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## Multiple Hydrogen-Bonding Modes in Furan-2,5-dimethanol and Furan-2,5-diylbis(diphenylmethanol)

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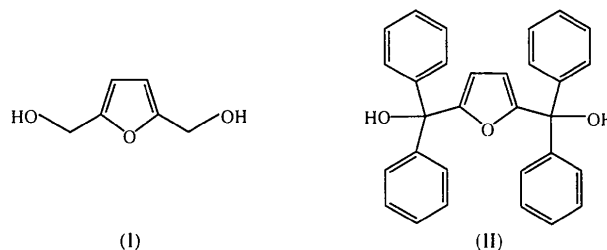
### Abstract

Molecules of furan-2,5-dimethanol, C<sub>6</sub>H<sub>8</sub>O<sub>3</sub>, are linked by O—H···O hydrogen bonds involving only the hydroxy O atoms. Different hydrogen-bonding motifs link the molecules into chains parallel to both the **a** and **b** directions, and thus into sheets normal to the **c** direction containing rhomboidal nets of hydrogen bonds. Molecules of furan-2,5-diylbis(diphenylmethanol), C<sub>30</sub>H<sub>24</sub>O<sub>3</sub>, are linked by multiple weak O—H···O hydrogen bonds to form hydrogen-bonded dimers lying about a twofold axis. (2-Furyl)diphenylmethanol, C<sub>17</sub>H<sub>14</sub>O<sub>2</sub>, is isomorphous with triphenylmethanol, Ph<sub>3</sub>COH.

### Comment

Organic diols of the type Ph<sub>2</sub>C(OH)—X—CPh<sub>2</sub>(OH), consisting of two Ph<sub>2</sub>C(OH) groups separated by a spacer *X* of generally limited flexibility, where *X* can be an aromatic group (Toda, Kai, Toyotaka, Yip & Mak, 1989), a heterocyclic group (Mazaki, Hayashi & Kobayashi, 1992), a poly '-yne' (Bourne, Johnson, Marais, Nassimbeni, Weber, Skobridis & Toda, 1991; Johnson, Nassimbeni & Toda, 1992) or a ferrocene group (Ferguson, Gallagher, Glidewell & Zakaria, 1993; Glidewell, Ferguson, Lough & Zakaria, 1994), have been found to be effective hosts for the capture of a wide range of guest molecules. In all the examples hitherto studied, the spacer *X* contains no functionality observed to act as a hydrogen-bond donor or acceptor. We have now developed this type of diol to produce a system in which the spacer *X* is a furyl group, which can act as a hydrogen-bond acceptor. In functionalized furans containing hydroxy or carboxy groups, the furan O atom sometimes plays a role in the hydrogen-bonding scheme, as in furan-3,4-dicarboxylic acid (Semmingsen, Nordenson & Aasen, 1986). In other similar compounds, this O atom takes no part in the hydrogen bonding, as in furan-2,5-dicarboxylic acid (Martuscelli & Pedone, 1968) and ferrocenyl(2-furyl)phenylmethanol (Ferguson,

Glidewell, Opromolla, Zakaria & Zanello, 1995). As part of a study of the hydrogen-bonding patterns in alcohols and diols, we now report the structures of furan-2,5-dimethanol, HOCH<sub>2</sub>(2,5-C<sub>4</sub>H<sub>2</sub>O)CH<sub>2</sub>OH, (I), and furan-2,5-diylbis(diphenylmethanol), HOCPh<sub>2</sub>(2,5-C<sub>4</sub>H<sub>2</sub>O)CPh<sub>2</sub>OH, (II).



Compound (I) crystallizes in the orthorhombic space group *P2<sub>1</sub>cn*, with one molecule in the asymmetric unit. Although no crystallographic symmetry is imposed, the molecules nonetheless exhibit almost perfect *m* (*C<sub>s</sub>*) symmetry across a plane normal to the furan ring and running through the O atom and the C3—C4 bond (Fig. 1). Of all the pairs of heavy-atom structural parameters related by this notional plane, only the exocyclic O—C—C angles and O—C—C—O torsion angles (Table 2) show any differences that are significant, and even these differences are small. The bond lengths within the furan ring are not significantly different from those observed in the ordered low-temperature phase of furan itself (Fourme, 1972).

