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Chiral crystalline hosts derived from lactic acid.* X-ray crystal structures of optically resolved and racemic host compound 1,1-diphenyl-1,2-propanediol and a 2:1 complex between optically resolved host and 3-picoline

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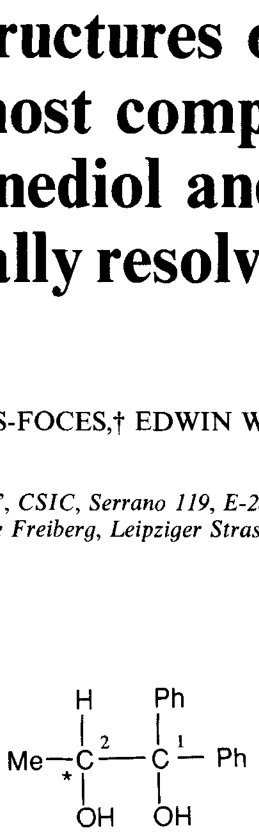
The X-ray crystal structures of racemic (1) and *S*-1,1-diphenyl-1,2-propanediol (2), and of a 2:1 inclusion complex (3) of 2 with 3-picoline are reported. Three different binding schemes characterize the packing of these structures. Only one of the two hydroxy groups (that which is not related to the asymmetric carbon) is involved in O–H···O hydrogen bonds responsible for the formation of dimers and chains in 1 and 2, leaving the other OH group for stabilization of dimers through OH···phenyl interactions in 1 or free of interactions in 2. In the crystal structure of the chiral complex 3, the hydroxy groups link the two independent host molecules, A and B, the 3-picoline guest to the B host molecule, and a remaining one forms H-bonded chains along the *c* axis.

INTRODUCTION

Considerable interest in crystalline inclusion compounds (clathrates)[2,3] has arisen over the past few years in view of their practical uses.[4–7] Among them, optical resolution of guest compounds via clathrate formation is an important problem.[8–10] This requires chiral host molecules that can easily be separated into enantiomers or are readily available in optically pure form from natural chiral sources.

Recently we have shown that natural lactic acid when modified by addition of two bulky aromatic units yields optically active crystalline hosts of type **I** that are efficient in the enantio separation of different guest compounds either by inclusion crystallization or vapour sorption.[1] In this context, the racemate resolution of 3-methylcyclohexanone with **2** to give an inclusion compound **4** has proved particularly useful.[11]



$$\mathrm{Me}-\overset{\mathrm{H}}{\underset{\mathrm{OH}}{\overset{2}{\mathrm{C}}}}{}^{*}-\overset{\mathrm{Ph}}{\underset{\mathrm{OH}}{\overset{1}{\mathrm{C}}}}-\mathrm{Ph}$$

I

1: (2*RS*) (racemic)
2: (2*S*)
3: 2·3-picoline (2:1)
4: 2·3-methylcyclohexanone (2:1)

In order to increase background knowledge, we became interested in the crystal structures of the free host compound **2**, its racemic analogue **1** and an inclusion compound of **2** with 3-picoline (**3**). Here we report structural determinations of these three species (**1–3**).

RESULTS AND DISCUSSION

Molecular structure

The geometrical characteristics derived from the crystallographic analyses of compounds **1**, **2** and **3**, together with that of **4** (already briefly reported[1]), are given in Tables 1 and 2. The content of the asymmetric unit (the guest and two host molecules) of **3** and **4** are illustrated in figure 1 for comparison.

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* Part 2. For part 1 of this series see ref 1. †To whom correspondence should be addressed.

Table 1 Selected bond distances, bond angles and torsion angles for the host molecules

