1)...O(11')	(1.5-x, 0.5+y, 0.5-z)	2.55(1)	0.98	1.61	158
O(10)...O(B1)	(x, y, z)	2.68(1)			

<sup>a</sup>Standard deviations were omitted because these parameters tend to be seriously underestimated under the circumstances. Thus, H atoms in the present model should rather be taken as indicative of a feasible hydrogen bonding.

<sup>b</sup>The H atom positions of the —OH groups are dubious in this structure. Consequently, they are only indicative of a possible H-bonding arrangement.

<sup>c</sup>Only one of the two possible H atom positions for the guest is used here.

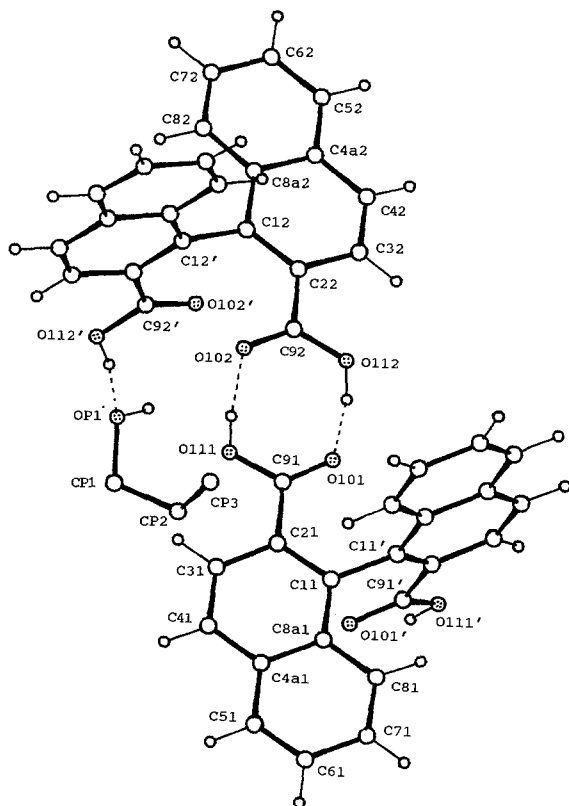


Fig. 1. Perspective view of the asymmetric unit in the 1:1-PrOH (2:1) coordinatoclathrate. H atoms of the C—H type in the guest molecule were omitted for the sake of clarity. O atoms are dotted; solid and dashed lines represent covalent and H bonds, respectively.

The dihedral angles (Table IV) of the relevant planar moieties in the host molecules indicate that their main planes are nearly orthogonal to each other. A similar behaviour of host **1** was found in previously described alcohol inclusion compounds [2]. Much the same applies to the mutual alignment of the intramolecular carboxyl groups which are also nearly orthogonal to each other. While these —COOH groups incline only slightly with respect to their anchoring naphthyl planes in the 1·PrOH complex, the same parameter in the 1·*t*-BuOH case shows an appreciable twist which may be attributed to the increased steric requirements of the guest molecule. Also in this regard, a similarity between the present and the previously described alcohol inclusion compounds [2] is evident.

### 3.2. PACKING RELATIONS AND HOST-GUEST INTERACTIONS

The analysis of the intermolecular relationships in the structure of the 1·PrOH (2:1) inclusion compound (Table V and Figure 3) indicates that the carboxyl functions of **1** play a somewhat different role from those in the other alcohol



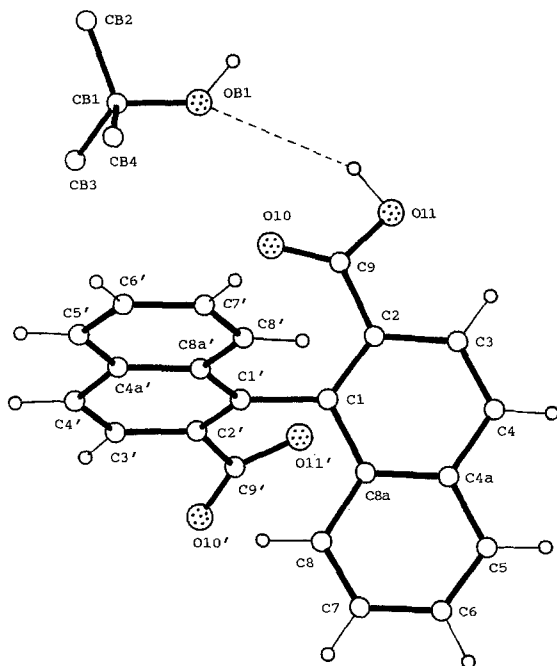


Fig. 2. Perspective view of the asymmetric unit in the 1-*t*-BuOH (1:1) coordinatoclathrate. H atoms of the C—H type in the guest molecule were omitted for the sake of clarity. O atoms are dotted; solid and dashed lines represent covalent and H bonds, respectively.

inclusion compounds of this host [2]. Three out of the four stoichiometric functions establish dimer-like interactions between —COOH groups fused into infinite chains. The helix formed by the so fused hosts encloses a channel interwoven by one of the carboxylic dimers. The fourth —COOH group protrudes into this cavity and binds the alcohol molecule there. The loop of hydrogen bonding is formed around a center of symmetry thus yielding the analogous 12-membered ring pattern observed for some of the simple alcohol inclusion compounds of this host (with MeOH, EtOH, and 2-PrOH) [2] although these have a different stoichiometry of 1:2.

