

## Inclusion Compounds of *cis*- and *trans*-3,3-Bis(4-methylphenyl)cyclopropane-1,2-dicarboxylic Acid Hosts with Ethanol Guests.† Crystal Structures and Selective Inclusion Modes

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Structures of the crystalline inclusion complexes of *cis*- and *trans*-3,3-bis(4-methylphenyl)cyclopropane-1,2-dicarboxylic acid hosts, **1** and **2**, have been studied by X-ray diffraction. Crystal data: **1**·EtOH (1:1), monoclinic,  $C2/c$ ,  $a = 21.9870(7)$ ,  $b = 10.7398(3)$ ,  $c = 18.2478(4)$  Å,  $\beta = 110.46(2)^\circ$ ,  $Z = 8$ ,  $R = 0.051$  for 2085 reflections collected at room temperature. **2**·EtOH (1:2), monoclinic,  $P2_1/c$ ,  $a = 14.619(2)$ ,  $b = 11.032(2)$ ,  $c = 15.595(3)$  Å,  $\beta = 116.37(1)^\circ$ ,  $Z = 4$ ,  $R = 0.052$  for 1881 reflections collected at 173(1) K. The molecular geometries of **1** and **2** are rather similar, but **1** behaves as a monofunctional host owing to the intramolecular hydrogen bond between the two carboxyl groups, whereas **2** is a typical bifunctional host, as revealed by the different host:guest stoichiometries. Both hosts form endless H-bonded chains with ethanol guests, but the chains are differently organized: **1** creates supramolecular helices with alcoholic guests, whereas the H-bond scheme in **2**·EtOH (1:2) is characterized by closed loops of hydrogen bonds.

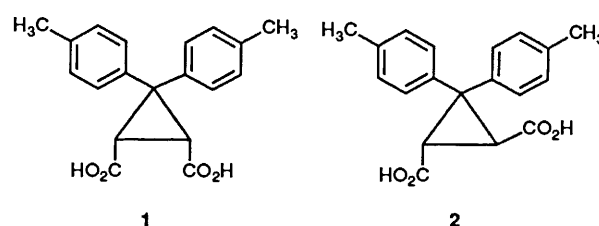
Small-ring compounds, *i.e.* molecules with a structure based on a three- or four-membered ring element with various substituents attached, proved to be a propitious source of clathrate hosts forming numerous crystalline inclusions of varying character.<sup>1</sup> We have seen that variation of the substituents and/or the functional groups around the central small-ring elements leads to altered clathrate forming ability.<sup>2</sup> Nevertheless, geometric parameters of the host molecule, such as the position and/or orientation of the functional groups are certainly important, but they have not been scrutinized systematically.

A previous X-ray study<sup>1</sup> on the crystalline inclusion compound between *cis*-3,3-diphenylcyclopropane-1,2-dicarboxylic acid (**1**) and Bu'OH (1:1) revealed formation of hydrogen-bonded helices with alternating host and guest molecules, including a characteristic intramolecular H bond between the *cis*-positioned carboxylic groups. All helices have the same sense so as to yield the chiral space group  $P2_12_12_1$ . In order to learn whether this behaviour is unique for *cis*-cyclopropane-1,2-dicarboxylic acids, we wanted to carry out further structural studies involving this host type and alcohol guests.

In contrast, the alcohol inclusions of the *trans*-cyclopropane-1,2-dicarboxylic acid host (**2**) typically give a 1:2 host:guest ratio instead of the 1:1 *cis* dicarboxylic acid stoichiometry.<sup>1,2</sup> This points both to a different mode of binding between host and guest and to a different packing in the crystal. We have therefore determined the crystal structures of EtOH inclusion compounds of *cis*- and *trans*-isomers of 3,3-bis(4-methylphenyl)cyclopropane-1,2-dicarboxylic acid, **1** and **2**, namely **1**·EtOH (1:1) and **2**·EtOH (1:2) using X-ray diffraction.

### Results and Discussion

Figs. 1(a) and 1(b) show perspective views of the stoichiometric



units of **1**·EtOH (1:1) and **2**·EtOH (1:2), respectively, with the crystallographic labelling of the atoms. Fig. 2 gives a specification of the hydrogen bond host-guest contacts and Fig. 3. comprises stereo packing diagrams. Refined atomic coordinates of the non-hydrogen atoms, dihedral angles between the least-squares planes and the geometry of the hydrogen bonds are listed in Tables 1–3.