	Compound					
	1	**2**	**3**		**4**	
Bond distances (Å)			*A*	*B*	*A*	*B*
C(1)–C(2)	1.546(2)	1.550(3)	1.547(14)	1.546(15)	1.536(5)	1.545(5)
C(1)–O(4)	1.430(2)	1.422(3)	1.430(12)	1.430(12)	1.436(4)	1.425(4)
C(1)–C(11)	1.534(2)	1.542(3)	1.542(14)	1.520(14)	1.533(5)	1.532(5)
C(1)–C(21)	1.528(2)	1.532(3)	1.534(13)	1.548(12)	1.534(4)	1.531(5)
C(2)–C(3)	1.517(2)	1.506(4)	1.509(14)	1.519(13)	1.517(6)	1.522(6)
C(2)–O(5)	1.437(2)	1.436(3)	1.443(11)	1.461(11)	1.434(4)	1.446(4)
Bond angles (°)						
C(11)–C(1)–C(21)	108.9(1)	110.2(2)	109.3(7)	109.2(8)	108.0(2)	107.6(3)
O(4)–C(1)–C(21)	107.6(1)	106.4(2)	105.5(8)	106.5(7)	106.1(3)	108.1(3)
O(4)–C(1)–C(11)	109.9(1)	110.4(2)	111.7(8)	111.7(8)	109.5(3)	108.5(3)
C(2)–C(1)–C(21)	109.6(1)	111.7(2)	111.7(8)	108.9(8)	110.0(3)	109.4(3)
C(2)–C(1)–C(11)	113.0(1)	109.6(2)	109.7(8)	112.4(8)	114.2(3)	113.0(3)
C(2)–C(1)–O(4)	108.5(1)	108.6(2)	108.9(7)	107.9(7)	108.8(2)	110.2(3)
C(1)–C(2)–O(5)	108.7(1)	109.0(1)	106.1(7)	109.4(8)	107.3(3)	110.5(3)
C(1)–C(2)–C(3)	113.4(1)	113.7(2)	112.7(8)	115.2(9)	113.0(3)	111.9(3)
C(3)–C(2)–O(5)	109.5(1)	106.2(2)	109.8(7)	105.8(7)	109.7(3)	105.6(3)
C(12)–C(11)–C(16)	118.4(1)	117.9(2)	118.7(9)	117.8(9)	118.7(3)	118.2(3)
C(22)–C(21)–C(26)	118.6(1)	117.8(2)	118.2(9)	118.7(9)	118.8(3)	118.6(3)
C(1)–C(11)–C(12)	124.2(1)	120.8(2)	121.2(9)	122.6(9)	123.6(3)	123.8(3)
C(1)–C(21)–C(22)	119.7(1)	122.0(2)	120.8(9)	120.4(9)	118.7(3)	120.3(3)
Torsion angles (°)						
C(2)–C(1)–C(21)–C(22)	−71.6(2)	−45.3(3)	−51.6(12)	−49.3(12)	−69.4(4)	−68.4(4)
C(2)–C(1)–C(11)–C(12)	5.6(2)	53.9(3)	47.3(12)	55.1(13)	14.3(5)	14.2(5)
O(4)–C(1)–C(2)–O(5)	−59.4(1)	−64.3(2)	−59.2(9)	−60.8(9)	−59.6(3)	−54.1(4)
O(4)–C(1)–C(2)–C(3)	62.7(2)	54.0(3)	61.0(10)	58.2(10)	61.4(4)	63.3(4)
C(11)–C(1)–C(2)–O(5)	61.8(1)	56.3(2)	63.4(9)	62.8(10)	63.0(4)	67.4(4)
C(11)–C(1)–C(2)–C(3)	−176.2(1)	174.6(2)	−176.5(8)	−178.2(8)	−176.0(3)	−175.3(3)
C(21)–C(1)–C(2)–O(5)	−176.6(1)	178.7(2)	−175.3(7)	−176.1(7)	−175.5(3)	−172.8(3)
C(21)–C(1)–C(2)–C(3)	−54.6(2)	−63.1(3)	−55.2(11)	−57.0(11)	−54.5(4)	−55.4(4)
C(11A)–C(1A)···C(1B)–C(11B)			−11.6(8)		14.2(3)	
C(11A)–C(1A)···C(1B)–O(4B)			−119.0(8)		−81.3(3)	
C(11A)–C(1A)···C(1B)–C(21B)			−172.7(12)		−154.5(4)	
C(11A)–C(1A)···C(1B)–C(2B)			97.5(8)		118.4(3)	

For all compounds, the OH groups are *gauche* and for all phenyl rings the *ipso* angle [C(*i*2)–C(*i*1)–C(*i*6); $i = 1, 2$] reflects the influence of the σ electron withdrawal of the substituent (Table 1). The weighted mean value of 118.4(3)° is in good agreement with the values reported by different techniques for toluene (117.5–118.7°) and the averaging X-ray data on C_6H_5–C(C)$_3$ moieties [117.4(1)°].[12]

The differences between molecules are mainly due to the conformation of the C(11)–C(16) phenyl ring. The almost co-planarity of the C(2) atom with that ring in **1** and **4** gives rise to angular distortion at C(1) and C(11) in such a way that the lower the C(2)–C(1)–C(11)–C(12) torsion angle is, the greater the C(2)–C(1)–C(11) and the C(1)–C(11)–C(12) angles. To a lesser extent, this type of deformation is also present in the C(21)–C(26) ring. The racemic and the S-configured hosts (**1** and **2**, respectively) are at both ends of the range. The increase in size of the guest molecule in **4** with respect to that of **3** (see below) could be responsible for these conformational changes since the guest (Fig 1) is allocated between the two independent C(11)–C(16) rings (A and B). In **1**, it could be attributed to the O–H···phenyl interaction (Table 3 and Fig 2).

The geometrical comparison between both crystallographically independent host molecules in compounds **3** and **4** has been performed by means of least-squares procedures[13] showing no differences in terms of the χ^2 values. However, these host molecules are differently oriented to each other as measured by the pseudo torsion angles around the C(1A)···C(1B) line (Table 1).

Table 2 Selected bond distances, bond angles and torsion angles for guest molecules in (a) 3-picoline in **3**, and (b) 3-methylcyclohexanone in **4**

	Bond distances (Å)		*Bond angles* (°)
(*a*)			
N(31)–C(32)	1.336(14)	C(32)–N(31)–C(36)	116.5(9)
C(32)–C(33)	1.386(15)	C(32)–C(33)–C(37)	119.7(12)
C(34)–C(35)	1.403(17)	C(34)–C(33)–C(37)	122.2(11)
C(33)–C(37)	1.533(21)	C(34)–C(35)–C(36)	118.3(11)
N(31)–C(36)	1.334(15)	N(31)–C(32)–C(33)	123.9(10)
C(33)–C(34)	1.344(18)	C(32)–C(33)–C(34)	118.1(10)
C(35)–C(36)	1.356(16)	C(33)–C(34)–C(35)	119.3(11)
		N(31)–C(36)–C(35)	123.7(11)
(*b*)			
C(31)–C(32)	1.490(7)	C(32)–C(31)–C(36)	116.4(5)
C(32)–C(33)	1.498(7)	C(32)–C(33)–C(34)	110.4(4)
C(34)–C(35)	1.517(8)	C(34)–C(35)–C(36)	111.4(5)
C(33)–C(38)	1.524(8)	C(36)–C(31)–O(37)	121.9(5)
C(31)–C(36)	1.497(9)	C(32)–C(33)–C(38)	111.0(5)
C(33)–C(34)	1.536(9)	C(31)–C(32)–C(33)	112.7(4)
C(35)–C(36)	1.505(9)	C(33)–C(34)–C(35)	111.9(5)
C(31)–O(37)	1.209(7)	C(31)–C(36)–C(35)	112.9(5)
		C(32)–C(31)–O(37)	121.7(5)
		C(34)–C(33)–C(38)	111.3(5)
	Torsion angles (°)		*Torsion angles* (°)
C(31)–C(32)–C(33)–C(34)	51.8(6)	C(32)–C(33)–C(34)–C(35)	−56.8(6)
C(33)–C(34)–C(35)–C(36)	55.3(7)	C(34)–C(35)–C(36)–C(31)	−48.8(7)
C(35)–C(36)–C(31)–C(32)	45.9(7)	C(36)–C(31)–C(32)–C(33)	−47.8(6)
C(31)–C(32)–C(33)–C(38)	175.6(5)	C(31)–C(32)–C(33)–H(33)	−68.(4)