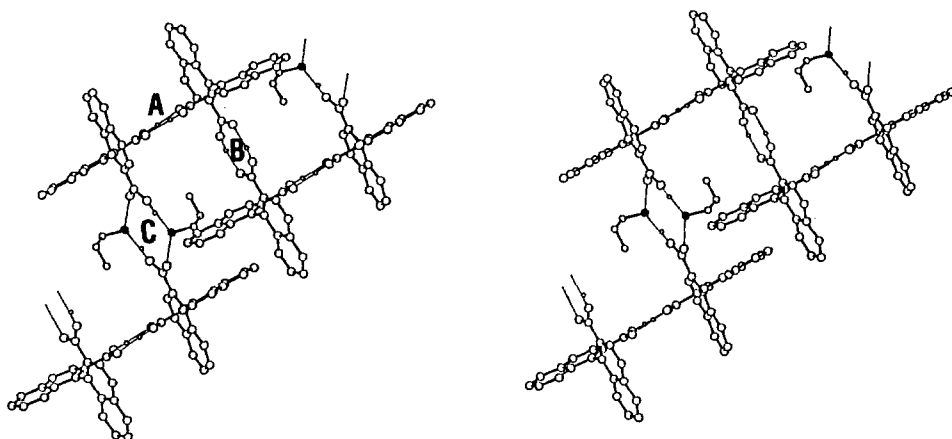
Unlike 1:1-PrOH (2:1) the 1-*t*-BuOH structure is a close analogy of the 2-BuOH 1:1 associate of **1** [2]. One finds the same non-symmetric 10-membered H bond ring system which has been observed in that crystal (Figure 4).

Determination of the H atomic positions in both crystals presented here should be taken as only indicative of perhaps one of the possible H-bonding arrangements. Disorder and the lower quality of the data (Table V) do not permit far-reaching conclusions as to the precise location of these atoms.

### 3.3. CONCLUDING REMARKS

This report concludes that the two structures presented present further corroboration of the structural and design principles outlined previously [2]. Thus, the

(a)



(b)

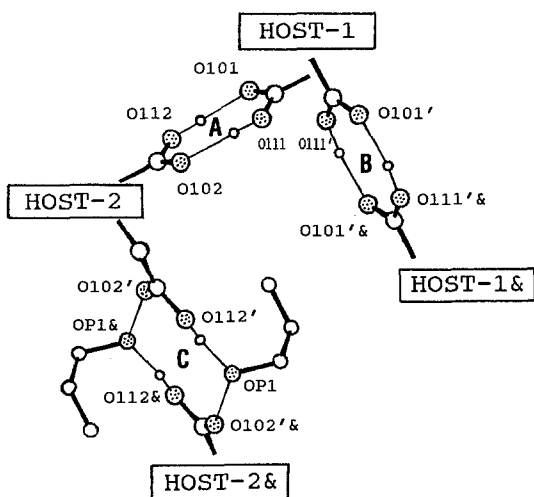
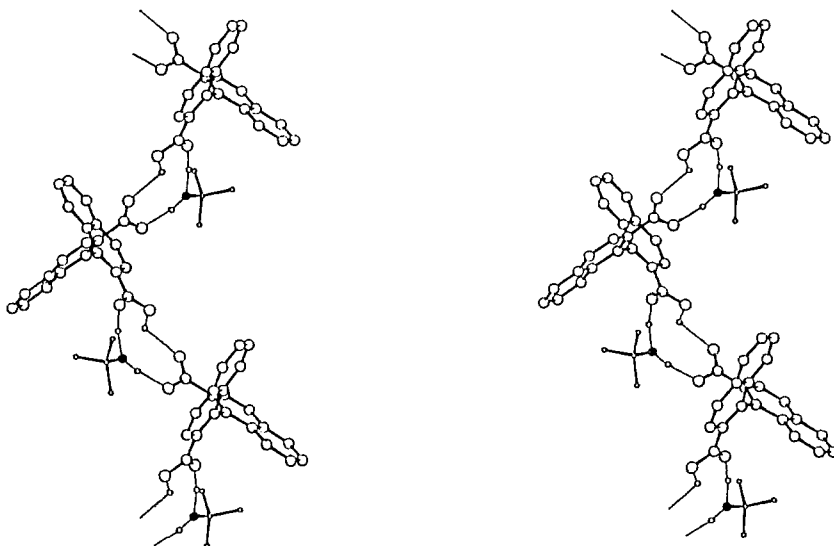