The compounds **1**·EtOH (1:1) and **2**·EtOH (1:2) are based on the same fundamental components. Analogous bond lengths and bond angles involving the non-hydrogen atoms in the hosts **1** and **2** agree well with each other and also with those of previously published related molecules.<sup>1</sup> The molecular geometries are similar too; only the attachment of the carboxyl groups in the *trans*-isomer differs significantly from that of related *cis*-cyclopropanedicarboxylic acids (*cf.* Table 2). The geometries of the guest molecules, however, show larger deviations from the expected ones [especially in **1**·EtOH (1:1) studied at room temperature], most likely depending on the relatively high thermal mobility (or disorder) of the alcoholic carbon atoms, as compared with the other non-hydrogen atoms of the structures.

Nevertheless, the *cis*-positioning of the two  $-CO_2H$  groups in **1** makes possible a rather short intramolecular hydrogen bond between them (Table 3). As a consequence, **1** behaves as a monovalent host, generally forming inclusions with 1:1 host:guest stoichiometry. In contrast, the *trans*-cyclopropanedicarboxylic acid, **2**, is bivalent, usually giving rise to clathrate compounds with 1:2 stoichiometry.

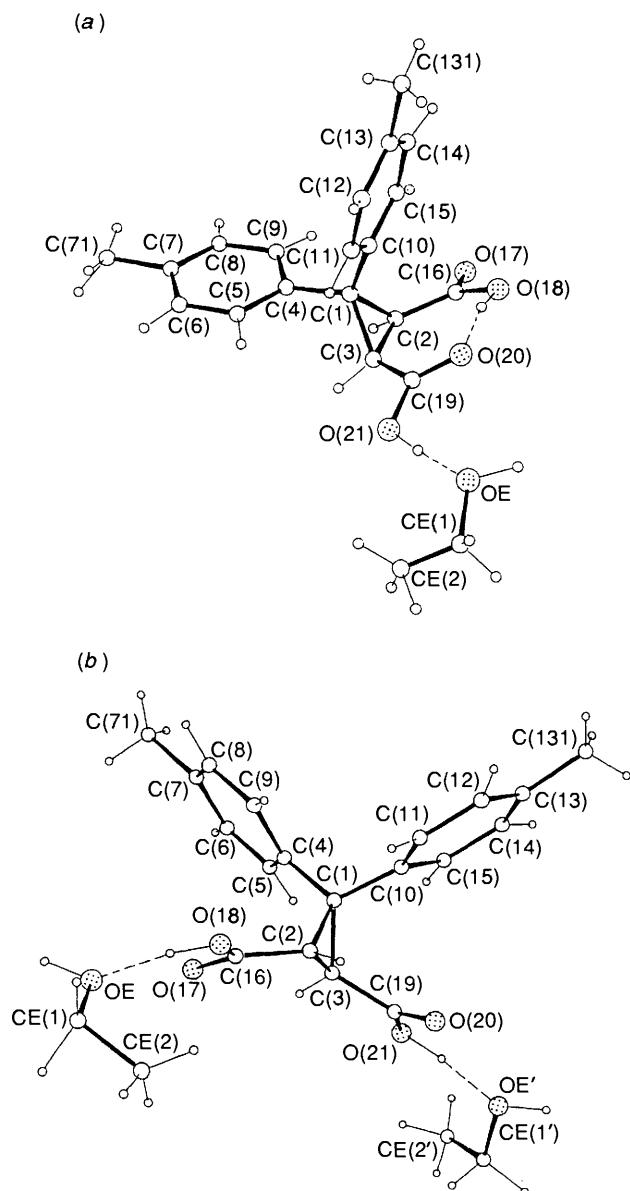
The hydrogen bonding schemes are also different (Fig. 2). In **1**·EtOH (1:1) infinite helical chains are created, resembling

† Small-Ring Inclusion Hosts. Part 5. For Part 4 of this series see I. Csöreg, O. Gallardo, E. Weber, M. Hecker and A. Wierig, *J. Incl. Phenom.*, in the press.

**Table 1** Fractional atomic coordinates of the non-hydrogen atoms in **1**·EtOH (1:1) and **2**·EtOH (1:2) with esds in parentheses