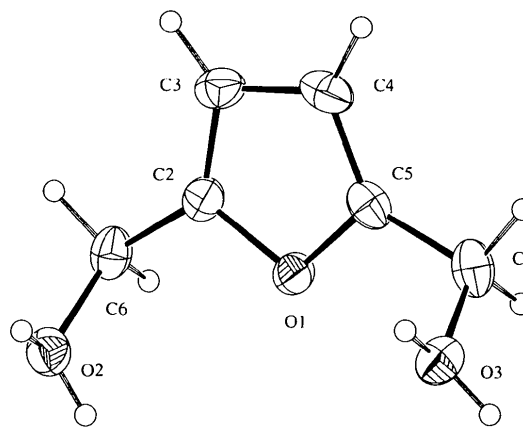


Fig. 1. A view of compound (I) with the atomic numbering scheme. Displacement ellipsoids are at the 30% probability level.

The hydroxy H atoms are both disordered, with site occupancies estimated from difference maps to be 0.62 for H2A and H3A, and 0.38 for H2B and H3B. All four sites take part in O—H···O hydrogen bonds, but these involve only the hydroxy O atoms. The furan O atom plays no role in the hydrogen-

bonding scheme, in contrast to the furan O atom in compound (II) (see below). Because of the disorder, it is convenient to consider major and minor contributions to the hydrogen-bonding network. The hydrogen bonds (Table 3), whether arising from the major or minor hydroxy H-atom sites, serve to link the molecules into chains parallel to the **a** direction. If in a given O···O interaction, the H2A and H3B (or H3A and H2B) sites are both occupied, an (OH)<sub>2</sub> ring results, with graph set *DD* since the two hydrogen bonds are non-equivalent, rather than the graph set *R*<sub>2</sub><sup>2</sup>(4) normally found when the two hydrogen bonds in such a ring are symmetry equivalent (Etter, 1990; Etter, MacDonald & Bernstein, 1990; Bernstein, Davis, Shimoni & Chang, 1995). If just one of the systems, major or minor, is considered, the unitary graph set for the hydrogen-bonding motif forming the chains parallel to **a** is *DD*, while the binary graph set for this motif is *C*<sub>2</sub><sup>2</sup>(4).

In addition to chains parallel to **a** (Fig. 2), there are also hydrogen-bonded chains parallel to the **b** direction, having graph set *C*<sub>2</sub><sup>2</sup>(16) and generated by the molecule at (*x*, *y*, *z*) acting as a double hydrogen-bond donor to the molecules at ( $\frac{1}{2} + x$ ,  $\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ) and ( $-\frac{1}{2} + x$ ,  $-\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ), while the molecule at ( $-\frac{1}{2} + x$ ,  $\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ) acts as a double hydrogen-bond acceptor from the molecules at (*x*, *y*, *z*) and (*x*, *1* + *y*, *z*), and so on. Finally, the combination of the *C*<sub>2</sub><sup>2</sup>(4) and *C*<sub>2</sub><sup>2</sup>(16) chains parallel to **a** and **b**, respectively, leads to a two-dimensional rhomboidal net of hydrogen bonds. This may alternatively be envisaged as arising from the action of the *n*-glide plane; atom O3 of the molecule at (*x*, *y*, *z*) acts as donor to atom O2 of the molecule at ( $\frac{1}{2} + x$ ,

$\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ), while atom O3 of this second molecule acts as donor to atom O2 of the molecule at (*1* + *x*, *1* + *y*, *z*), generating chains parallel to **a** + **b** with graph set *C*(8). Similar *C*(8) chains, formed by atom O2 of the molecule at (*x*, *y*, *z*) acting as donor to atom O3 of the molecule at ( $\frac{1}{2} + x$ ,  $-\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ), and so on, run parallel to **a** - **b**.

There is, however, no development of any hydrogen-bonding in the **c** direction, as intermolecular contacts in this direction involve mainly C—H bonds. The structure of compound (I), therefore, comprises hydrogen-bonded sheets, normal to **c**, held together by van der Waals forces.