The picoline guest molecule is planar within the achieved precision and the methyl group deviates 0.088(19) Å from it. As far as we know (Cambridge Structural Data Base, January 1992 release)[14] it is the first time that the geometry of the ketone molecule has been determined by X-ray diffraction (Table 3). The *R*-enantiomer present in the crystal displays a chair conformation slightly distorted towards a half-chair at C(31)–C(36). The Cremer and Pople parameters[15] are $q2 = 0.083(6)$ Å, $q3 = 0.510(6)$ Å, $\phi 2 = 166(4)^\circ$ and $\theta 2 = 9.2(6)^\circ$.

Crystal packing

Compound **1** contains molecules of both enantiomers by hydrogen bonding across symmetry centres producing dimers of enantiomeric molecules reinforced by OH···phenyl interactions within the dimer (Table 3). These dimers are linked along the $\langle -1\ 0\ 1\rangle$ direction by 'herringbone' or 'T' phenyl interactions,[16] and so forms chains of dimers along *b* through a 2-fold screw axis (Fig 2). These chains seem to be packed by isotropic interactions to finally build the whole crystal.

Compound **2** contains molecules of just one enantiomer (*S*). The strongest directional interactions are hydrogen bonds forming chains of molecules along the *b* direction through a 2-fold screw axis (Fig 3). These chains are packed, one with another, by phenyl–phenyl 'herringbone' interactions, again along *b* and through other 2-fold screw axes. This type of packing leaves one hydroxy group free of directional interaction; that of O(5). The torsion angles that describe the orientation of the hydrogen atom are C(1)–C(2)–O(5)–H(5) = 62(3) and C(2)–C(1)–O(4)–H(4) = −63(3)° compared with those for compound **1** of 91(2) and 81(2)°, respectively.

In the packing of compound **3**, all the hydroxy groups are involved in hydrogen bonds joining together the two independent molecules A and B (*S* configuration) and the 3-picoline molecule, while the intermolecular O(5A)–H(5A)···O(5B) contacts are responsible for chain formation along the *c* axis (Table 3 and Fig 4). These chains are packed in sheets by phenyl–phenyl herringbone interactions through a 2-fold screw axis parallel to the *c* axis. These sheets are packed together by the remaining herringbone interaction through another screw axis along the *b* direction, and isotropic interactions between methyl groups give rise to the whole crystal. Compound **4** roughly displays the same packing scheme. Although several interactions between phenyl rings remain unchanged, the hydrogen atoms involved in them are different (Table 3*c* and 3*d*). There are also two herringbone hydrogen interactions between the host

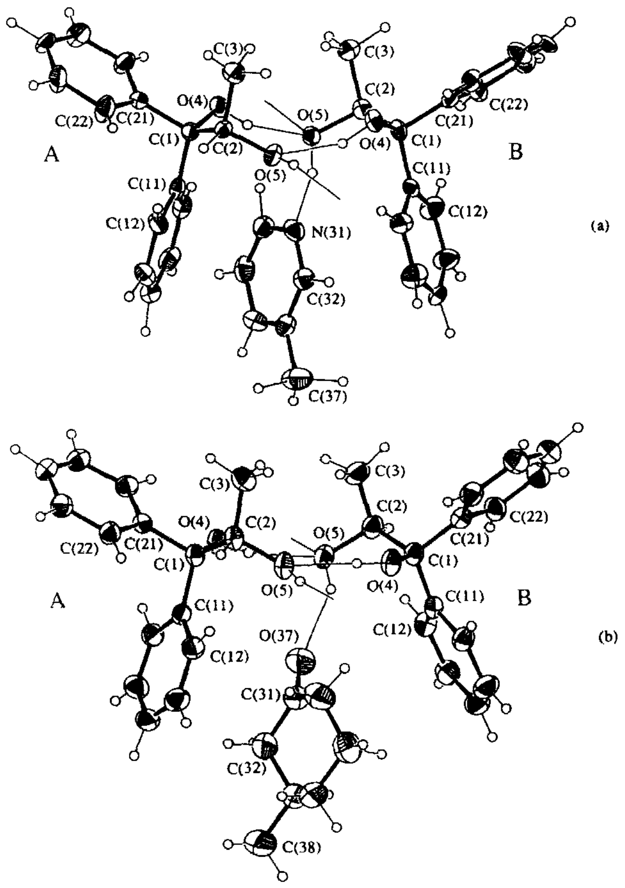

Figure 1 An Ortep[22] view of the asymmetric unit of compound 3 (a) and 4 (b) showing the hydrogen interactions (fine lines) and the atomic numbering. The same projection has been used for both compounds in order to show the influence of the guest on the host conformation. Ellipsoids are drawn at 30% probability level. The same numbering scheme was used for the remaining compounds.

matrix and the 3-picoline guest molecule inside the cavity.