Atom	x/a	y/b	z/c
<b>1</b> ·EtOH (1:1)			
C(1)	0.3102(2)	-0.0586(3)	0.3577(2)
C(2)	0.2438(2)	-0.0279(3)	0.2984(2)
C(3)	0.3037(2)	-0.0134(3)	0.2758(2)
C(4)	0.3249(2)	-0.1945(3)	0.3747(2)
C(5)	0.3783(2)	-0.2491(3)	0.3648(2)
C(6)	0.3930(2)	-0.3725(4)	0.3847(2)
C(7)	0.3560(3)	-0.4455(3)	0.4140(3)
C(71)	0.3727(4)	-0.5796(4)	0.4381(4)
C(8)	0.3032(3)	-0.3907(4)	0.4224(3)
C(9)	0.2865(2)	-0.2661(3)	0.4037(2)
C(10)	0.3413(2)	0.0230(3)	0.4282(2)
C(11)	0.4056(2)	0.0582(3)	0.4484(2)
C(12)	0.4368(2)	0.1205(3)	0.5182(2)
C(13)	0.4042(2)	0.1488(3)	0.5690(2)
C(131)	0.4384(2)	0.2130(5)	0.6467(3)
C(14)	0.3402(2)	0.1161(4)	0.5476(2)
C(15)	0.3094(2)	0.0535(3)	0.4787(2)
C(16)	0.2005(2)	0.0694(3)	0.3120(2)
O(17)	0.1490(2)	0.0387(3)	0.3162(2)
O(18)	0.2182(1)	0.1870(2)	0.3215(3)
C(19)	0.3310(2)	0.1063(3)	0.2634(2)
O(20)	0.3204(1)	0.2069(2)	0.2877(1)
O(21)	0.3696(1)	0.0950(2)	0.2234(2)
O(E)	0.4032(2)	0.3063(3)	0.1835(2)
C(E1)	0.4348(4)	0.3207(8)	0.1304(4)
C(E2)	0.4579(4)	0.2137(9)	0.1106(5)
<b>2</b> ·EtOH (1:2)			
C(1)	0.8841(3)	0.3479(3)	0.7255(3)
C(2)	0.8325(3)	0.2315(4)	0.7335(3)
C(3)	0.7919(3)	0.3534(4)	0.7458(3)
C(4)	0.9861(3)	0.3832(4)	0.8059(3)
C(5)	0.9959(3)	0.4793(4)	0.8651(3)
C(6)	1.0908(3)	0.5123(4)	0.9368(3)
C(7)	1.1778(3)	0.4493(4)	0.9520(3)
C(71)	1.2821(3)	0.4824(5)	1.0328(3)
C(8)	1.1675(3)	0.3514(4)	0.8921(3)
C(9)	1.0733(3)	0.3192(4)	0.8199(3)
C(10)	0.8729(3)	0.3815(4)	0.6277(3)
C(11)	0.8892(3)	0.2990(4)	0.5692(3)
C(12)	0.8867(3)	0.3350(4)	0.4822(3)
C(13)	0.8701(3)	0.4554(4)	0.4537(3)
C(131)	0.8724(3)	0.4963(4)	0.3622(3)
C(14)	0.8526(3)	0.5381(4)	0.5120(3)
C(15)	0.8539(3)	0.5024(4)	0.5973(3)
C(16)	0.8814(3)	0.1546(4)	0.8198(3)
O(17)	0.9027(2)	0.1883(3)	0.9007(2)
O(18)	0.8991(2)	0.0437(3)	0.7987(2)
C(19)	0.6913(3)	0.3936(4)	0.6705(3)
O(20)	0.6442(2)	0.3434(3)	0.5945(2)
O(21)	0.6601(2)	0.4938(3)	0.6962(2)
O(E)	0.9438(2)	-0.1123(3)	0.9333(2)
C(E1)	0.8962(4)	-0.2266(5)	0.9136(4)
C(E2)	0.7907(4)	-0.2184(5)	0.8355(4)
O(E')	0.4813(3)	0.5671(4)	0.5804(3)
C(E1')	0.4194(5)	0.6091(7)	0.6252(4)
C(E2')	0.4781(6)	0.6912(5)	0.7022(6)

those formed by the non-methyl analogue of the *cis*-dicarboxylic acid host with Bu'OH as guest.<sup>1</sup> In these H-bonded helices host and guest molecules alternate [Fig. 2(a)]. It is worth noting that all helices have the same sense in the Bu'OH inclusion, so as to yield the chiral space group  $P2_12_12_1$ , whereas the crystals of **1**·EtOH are centrosymmetric ( $C2/c$ ) and contain supramolecular helices with opposite turns [Fig. 3(a)]. On the other hand, the host-guest interaction between the *trans*-dicarboxylic acid **2** and its ethanol guest is characterized by 12-membered (including the H atoms) closed H-bonded loops, each involving two host and two guest molecules [Fig. 2(b)]. Owing to the bifunctionality of the host and the *trans* configur-

**Fig. 1** Perspective views of the asymmetric structure units of (a) **1**·EtOH (1:1) and (b) **2**·EtOH (1:2), with crystallographic labelling of the atoms. Oxygen atoms are dotted. Solid and dashed lines represent covalent and hydrogen bonds, respectively.**Table 2** Dihedral angles (°) between different LS planes<sup>a</sup> calculated for different *cis*-cyclopropanedicarboxylic acids including **1**, and the *trans*-cyclopropanedicarboxylic acid **2**. Esds are given in parentheses.