Compound (II) crystallizes in the orthorhombic space group *F*2*dd*, with one molecule in the asymmetric unit (Fig. 3). The dominant structural motif is a hydrogen-bonded dimer, comprising molecules at (*x*, *y*, *z*) and (*x*, -*y*, -*z*) packed around a twofold rotation axis and linked by a variety of weak hydrogen bonds. Within the asymmetric unit, the hydroxy H atom bonded to O2 is disordered equally over two sites, but only one of these sites (H2A) takes part in the hydrogen bonding. There are both intra- and intermolecular O—H···O hydrogen bonds. Of these, the intermolecular hydrogen bonds are characterized by the shorter H···O distances (Table 6), well within the sum of the van der Waals radii (Bondi, 1964; Baur, 1972). There are two weak intramolecular hydrogen bonds, involving O1—H1···O51 and O2—H2A···O51 interactions (Table 6), where the hydroxy groups both act as hydrogen-bond donors and the furan O atom acts as a hydrogen-bond acceptor. The graph set (Etter, 1990; Etter, MacDonald & Bernstein, 1990) for each of these interactions is *S*(5). In addition, there are intermolecular hydrogen bonds; atoms H1 and H2A of the molecule at (*x*, *y*, *z*) act as donors to atoms O2 and O51, respectively, of the molecule at (*x*, -*y*, -*z*). Both H1 and H2A are thus participants in three-centre hydrogen bonds (Taylor, Kennard & Versichel, 1984). Each of these intermolecular hydrogen bonds is weak (Table 6) and both have the graph set *D*. The overall unitary

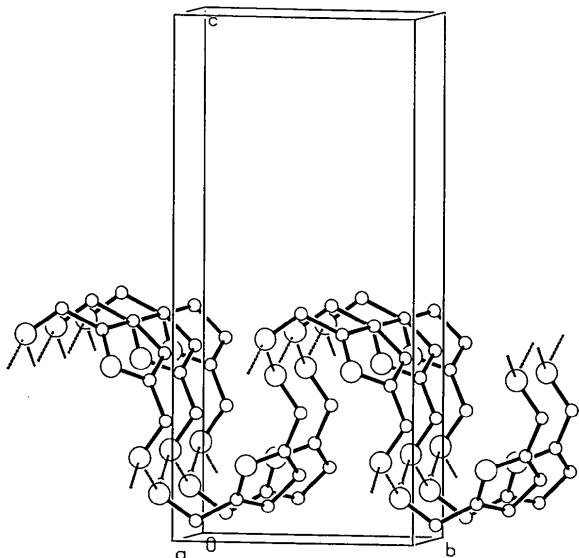


Fig. 2. A view of part of the crystal structure of compound (I), showing the part of the hydrogen-bonded sheet arising from the major components of the disordered hydroxy H2A and H3A atoms. H atoms bonded to C atoms are omitted for clarity.

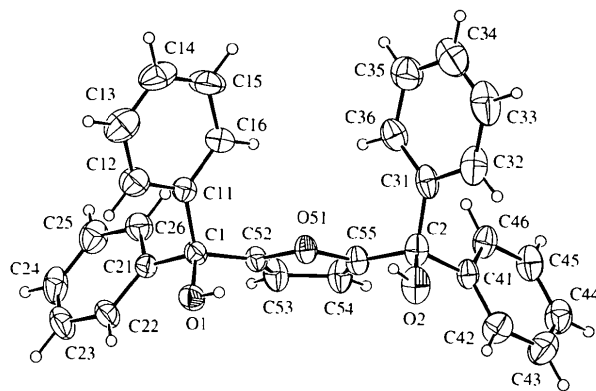


Fig. 3. A view of compound (II) with the atomic numbering scheme. Displacement ellipsoids are at the 30% probability level.

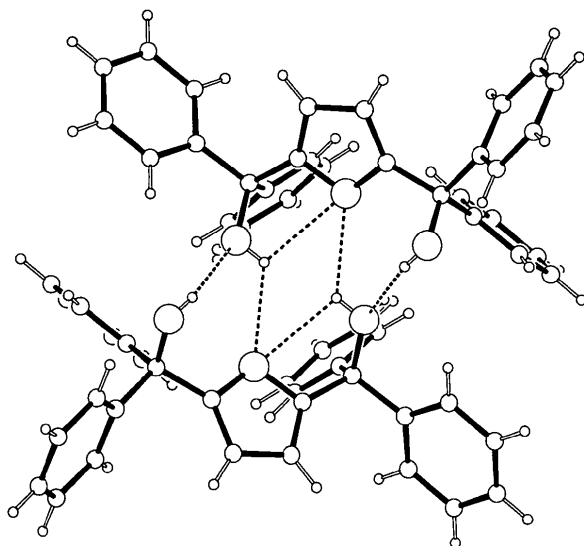


Fig. 4. A view of the hydrogen-bonded dimer of compound (II).

graph set  $N1$  is  $S(5)S(5)DD$  (Bernstein, Davis, Shimoni & Chang, 1995). There are also higher order graph sets involving several types of hydrogen bonds, for example, the ring involving only the two furan O atoms has the binary graph set  $R_2^2(4)$ , while the ring involving all six O atoms has the ternary graph set  $R_6^4(14)$  (Bernstein, Davis, Shimoni & Chang, 1995).