Size and shape of cavities in the crystals

The location and characterization of cavities in the crystals have been carried out using a model of interpenetrating spheres of van der Waals radii. The resulting surface was smoothed by rolling a sphere of 1.4 Å radius.[17] Neither voids in **1** nor **2** could be found and both compounds displayed the same total packing coefficient of 0.67. The guest molecules in **3** and **4** (surface of 122.4, 138.0 Å^2 and volume of 95.9, 119.9 Å^3 respectively) were located in channels along the *c* axis of dimensions ±4.52, ±4.55, ±3.11 Å (Fig 5) and ±4.84, ±3.18, ±4.05 Å, respectively. The total $[C_k^{all} = (V_{host} + V_{guest})/\text{unit cell volume}]$ and local $(C_k^l = V_{guest}/V_{hole})$ packing coefficients in **4** (0.66, 0.51) are lower than those in **3** (0.68, 0.57), possibly due to the presence of an almost spherical void (12.4 Å^3) centred at 3.02, 3.24 Å of C(35) and C(34) atoms of the 3-methylcyclohexanone and at 3.24 Å of C(3A) $(x, y, z + 1)$.

The shapes of the channels and guests (circular cylinders and prolate spheroids) were estimated by means of the quotients (Q_{ij}^2) of the planar specific inertial moments of volume (i_{ij}^V) over the superficial ones (i_{ij}^s) with respect to the eigensystems.[17] $Q_{ij}^2 = 0.60$, 0.58, 0.79 for **3**; 0.58, 0.59, 0.87 for **4**; 0.53, 0.65, 0.72 for 3-picoline and 0.55, 0.65, 0.68 for 3-methylcyclohexanone. These values reveal the lack of rotational symmetry when compared with theoretical values (Fig 5).

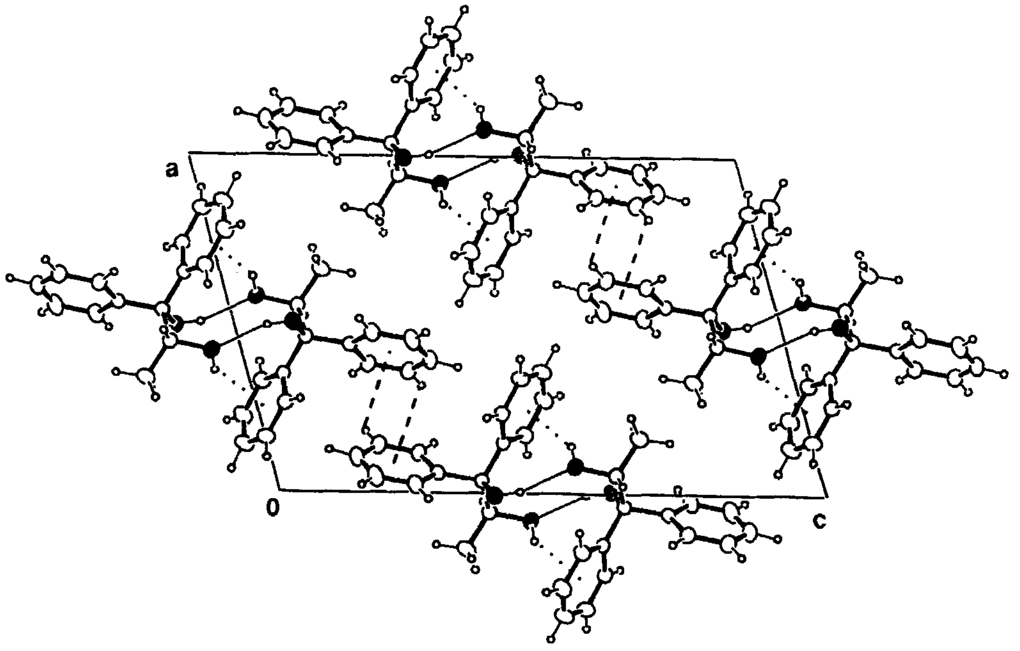

Figure 2 Crystal packing of compound **1** as projected along the *b* axis. The oxygens of the OH groups are shown as black.