Planes	Compounds			
	<b>1</b>	<b>2</b>	<b>3</b> <sup>b</sup>	<b>4</b> <sup>c</sup>
Cyclopropane/phenyl-1	56.4(2)	56.3(2)	71.8(1)	58.4(3)
Cyclopropane/phenyl-2	47.3(2)	51.8(2)	39.1(1)	50.8(3)
Cyclopropane/-COO(H)-1	80.1(3)	73.7(4)	88.6(4)	80.8(5)
Cyclopropane/-COO(H)-2	82.4(3)	74.2(4)	80.9(4)	80.0(4)
Phenyl-1/phenyl-2	76.8(1)	77.2(2)	72.8(2)	70.8(2)
-COO(H)-1/-COO(H)-2	24.8(3)	36.0(5)	23.8(5)	24.6(5)

<sup>a</sup> Least-squares planes, atomic deviations from the planes and dihedral angles between the planes were calculated according to Nardelli *et al.*<sup>13</sup> Details of the LS plane calculations are given in the Supplementary Material. <sup>b</sup> **3**: *cis*-3,3-Diphenylcyclopropanedicarboxylic acid (unsolvated). <sup>c</sup> **4**: *cis*-3,3-Diphenylcyclopropanedicarboxylic acid in its Bu'OH (1:1) inclusion complex.<sup>1</sup>

**Table 3** Bond distances (Å) and angles (°) in possible hydrogen bonds of 1·EtOH (1:1) and 2·EtOH (1:2) with standard deviations, where given,<sup>a</sup> in parentheses

Atoms involved	Symmetry	Distance/Å			Angle/°
		O...O	O-H	H...O	<O-H...O
<b>1·EtOH (1:1)</b>					
O(18)-H(18)...O(20)	<i>x, y, z</i>	2.537(4)	1.07	1.51	160
O(21)-H(21)...O(E)	<i>x, y, z</i>	2.569(4)	0.96	1.61	175
O(E)-H(OE)...O(17)	0.5 - <i>x</i> , 0.5 + <i>y</i> , 0.5 - <i>z</i>	2.749(4)	1.07	1.70	168
<b>2·EtOH (1:2)</b>					
O(18)-H(18)...O(E)	<i>x, y, z</i>	2.565(4)	0.96	1.63	162
O(21)-H(21)...O(E')	<i>x, y, z</i>	2.560(4)	0.99	1.60	163
O(E)-H(OE)...O(17)	2 - <i>x</i> , - <i>y</i> , 2 - <i>z</i>	2.698(3)	0.99	1.76	156
O(E')-H(OE')...O(20)	1 - <i>x</i> , 1 - <i>y</i> , 1 - <i>z</i>	2.708(4)	1.02	1.75	153

<sup>a</sup> H Atom positions are not refined (*cf.* the text).**Table 4** Crystal data and details of the data reduction and structure refinement calculations. Esds, where given, in parentheses.