Although no crystallographic symmetry is imposed upon the molecules of compound (II), the two sides of the molecule nonetheless exhibit very closely similar dimensions. In particular, the C52—C53 and C54—C55, and O51—C52 and O51—C55 pairs of bond lengths within the furan fragment are identical within experimental uncertainty. In this respect, the furan ring in compound (II) differs from that in ferrocenyl(2-furyl)phenylmethanol (Ferguson, Glidewell, Opromolla, Zakaria & Zanello, 1995), where the C=C and C—O bond lengths at the *ipso*-C atom of the furan differ significantly from those at the corresponding unsubstituted C atom. All of the bond distances in (II) are typical of their types (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). However, both the exocyclic C—CH<sub>2</sub> and the O—CH<sub>2</sub> bonds are significantly shorter in (I) than in (II) (Tables 2 and 5). We have previously observed (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995) a correlation between the C—O bond lengths in the series  $RCPH_2OH$  and the steric demands of the  $R$  group. Extension of this idea to compound (II) suggests that the steric demands of the furyl group in (II) are very similar to those of phenyl, pyridyl and isopropyl groups.

The closely related compound (2-furyl)diphenylmethanol, (2-C<sub>4</sub>H<sub>3</sub>O)CPh<sub>2</sub>OH, (III), crystallizes in space group  $R\bar{3}$  with  $Z = 24$  in a hexagonal unit cell having  $a = 18.966(6)$ ,  $c = 26.328(3)$  Å and  $V =$

8202(10) Å<sup>3</sup>. The compound is therefore isomorphous with both Ph<sub>3</sub>COH (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992) and (2-thienyl)CPh<sub>2</sub>OH (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995). The structure of Ph<sub>3</sub>COH contains tetrameric aggregates lying on crystallographic threefold axes and held together by O—H...O hydrogen bonds involving only the hydroxy groups. The structure of (2-thienyl)CPh<sub>2</sub>OH, the closest analogue of (III), must contain at least two types of disorder and may contain as many as four independent types of disorder (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995), and accordingly the structure analysis of compound (III) was not pursued. Nonetheless, based upon the known structure of Ph<sub>3</sub>COH, it is clear that the crystal structure of compound (III) differs markedly from that of (II) in that the furan O atom in (III) is not involved in the hydrogen-bonding scheme. Similarly, there is no participation of the furan O atom in the hydrogen-bonding scheme either in compound (I) or in ferrocenyl(2-furyl)phenylmethanol (Ferguson, Glidewell, Opromolla, Zakaria & Zanello, 1995), where the molecules are linked into centrosymmetric dimers by hydrogen bonds involving only the hydroxy groups, forming a cyclic (OH)<sub>2</sub> motif with graph set  $R_2^2(4)$ .

## Experimental

Compound (I) was obtained from Aldrich. Samples of compounds (II) and (III) were prepared by reaction of dilithiofuran or monolithiofuran, respectively, with benzophenone, followed by acid work-up and crystallization from light petroleum. Analysis of (II): found C 83.1, H 5.4%; C<sub>30</sub>H<sub>24</sub>O<sub>3</sub> requires C 83.3, H 5.6%. Analysis of (III): found C 81.0, H 5.2%; C<sub>17</sub>H<sub>14</sub>O<sub>2</sub> requires C 81.6, H 5.6%. NMR: (II)  $\delta_H$  3.37 (*s*, br, 2H, 2 × OH), 5.80 (*s*, 2H, C<sub>4</sub>H<sub>2</sub>O), 7.2–7.4 (*m*, 20H, 4 × C<sub>6</sub>H<sub>5</sub>);  $\delta_C$  78.0 (*s*, C—OH), 110.0 (*d*) and 158.2 (*s*) (C<sub>4</sub>H<sub>2</sub>O), 127.2 (*d*), 127.6 (*d*), 127.9 (*d*) and 144.3 (*s*) (C<sub>6</sub>H<sub>5</sub>); (III)  $\delta_H$  3.10 (*s*, br, 1H, OH), 5.91 (*m*, 1H), 6.33 (*m*, 1H) and 7.45 (*m*, 1H) (C<sub>4</sub>H<sub>3</sub>O), 7.3–7.4 (*m*, 10H, 2 × C<sub>6</sub>H<sub>5</sub>);  $\delta_C$  78.0 (*s*, C—OH), 109.6 (*d*), 110.1 (*d*), 142.6 (*s*) and 157.8 (*s*) (C<sub>4</sub>H<sub>3</sub>O), 127.2 (*d*), 127.6 (*d*), 128.0 (*d*) and 144.6 p.p.m. (*s*) (C<sub>6</sub>H<sub>5</sub>). Crystals of compounds (I), (II) and (III) suitable for single-crystal diffraction were grown by slow evaporation of dichloromethane/light petroleum (b.p. 313–333 K) solutions.