Table 3 Hydrogen interactions

	X–H (Å)	*X···£ (Å)*	*H···£ (Å)*	*X–H···Y (°)*	*γ (°)*
(a) Compound 1					
O(4)–H(4)...O(5)$_1$	0.87(3)	2.922(1)	2.08(3)	162(2)	
O(5)–H(5)...C(11–16)$_1$	0.92(3)	3.363(1)	2.56(3)	147(2)	
C(23)–H(23)...C(21–26)$_2$	1.00(3)	3.752(2)	3.07(2)	126(2)	54.6(1)
1: $-x, -y, 1-z$	2: $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$				
(b) Compound 2					
O(4)–H(4)...O(5)$_1$	0.87(4)	2.835(2)	2.09(4)	144(4)	
C(23)–H(23)...C(11–16)$_2$	1.02(4)	3.689(3)	2.80(5)	164(4)	69.1(1)
C(25)–H(25)...C(21–26)$_3$	0.89(6)	3.989(4)	3.15(5)	159(3)	78.6(1)
1: $1-x, -\frac{1}{2}+y, 1-z$	2: $-x, \frac{1}{2}+y, -z$		3: $-x, \frac{1}{2}+y, 1-z$		
(c) Compound 3					
O(4A)–H(4A)...O(5B)	0.88(12)	2.805(9)	2.01(12)	149(12)	
O(5A)–H(5A)...O(5B)$_1$	0.82(14)	3.036(10)	2.23(15)	150(12)	
O(4B)–H(4B)...O(5A)	0.99(11)	2.938(8)	2.10(12)	141(10)	
O(5B)–H(5B)...N(31)	1.03(10)	2.747(10)	1.75(10)	161(9)	
C(13A)–H(13A)...C(11–16)A$_2$	1.09(9)	3.639(12)	2.88(9)	127(6)	45.8(3)
C(23B)–H(23B)...C(21–26)B$_3$	0.89(12)	3.824(13)	3.18(11)	131(8)	47.7(3)
C(14A)–H(14A)...C(21–26)A$_2$	0.99(8)	4.083(13)	3.31(8)	136(6)	86.7(3)
C(14B)–H(14B)...C(21–26)A$_4$	0.99(8)	4.008(12)	3.36(8)	125(6)	52.6(3)
C(32)–H(32)...C(11–16)B	1.05(11)	3.528(11)	2.71(10)	135(7)	57.5(3)
C(15A)–H(15A)...C(31–36)	1.02(12)	3.572(13)	2.94(10)	121(7)	47.8(3)
1: $x, y, -1+z$	2: $3/2-x, -y, -\frac{1}{2}+z$		3: $\frac{1}{2}-x, -y, \frac{1}{2}+z$		
4: $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$					
(d) Compound 4					
O(4A)–H(4A)...O(5B)	0.85(4)	2.936(3)	2.18(4)	148(5)	
O(5A)–H(5A)...O(5B)$_1$	0.92(8)	2.902(4)	2.00(8)	168(6)	
O(4B)–H(4B)...O(5A)	0.87(6)	2.886(3)	2.12(6)	146(6)	
O(5B)–H(5B)...O(37)	0.83(6)	2.725(5)	2.01(6)	143(5)	
C(15A)–H(15A)...C(11–16)A$_2$	1.02(5)	3.813(5)	3.18(5)	121(3)	75.1(1)
C(23B)–H(23B)...C(21–26)B$_3$	0.92(6)	3.753(5)	3.09(5)	131(4)	50.9(1)
C(14A)–H(14A)...C(21–26)A$_4$	0.97(5)	4.130(5)	3.25(5)	151(4)	75.1(1)
C(15B)–H(15B)...C(21–26)A$_5$	0.94(7)	4.023(5)	3.25(6)	140(5)	81.8(1)
C(32)–H(322)...C(11–16)A	1.05(6)	4.033(5)	3.16(6)	141(4)	
C(36)–H(361)...C(11–16)A	1.03(8)	3.998(6)	3.17(8)	139(6)	
1: $x, y, -1+z$	2: $3/2-x, -y, \frac{1}{2}+z$		3: $\frac{1}{2}-x, -y, \frac{1}{2}+z$		
4: $3/2-x, -y, -\frac{1}{2}+z$	5: $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$				

Subscripts stand for symmetry operations, C(11–16), C(21–26) and C(31–36) for the centroids of the corresponding rings and γ for the angle between aromatic rings.

EXPERIMENTAL

Sample preparation

Compounds **1** and **2** were synthesized as reported previously.[1] Single crystals of **1** and **2** suitable for X-ray diffraction were obtained by slow cooling of solutions of **1** and **2** in toluene. Crystals of inclusion compound *3* were prepared by slow cooling of a solution of **2** in 3-picoline.

X-ray structure determination

X-ray data for compounds **1**, **2** and **3** were recorded on crystals sealed in Lindemann capillaries. Crystal data and experimental details are given in table 4. The structures were determined by direct methods,[18] the non-hydrogen atoms were refined anisotropically and the hydrogen atoms were included as isotropic. Five, eight and three reflexions were affected by secondary extinction and were considered as unobserved in the