	1·EtOH (1:1)	2·EtOH (1:2)
Formula	C <sub>19</sub> H <sub>24</sub> O <sub>4</sub> ·C <sub>2</sub> H <sub>6</sub> O	C <sub>19</sub> H <sub>24</sub> O <sub>4</sub> ·2(C <sub>2</sub> H <sub>6</sub> O)
<i>M<sub>w</sub></i>	356.4	402.5
Crystal system	monoclinic	monoclinic
Space group	<i>C</i> 2/ <i>c</i>	<i>P</i> 2 <sub>1</sub> / <i>c</i>
<i>a</i> /Å	21.9870(7)	14.619(2)
<i>b</i> /Å	10.7398(3)	11.032(2)
<i>c</i> /Å	18.2478(4)	15.595(3)
β/°	110.46(2)	116.37(1)
<i>V<sub>calc</sub></i> /Å <sup>3</sup>	4037.1(2)	2253.4(7)
<i>Z</i>	8	4
<i>D<sub>c</sub></i> /g cm <sup>-3</sup>	1.1728(1)	1.1864(4)
<i>F</i> (000)	1520	864
Crystal size/mm	0.28 × 0.32 × 0.40	0.20 × 0.27 × 0.30
<i>T</i> /K	291(1)	173(1)
Radiation (λ/Å)	Cu-Kα/1.541 83	Mo-Kα/0.710 69
μ <sub>c</sub> /cm <sup>-1</sup>	6.44	0.793
Scan type	ω-2θ	ω-2θ
Range of 2θ/°	3-120	3-60
Range of <i>h, k, l</i>	0→24, 0→12, -20→20	-20→20, 0→15, 0→21
No. of collected reflections	3286	6905
No. of standard reflections	4	5
Time interval between the standards/min	60	60
Intensity instability	<3%	<1%
<i>R<sub>int</sub></i>	0.017	—
No. of unique non-zero reflections	2810	4979
No. of significantly observed reflections	2114	1881
Criterion of significance	<i>I</i> /σ( <i>I</i> ) > 3	<i>I</i> /σ( <i>I</i> ) > 2
No. of refined parameters	256	279
Final agreement factors:		
<i>R</i> = Σ Δ <i>F</i>  /Σ  <i>F<sub>o</sub></i>	0.052	0.052
<i>wR</i> = [Σ <i>w</i>  Δ <i>F</i>   <sup>2</sup> /Σ <i>w</i>   <i>F<sub>o</sub></i>   <sup>2</sup> ] <sup>1/2</sup>	0.074	0.053
<i>wR<sub>tot</sub></i>	0.080	0.065
Weighting: <i>w</i> = [σ <sup>2</sup> ( <i>F</i> ) + <i>g</i> · <i>F</i> <sup>2</sup> ] <sup>-1</sup> with <i>g</i> =	0.000 25	0.000 34
Final Δρ <sub>max</sub> /Δρ <sub>min</sub> [e <sup>-</sup> Å <sup>-3</sup> ]	0.35/-0.17	0.29/-0.22

ation of the -CO<sub>2</sub>H groups, the H-bonded rings link the molecules into infinite chains [Fig. 3(b)]. Thus, the tendency to form extended, closed loops *via* hydrogen-bond interactions between hosts and guests is retained by host **2**; this basic structural pattern for recognition of alcohols by carboxylic acids has previously been observed with 1,1'-binaphthyl-2,2'-dicarboxylic acid,<sup>3,4</sup> *trans*-9,10-dihydro-9,10-ethanoanthracene-11,12-dicarboxylic acid,<sup>4,5</sup> and 9-phenylfluorene-9-carboxylic acid.<sup>6</sup>

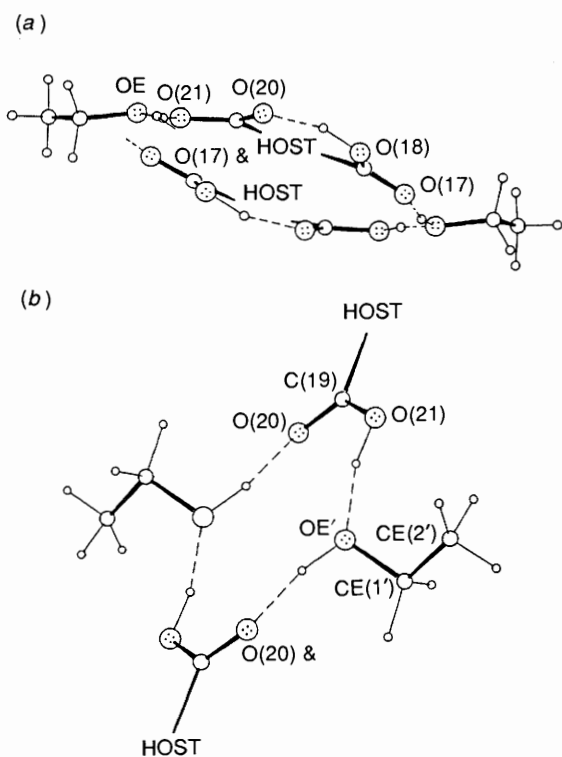
In conclusion, this study demonstrates the crucial effect of the host geometry on the inclusion properties. Although hosts **1** and **2** differ only in the relative orientation of the two coordinative, highly polar carboxyl groups, they have different guest selectivities,<sup>1,2</sup> and with the same guest they frequently yield different host-guest stoichiometries and consequently different packing relations (Fig. 3). Knowing the regular modes

of interaction and characteristic structures of active groups in different geometric environments is thus crucial in the engineering of host-guest crystals.<sup>7,8</sup>

## Experimental

**Sample Preparation.**—The host compounds **1** and **2**, synthesized as described previously,<sup>1</sup> were dissolved in a minimum amount of ethanol under heating, followed by slow cooling to room temperature. The crystals of the clathrates were grown by slow evaporation.