## Compound (I)

### Crystal data

C<sub>6</sub>H<sub>8</sub>O<sub>3</sub>  
 $M_r = 128.12$   
 Orthorhombic  
 $P2_1cn$   
 $a = 4.9220(5)$  Å  
 $b = 7.7525(6)$  Å  
 $c = 16.3663(11)$  Å  
 $V = 624.50(9)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.363$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107$  Å  
 Cell parameters from 25 reflections  
 $\theta = 9.05$ – $19.40^\circ$   
 $\mu = 0.110$  mm<sup>-1</sup>  
 $T = 294(1)$  K  
 Block  
 0.39 × 0.32 × 0.26 mm  
 Colourless

## Data collection

Enraf–Nonius CAD-4  
diffractometer  
 $\theta/2\theta$  scans  
Absorption correction:  
none  
757 measured reflections  
757 independent reflections  
609 observed reflections  
[ $I > 2\sigma(I)$ ]

## Refinement

Refinement on  $F^2$   
 $R(F) = 0.0301$   
 $wR(F^2) = 0.0834$   
 $S = 1.045$   
757 reflections  
96 parameters  
H atoms riding (C—H 0.93–  
0.97, O—H 0.82 Å)  
 $w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.0414P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.005$   
 $\Delta\rho_{\max} = 0.150 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.110 \text{ e } \text{Å}^{-3}$

$\theta_{\max} = 26.90^\circ$   
 $h = 0 \rightarrow 6$   
 $k = 0 \rightarrow 9$   
 $l = 0 \rightarrow 20$   
3 standard reflections  
frequency: 60 min  
intensity decay: none

Extinction correction:  
*SHELXL93* (Sheldrick,  
1993)  
Extinction coefficient:  
0.025 (7)  
Atomic scattering factors  
from *International Tables*  
for *Crystallography* (1992,  
Vol. C, Tables 4.2.6.8 and  
6.1.1.4)  
Absolute configuration:  
Flack (1983) parameter  
= 0.7 (19)

## Compound (II)

## Crystal data

C<sub>30</sub>H<sub>24</sub>O<sub>3</sub>  
 $M_r = 432.49$   
Orthorhombic  
*F2dd*  
 $a = 9.1669 (12) \text{ Å}$   
 $b = 24.060 (3) \text{ Å}$   
 $c = 41.768 (4) \text{ Å}$   
 $V = 9212.2 (18) \text{ Å}^3$   
 $Z = 16$   
 $D_x = 1.247 \text{ Mg m}^{-3}$   
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107 \text{ Å}$   
Cell parameters from 25  
reflections  
 $\theta = 9.55\text{--}18.15^\circ$   
 $\mu = 0.079 \text{ mm}^{-1}$   
 $T = 294 (1) \text{ K}$   
Triangular-shaped plate  
 $0.42 \times 0.37 \times 0.25 \text{ mm}$   
Colourless

## Data collection

Enraf–Nonius CAD-4  
diffractometer  
 $\theta/2\theta$  scans  
Absorption correction:  
none  
3537 measured reflections  
3537 independent reflections  
1742 observed reflections  
[ $I > 2\sigma(I)$ ]

$\theta_{\max} = 29.90^\circ$   
 $h = 0 \rightarrow 12$   
 $k = 0 \rightarrow 33$   
 $l = 0 \rightarrow 58$   
3 standard reflections  
frequency: 120 min  
intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R(F) = 0.0394$   
 $wR(F^2) = 0.0989$   
 $S = 0.930$   
3537 reflections  
306 parameters  
H atoms riding (C—H 0.93,  
O—H 0.82 Å)  
 $w = 1/[\sigma^2(F_o^2) + (0.0494P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.006$   
 $\Delta\rho_{\max} = 0.118 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.118 \text{ e } \text{Å}^{-3}$