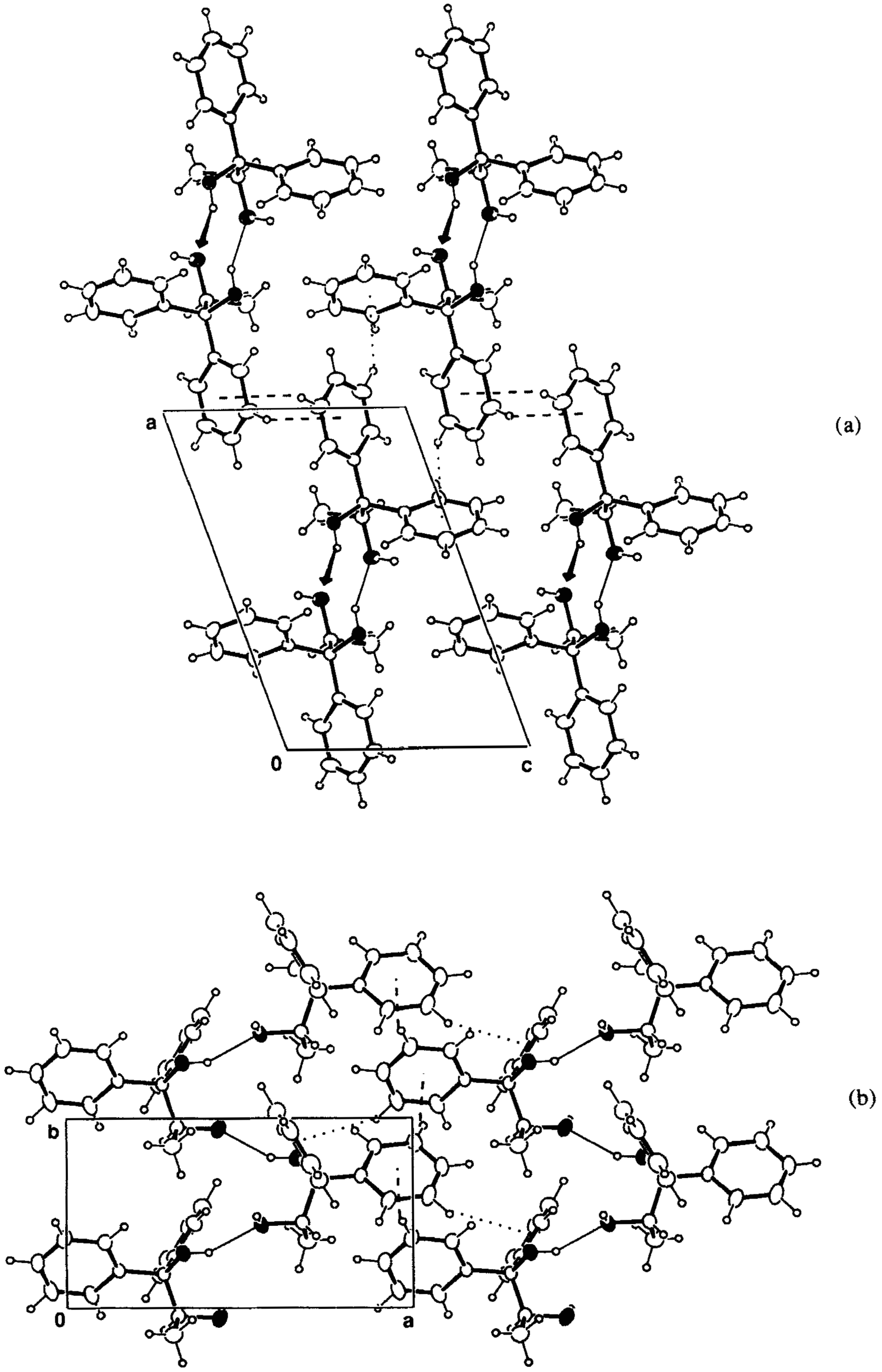

Figure 3 Crystal packing of compound **2** (a) along the *b* axis, (b) along the *c* axis, showing the infinite chains along the *b* axis. The oxygens of the OH groups are shown as black.

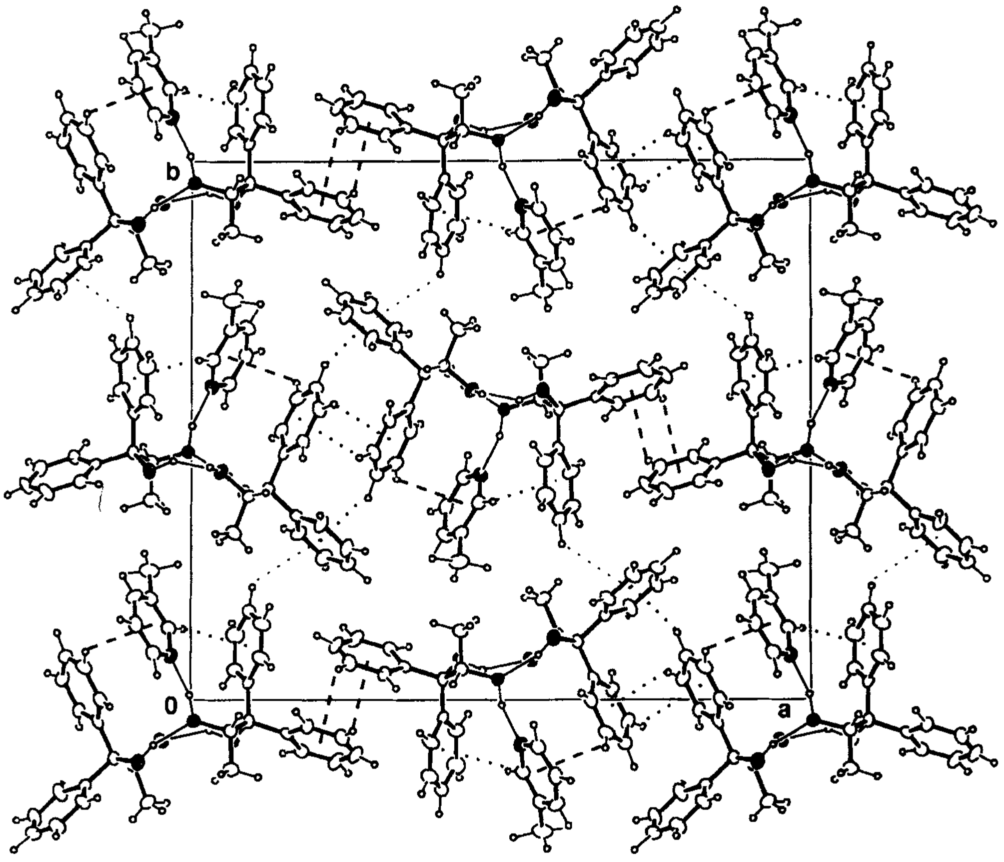

Figure 4 Crystal packing of compound **3** along the *c* axis. Atoms involved in O–H···O and O–H···N interactions are shown as black.