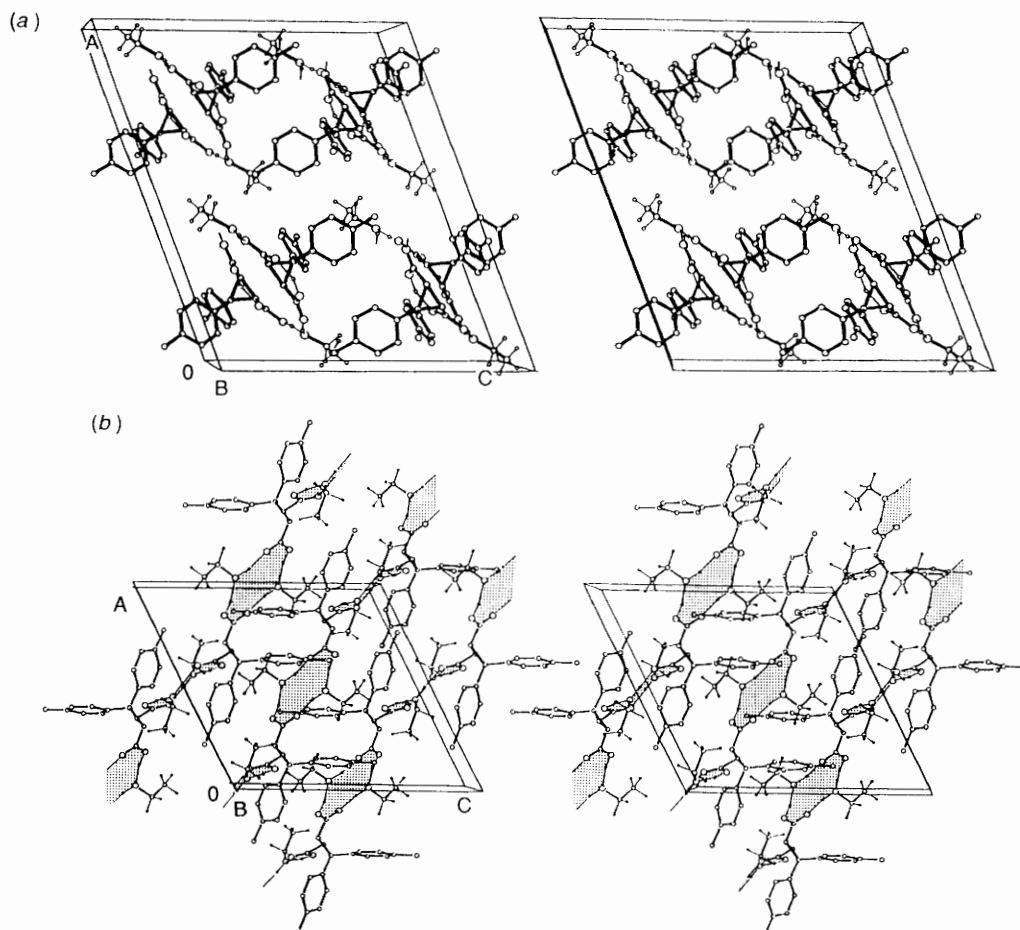
**X-Ray Structure Determinations.**—Intensity data were collected on a Siemens/AED2 diffractometer and corrected for background, Lorentz and polarization, but not for absorption and extinction effects. In the least-squares refinement of the unit



**Fig. 2** Host-guest hydrogen bonding schemes of (a) 1-EtOH (1:1) and (b) 2-EtOH (1:2). Oxygen atoms are dotted. Solid and dashed lines represent covalent and hydrogen bonds, respectively.

**Table 5** Intramolecular bond distances/Å between the non-hydrogen atoms of 1-EtOH (1:1) and 2-EtOH (1:2) with standard deviations in units of the last significant figure in parentheses