Fig. 3. Stereo packing illustration (a) of the 1:1-PrOH (2:1) coordinatocathrate (guest O atom as bold dot). Hydrogen-bonded rings are shown as thin lines and are also illustrated in schematics in the insert (b) with relevant labelling of atoms. H atoms of the guest molecule as well as those of the host skeleton were omitted for the sake of clarity.

coordinatocathrate action of **1** seems to work even for an odd host-guest stoichiometry as in the 2:1 1:1-PrOH inclusion case. It indicates that future predictions with reference to the host-guest coordination are promising in this class of compounds. Structural identity up to a point which is close to crystalline isomorphy is also maintained between the *t*-BuOH and the 2-BuOH inclusion compounds of **1**.

(a)



(b)

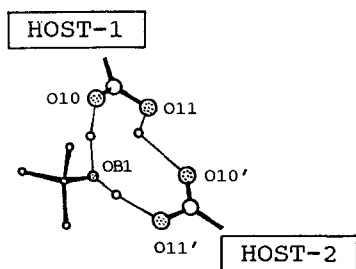


Fig. 4. Stereo packing illustration (a) of the 1-*t*-BuOH (1:1) coordinatoclathrate (guest O atom as bold dot). Hydrogen-bonded rings are shown as thin lines and are also illustrated in schematics in the insert (b) with relevant labelling of atoms. Two possible H atoms are drawn for the alcoholic OH (cf. Table IX). H atoms of the C—H type in the guest molecule as well as those of the host skeleton were omitted for the sake of clarity.

## Acknowledgements

We are indebted to the Deutsche Forschungsgemeinschaft (SFB 334), the Fonds der Chemischen Industrie, and the Hungarian Research Fund (Grant No. OTKA 1028) for financial support.

## References

1. E. Weber and M. Czugler: *Functional Group Assisted Clathrate Formation – Scissor-like and Roof-shaped Host Molecules* (Molecular Inclusion and Molecular Recognition – Clathrates – II, Topics in Current Chemistry, vol. 149, Ed. E. Weber), p. 45. Springer-Verlag, Berlin–Heidelberg–New York (1988).

2. E. Weber, I. Csöregyh, B. Stensland, and M. Czugler: *J. Am. Chem. Soc.*, **106**, 3297 (1984).
3. I. Csöregyh, M. Czugler, and E. Weber: *Structural Studies of the Inclusion Properties of a Novel Coordinato-clathrate Host* (Molecular Structure: Chemical Reactivity and Biological Activity, Eds. J. J. Stezowski, J.-L. Huang, and M.-C. Shao), p. 390. Oxford University Press (1988).
4. I. Csöregyh, M. Czugler, and E. Weber: *Z. Kristallogr.* **185**, 201 (1988).
5. E. Weber, I. Csöregyh, J. Ahrendt, S. Finge, and M. Czugler: *J. Org. Chem.* **53**, 5831 (1988).
6. E. Weber, M. Hecker, I. Csöregyh, and M. Czugler: *J. Am. Chem. Soc.* **111**, 7866 (1989).
7. See also: (a) G. L. Gartland, G. R. Freeman, and C. E. Bugg: *Acta Crystallogr.* **B30**, 1841 (1974). (b) K. Kamiya, M. Takamoto, Y. Wada, and M. Asai: *Acta Crystallogr.* **B37**, 1626 (1981). (c) J. Rebek, Jr.: *Science* **235**, 1478 (1987). (d) J. Rebek, Jr.: *Recent Progress in Molecular Recognition* (Molecular Inclusion and Molecular Recognition – Clathrates – II, Topics in Current Chemistry, vol. 149, Ed. E. Weber), p. 189. Springer-Verlag, Berlin–Heidelberg–New York (1988).
8. E. Weber: *J. Mol. Graphics* **7**, 12 (1989).
9. D. T. Cromer and J. T. Waber: *International Tables for X-Ray Crystallography*, vol. IV. The Kynoch Press, Birmingham, England (1974) (Distr. Kluwer Academic Publisher, Dordrecht, The Netherlands); see Table 2.2B.
10. J. A. Ibers and W. C. Hamilton: *Acta Crystallogr.* **17**, 781 (1964).
11. See Table 2.3.1. in ref. [9].
12. B. A. Frenz: *The Enraf–Nonius CAD 4 SDP – A Real-time System for Concurrent X-Ray Data Collection and Crystal Structure Determination* (Computing in Crystallography, Eds. H. Schenk, R. Olthof-Häzekamp, H. van Koningsveld, and G. C. Bassi), p. 64. Delft University Press, Delft, Holland (1978).