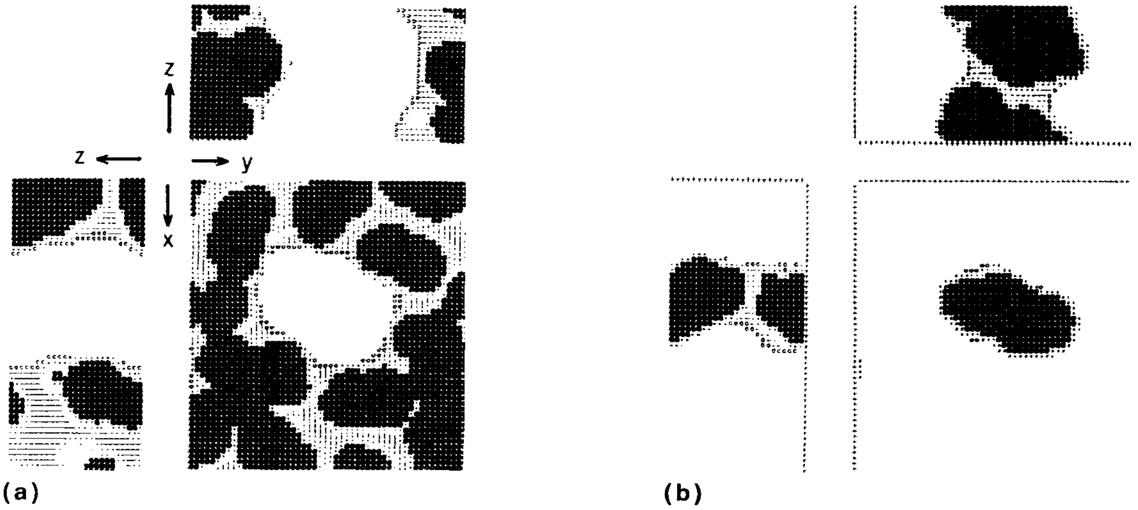

Figure 5 Three sections ($x = 0.4271$, $y = 0.3095$ and $z = 0.0$) through the centroids of the continuous channel (four in the unit cell) that hold the picoline molecule inside, (a) for the host, and (b) for the guest, molecules.

last cycles of refinement. The absolute structures were not determined (compounds **2** and **3**) since they were known by chemical synthesis. Most of the calculations were performed on a VAX6410 computer using an XRAY80 System.[19] The atomic scattering factors were taken from published tables.[20] Fractional atomic co-ordinates are given in Tables 5, 6 and 7.

The pseudoisomorphism of compounds **3** and **4** [replacement of the picoline by *R*-3-methylcyclohexanone: $P2_12_12_1$, $a = 25.0880(22)$, $b = 21.7182(17)$,

Table 4 Crystal analysis parameters at room temperature

	Compound		
	1	**2**	**3**
Crystal data			
Formula	$C_{15}H_{16}O_2$	$C_{15}H_{16}O_2$	$2(C_{15}H_{16}O_2)\cdot C_6H_7N$
Crystal habit	Colourless prism	Colourless prism	Colourless prism
Crystal size (mm)	0.40 × 0.33 × 0.26	0.50 × 0.50 × 0.33	0.66 × 0.17 × 0.10
Symmetry	Monoclinic, $P2_1/n$	Monoclinic, $P2_1$	Orthorhombic, $P2_12_12_1$
Unit cell determination	Least-squares fit from 77 reflexions ($\theta < 45°$)	Least-squares fit from 76 reflexions ($\theta < 45°$)	Least-squares fit from 76 reflexions ($\theta < 45°$)
Unit cell dimensions (Å^0)	$a = 15.0594(4)$ $b = 5.7940(1)$ $c = 18.5993(8)$ 90, 105.391(4), 90	$a = 12.3277(6)$ $b = 6.5165(3)$ $c = 8.1958(3)$ 90, 108.831(3), 90	$a = 23.9280(19)$ $b = 20.9375(12)$ $c = 5.9822(1)$ 90, 90, 90
Packing: V (Å^3), Z	1248.3(1), 4	623.16(5), 2	2997.0(3), 4
D_c (g/cm^3), M, $F(000)$	1.215, 228.29, 488	1.217, 228.29, 244	1.218, 549.71, 1176
μ (cm^{-1})	5.96	5.97	5.86
Experimental data			
Technique	Four circle diffractometer: Philips PW1100, bisecting geometry. Graphite oriented monochromator: CuK_α, $\omega/2\theta$ scans. Detector apertures 1 × 1°, $\theta_{max} = 65°$.		
Scan width (°)	1.5	1.4	1.4
Scan speed (min/reflex)	1	1	1.5
Number of reflexions			
Independent	2127	1165	2937
Observed	1960* (3σ(I) criterion)	1144* (3σ(I) criterion)	1880* (3σ(I) criterion)
Standard reflexions	2 reflexions every 90 min. No variation.		
Solution and refinement			
Solution	Direct Methods: SIR88		
Refinement			
Least-squares on F_0	Full matrix	Full matrix	2 blocks
Parameters			
Number of variables	218	217	526
Degrees of freedom	1742	927	1354
Ratio of freedom	8.9	5.3	3.6
H atoms	From difference synthesis.		
Weighting scheme	Empirical as to give no trends in $\langle \omega\Delta^2 F\rangle$ vs. $\langle \lvert F_{obs}\rvert \rangle$ and $\langle \sin\theta/\lambda \rangle$		
Max. thermal value (Å^2)	$U11[C(25)] = 0.091(1)$	$U22[C(14)] = 0.083(3)$	$U11[C(37)] = 0.103(12)$
Final ΔF peaks (eÅ^{-3})	±0.20	±0.28	±0.30
Final R and R_w	0.050, 0.050	0.047, 0.043	0.085, 0.092

* See experimental section.

Table 5 Final atomic co-ordinates for compound **1**

Atom	*x*	*y*	*z*
C(1)	0.03694(10)	−0.03718(21)	0.37585(7)
C(2)	−0.05607(11)	−0.22209(24)	0.37435(7)
C(3)	−0.16625(15)	−0.18262(40)	0.31325(10)
O(4)	−0.00614(8)	0.18159(16)	0.39173(6)
O(5)	−0.08113(9)	−0.22511(20)	0.44594(6)
C(11)	0.15077(10)	−0.08720(22)	0.43438(7)
C(12)	0.17299(13)	−0.28689(25)	0.47713(8)
C(13)	0.27780(15)	−0.31555(31)	0.53033(9)
C(14)	0.36113(14)	−0.14787(34)	0.54087(10)
C(15)	0.34047(13)	0.05064(31)	0.49819(9)
C(16)	0.23617(12)	0.08106(26)	0.44556(8)
C(21)	0.05968(11)	−0.02092(22)	0.29870(7)
C(22)	0.11766(13)	−0.19806(27)	0.27385(8)
C(23)	0.14000(15)	−0.18773(33)	0.20415(9)
C(24)	0.10492(16)	0.00006(34)	0.15874(8)
C(25)	0.04685(18)	0.17571(34)	0.18244(9)
C(26)	0.02313(15)	0.16646(28)	0.25217(8)