Atoms	Distance	Atoms	Distance
<b>1-EtOH (1:1)</b>		<b>2-EtOH (1:2)</b>	
C(1)-C(2)	1.519(4)	C(1)-C(2)	1.522(6)
C(1)-C(3)	1.531(4)	C(1)-C(3)	1.517(6)
C(1)-C(4)	1.504(4)	C(1)-C(4)	1.512(4)
C(1)-C(10)	1.508(4)	C(1)-C(10)	1.507(6)
C(2)-C(3)	1.520(5)	C(2)-C(3)	1.516(6)
C(2)-C(16)	1.493(5)	C(2)-C(16)	1.479(5)
C(3)-C(19)	1.469(5)	C(3)-C(19)	1.485(5)
C(4)-C(5)	1.379(5)	C(4)-C(5)	1.371(6)
C(4)-C(9)	1.376(6)	C(4)-C(9)	1.387(6)
C(5)-C(6)	1.383(5)	C(5)-C(6)	1.389(5)
C(6)-C(7)	1.366(7)	C(6)-C(7)	1.375(6)
C(7)-C(71)	1.513(6)	C(7)-C(71)	1.529(5)
C(7)-C(8)	1.358(8)	C(7)-C(8)	1.391(6)
C(8)-C(9)	1.398(5)	C(8)-C(9)	1.382(5)
C(10)-C(11)	1.383(4)	C(10)-C(11)	1.382(6)
C(10)-C(15)	1.380(5)	C(10)-C(15)	1.401(5)
C(11)-C(12)	1.389(4)	C(11)-C(12)	1.399(7)
C(12)-C(13)	1.390(5)	C(12)-C(13)	1.388(6)
C(13)-C(131)	1.519(5)	C(13)-C(131)	1.511(7)
C(13)-C(14)	1.368(5)	C(13)-C(14)	1.388(6)
C(14)-C(15)	1.377(4)	C(14)-C(15)	1.380(6)
C(16)-O(17)	1.206(5)	C(16)-O(17)	1.216(5)
C(16)-O(18)	1.315(4)	C(16)-O(18)	1.322(5)
C(19)-O(20)	1.222(4)	C(19)-O(20)	1.209(5)
C(19)-O(21)	1.302(5)	C(19)-O(21)	1.325(6)
O(E)-C(1E)	1.385(9)	O(E)-C(1E)	1.407(6)
C(1E)-C(2E)	1.355(13)	C(1E)-C(2E)	1.484(6)
		O(E')-C(E1')	1.443(9)
		C(E1')-C(E2')	1.444(9)



**Fig. 3** Stereo packing illustrations of (a) 1-EtOH (1:1) and (b) 2-EtOH (1:2) inclusion compounds. Thin lines represent hydrogen bonds. The carbon-bonded H atoms of the hosts are omitted for clarity. Hydrogen bonded ring systems in (b) are marked with dots.

**Table 6** Intramolecular bond angles/ $^{\circ}$  involving the non-hydrogen atoms of **1**-EtOH (1:1) and **2**-EtOH (1:2) with standard deviations in units of the last significant figure in parentheses

Atoms	Angle	Atoms	Angle
<b>1</b> -EtOH (1:1)			
C(4)–C(1)–C(10)	112.8(2)	C(7)–C(8)–C(9)	123.3(5)
C(3)–C(1)–C(10)	120.5(2)	C(4)–C(9)–C(8)	119.0(4)
C(3)–C(1)–C(4)	116.7(2)	C(1)–C(10)–C(15)	121.4(3)
C(2)–C(1)–C(10)	121.2(3)	C(1)–C(10)–C(11)	120.2(3)
C(2)–C(1)–C(4)	116.2(3)	C(11)–C(10)–C(15)	118.0(3)
C(2)–C(1)–C(3)	59.8(2)	C(10)–C(11)–C(12)	120.4(3)
C(1)–C(2)–C(16)	122.9(3)	C(11)–C(12)–C(13)	120.9(3)
C(1)–C(2)–C(3)	60.5(2)	C(12)–C(13)–C(14)	118.2(3)
C(3)–C(2)–C(16)	129.6(3)	C(12)–C(13)–C(131)	121.3(4)
C(1)–C(3)–C(2)	59.7(2)	C(131)–C(13)–C(14)	120.5(3)
C(2)–C(3)–C(19)	124.9(3)	C(13)–C(14)–C(15)	120.9(4)
C(1)–C(3)–C(19)	121.6(3)	C(10)–C(15)–C(14)	121.6(3)
C(1)–C(4)–C(9)	120.3(3)	C(2)–C(16)–O(18)	121.1(4)
C(1)–C(4)–C(5)	121.1(3)	C(2)–C(16)–O(17)	119.1(3)
C(5)–C(4)–C(9)	118.6(3)	O(17)–C(16)–O(18)	119.8(4)
C(4)–C(5)–C(6)	120.3(4)	C(3)–C(19)–O(21)	112.7(3)
C(5)–C(6)–C(7)	122.4(4)	C(3)–C(19)–O(20)	125.1(3)
C(6)–C(7)–C(8)	116.4(4)	O(20)–C(19)–O(21)	122.1(3)
C(6)–C(7)–C(71)	122.8(5)	O(E)–C(E1)–C(E2)	114.7(7)
C(71)–C(7)–C(8)	120.7(5)		
<b>2</b> -EtOH (1:2)			
C(4)–C(1)–C(10)	113.6(4)	C(7)–C(8)–C(9)	121.2(4)
C(3)–C(1)–C(10)	119.7(4)	C(4)–C(9)–C(8)	120.7(4)
C(3)–C(1)–C(4)	117.2(3)	C(1)–C(10)–C(15)	119.5(4)
C(2)–C(1)–C(10)	117.1(3)	C(1)–C(10)–C(11)	122.2(4)
C(2)–C(1)–C(4)	119.4(3)	C(11)–C(10)–C(15)	118.1(4)
C(2)–C(1)–C(3)	59.8(3)	C(10)–C(11)–C(12)	121.0(4)
C(1)–C(2)–C(16)	120.1(4)	C(11)–C(12)–C(13)	120.5(4)
C(1)–C(2)–C(3)	59.9(3)	C(12)–C(13)–C(14)	118.4(4)
C(3)–C(2)–C(16)	116.9(3)	C(12)–C(13)–C(131)	120.9(4)
C(1)–C(3)–C(2)	60.3(3)	C(131)–C(13)–C(14)	120.7(4)
C(2)–C(3)–C(19)	118.1(3)	C(13)–C(14)–C(15)	121.1(4)
C(1)–C(3)–C(19)	120.4(4)	C(10)–C(15)–C(14)	120.9(4)
C(1)–C(4)–C(9)	119.9(4)	C(2)–C(16)–O(18)	111.9(4)
C(1)–C(4)–C(5)	121.8(4)	C(2)–C(16)–O(17)	124.5(4)
C(5)–C(4)–C(9)	118.3(4)	O(17)–C(16)–O(18)	123.6(4)
C(4)–C(5)–C(6)	120.9(4)	C(3)–C(19)–O(21)	111.3(4)
C(5)–C(6)–C(7)	121.5(4)	C(3)–C(19)–O(20)	124.9(4)
C(6)–C(7)–C(8)	117.4(4)	O(20)–C(19)–O(21)	123.8(4)
C(6)–C(7)–C(71)	121.9(4)	O(E)–C(E1)–C(E2)	110.9(4)
C(71)–C(7)–C(8)	120.7(4)	O(E')–C(E1')–C(E2')	109.6(6)