Table 6 Final atomic co-ordinates for compound **2**

Atom	*x*	*y*	*z*
C(1)	0.2793(2)	0.1808(5)	0.3173(2)
C(2)	0.3195(2)	−0.0457	0.3433(3)
C(3)	0.3058(3)	−0.1435(6)	0.5021(4)
O(4)	0.3367(1)	0.2921(5)	0.4700(2)
O(5)	0.4396(1)	−0.0545(5)	0.3642(2)
C(11)	0.3076(2)	0.2723(5)	0.1623(2)
C(12)	0.2706(2)	0.1759(6)	0.0016(3)
C(13)	0.2944(2)	0.2590(7)	−0.1381(3)
C(14)	0.3545(2)	0.4388(7)	−0.1224(3)
C(15)	0.3921(2)	0.5350(7)	0.0349(4)
C(16)	0.3686(2)	0.4523(6)	0.1769(3)
C(21)	0.1508(2)	0.2005(5)	0.2899(2)
C(22)	0.0706(2)	0.0713(7)	0.1795(4)
C(23)	−0.0456(2)	0.0935(7)	0.1571(4)
C(24)	−0.0827(2)	0.2429(7)	0.2430(3)
C(25)	−0.0054(2)	0.3711(7)	0.3507(4)
C(26)	0.1113(2)	0.3512(6)	0.3746(3)

Table 7 Final atomic co-ordinates for compound **3**

Atom	x	y	z
C(1A)	0.6238(4)	0.1070(4)	0.1786(18)
C(2A)	0.5925(4)	0.1204(4)	−0.0426(17)
C(3A)	0.5689(5)	0.1872(5)	−0.0532(25)
O(4A)	0.5856(3)	0.1138(3)	0.3607(12)
O(5A)	0.5477(2)	0.0744(3)	−0.0534(13)
C(11A)	0.6499(4)	0.0397(5)	0.1712(18)
C(12A)	0.6800(4)	0.0190(4)	−0.0131(20)
C(13A)	0.7058(4)	−0.0399(6)	−0.0145(20)
C(14A)	0.7009(4)	−0.0798(5)	0.1646(25)
C(15A)	0.6712(5)	−0.0605(5)	0.3506(22)
C(16A)	0.6454(4)	−0.0004(5)	0.3549(20)
C(21A)	0.6700(4)	0.1564(4)	0.2212(18)
C(22A)	0.7100(5)	0.1701(6)	0.0568(21)
C(23A)	0.7512(4)	0.2158(6)	0.1008(22)
C(24A)	0.7533(5)	0.2455(5)	0.3050(27)
C(25A)	0.7143(5)	0.2326(5)	0.4596(21)
C(26A)	0.6733(5)	0.1880(5)	0.4190(20)
C(1B)	0.4065(4)	0.0388(5)	0.2952(17)
C(2B)	0.4362(4)	0.0566(4)	0.5160(19)
C(3B)	0.4355(4)	0.1273(4)	0.5738(18)
O(4B)	0.4327(2)	0.0736(3)	0.1183(11)
O(5B)	0.4951(2)	0.0388(3)	0.5018(13)
C(11B)	0.4071(4)	−0.0327(5)	0.2511(16)
C(12B)	0.3868(5)	−0.0769(5)	0.4037(21)
C(13B)	0.3849(5)	−0.1400(5)	0.3571(24)
C(14B)	0.4049(4)	−0.1637(5)	0.1547(23)
C(15B)	0.4279(5)	−0.1199(6)	0.0015(22)
C(16B)	0.4286(4)	−0.0557(5)	0.0501(20)
C(21B)	0.3452(4)	0.0622(4)	0.3058(17)
C(22B)	0.3121(4)	0.0484(5)	0.4913(22)
C(23B)	0.2565(4)	0.0688(6)	0.4943(25)
C(24B)	0.2346(4)	0.1009(6)	0.3128(27)
C(25B)	0.2662(5)	0.1126(6)	0.1305(22)
C(26B)	0.3216(4)	0.0935(4)	0.1295(19)
N(31)	0.5328(3)	−0.0819(4)	0.6005(17)
C(32)	0.5355(4)	−0.1323(5)	0.4643(20)
C(33)	0.5642(4)	−0.1880(5)	0.5141(21)
C(34)	0.5904(5)	−0.1919(5)	0.7123(22)
C(35)	0.5883(4)	−0.1397(6)	0.8594(21)
C(36)	0.5602(4)	−0.0867(5)	0.7938(20)
C(37)	0.5673(8)	−0.2417(7)	0.3402(33)

$c = 5.8775(2)$ Å] has been checked by means of full normal probability plots.[21] When comparing the 2930 common observed structure factors, an almost linear plot is obtained (correlation coefficient $\rho = 0.980$ and $\chi^2/N = 0.57$). However, if the atomic fractional co-ordinates for the non-hydrogen atoms are compared, differences in the range 36–178 times its pooled standard deviation correspond to the z co-ordinate of the phenyl rings of molecule B, all OH groups and to the x and y co-ordinates of the C(11)–C(16) rings ($\rho = 0.989$ and $\chi^2/N = 8.60$).

Supplementary material available

Thermal factors for the non-hydrogen atoms, hydrogen parameters; bond lengths and angles and Tables of structure factors. Ordering information is given on any current masthead page.

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