cell dimensions, the  $\theta$  values of  $55$  ( $34^{\circ} < 2\theta < 62^{\circ}$ ) and  $30$  ( $17^{\circ} < 2\theta < 21^{\circ}$ ) carefully centred reflections were used for **1**-EtOH (1:1) and **2**-EtOH (1:2), respectively. Crystal data and details of the refinement calculations are listed in Table 4.

The structures were solved by direct methods (SHELXS)<sup>9</sup> and refined by full-matrix least-squares calculations based on  $|F|$  (SHELX).<sup>10</sup> The hydrogen atoms, except five H atoms of the EtOH guest in **1**-EtOH (1:1), were located from difference electron density calculations and were held riding on their parent atoms in the subsequent refinements. The carbon-bonded H atoms of the EtOH molecule in **1**-EtOH (1:1) were assumed to have geometrically predicted positions with C–H = 1.00 Å. In the last stage of the refinements the non-hydrogen atom positions were refined together with their anisotropic displacement parameters, and isotropic vibration parameters were refined for the hydrogen atoms. In the case of **1**-EtOH (1:1), 29 low- $\theta$  reflections with considerably lower  $|F_c|$  than

$|F_c|$ , in all probability due to extinction effects, were excluded from the last calculation, which yielded the final  $R$  and  $wR$  values shown in Table 4. The  $wR_{\text{int}}$  values were calculated for the final structural models using all unique non-zero reflections. The atomic scattering factors for the non-hydrogen atoms were taken from Cromer and Mann,<sup>11</sup> and those for the H atoms from Stewart *et al.*<sup>12</sup> Refined atomic coordinates of the non-hydrogen atoms are listed in Table 1, bond lengths in Table 5, and bond angles in Table 6.

*Supplementary Data.*—Fractional atomic coordinates and isotropic displacement parameters of the hydrogen atoms, bond lengths and bond angles involving the hydrogens, least-squares planes, deviations of the atoms from the planes and dihedral angles between the planes, and anisotropic and equivalent isotropic displacement parameters of the non-hydrogen atoms have been deposited as supplementary data at the Cambridge Crystallographic Data Centre.\* Lists of the observed and calculated structure factors are available directly from the authors (I.C.).

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\* For details of the deposition scheme see 'Instructions for Authors,' *J. Chem. Soc., Perkin Trans. 2*, 1992, issue 1.

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