Extinction correction:  
*SHELXL93* (Sheldrick,  
1993)  
Extinction coefficient:  
0.00124 (10)  
Atomic scattering factors  
from *International Tables*  
for *Crystallography* (1992,  
Vol. C, Tables 4.2.6.8 and  
6.1.1.4)  
Absolute configuration:  
Flack (1983) parameter  
= 1.0 (14)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ ) for (I)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O1	0.0003 (5)	-0.1332 (2)	0.34511 (8)	0.0399 (4)
O2	-0.0312 (3)	-0.4950 (2)	0.40257 (10)	0.0478 (4)
O3	-0.0318 (4)	-0.0135 (2)	0.17376 (9)	0.0508 (4)
C2	-0.1388 (6)	-0.1891 (3)	0.41238 (11)	0.0408 (5)
C3	-0.3464 (6)	-0.0828 (3)	0.42745 (14)	0.0484 (6)
C4	-0.3384 (6)	0.0484 (3)	0.36707 (14)	0.0511 (6)
C5	-0.1260 (6)	0.0134 (2)	0.31810 (13)	0.0425 (5)
C6	-0.0358 (6)	-0.3460 (3)	0.45326 (11)	0.0475 (6)
C7	-0.0139 (7)	0.0946 (3)	0.24381 (12)	0.0522 (6)

Table 2. Selected geometric parameters ( $\text{Å}$ ,  $^\circ$ ) for (I)

O1—C2	1.367 (2)	C2—C6	1.478 (3)
O1—C5	1.369 (3)	C5—C7	1.476 (3)
C2—C3	1.336 (3)	C6—O2	1.422 (2)
C4—C5	1.345 (4)	C7—O3	1.422 (3)
C3—C4	1.419 (3)		
C2—O1—C5	107.2 (2)	O1—C5—C7	116.8 (2)
C3—C2—O1	109.7 (2)	C3—C2—C6	133.4 (2)
C4—C5—O1	109.1 (2)	C4—C5—C7	134.1 (2)
C2—C3—C4	107.0 (2)	O2—C6—C2	114.2 (2)
C5—C4—C3	107.0 (2)	O3—C7—C5	112.9 (2)
O1—C2—C6	117.0 (2)		
O1—C2—C6—O2	-60.0 (3)	H2A—O2—C6—H6A	-175
O1—C5—C7—O3	62.8 (3)	H3A—O3—C7—H7A	-57
		H2B—O2—C6—H6A	-34
		H3B—O3—C7—H7A	90

Table 3. Hydrogen-bonding geometry ( $\text{Å}$ ,  $^\circ$ ) for (I)

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
O2—H2A...O3 <sup>i</sup>	0.82	1.95	2.766 (2)	172
O3—H3A...O2 <sup>i</sup>	0.82	2.00	2.761 (2)	154
O2—H2B...O3 <sup>ii</sup>	0.82	1.94	2.761 (2)	175
O3—H3B...O2 <sup>iii</sup>	0.82	1.98	2.766 (2)	160

Symmetry codes: (i)  $x - \frac{1}{2}, \frac{1}{2} + y, \frac{1}{2} - z$ ; (ii)  $\frac{1}{2} + x, y - \frac{1}{2}, \frac{1}{2} - z$ ; (iii)  $\frac{1}{2} + x, \frac{1}{2} + y, \frac{1}{2} - z$ .

Table 4. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ ) for (II)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O1	0.1744 (2)	-0.03076 (7)	0.07209 (4)	0.0591 (4)
C1	0.2661 (3)	0.00340 (9)	0.09182 (5)	0.0462 (5)
C11	0.4017 (3)	-0.02994 (9)	0.10096 (5)	0.0481 (6)
C12	0.3841 (4)	-0.08304 (12)	0.11286 (7)	0.0750 (8)
C13	0.5024 (5)	-0.11463 (14)	0.12168 (9)	0.0965 (11)
C14	0.6396 (5)	-0.0943 (2)	0.11911 (8)	0.0957 (11)
C15	0.6592 (4)	-0.0418 (2)	0.10754 (8)	0.0996 (11)
C16	0.5394 (3)	-0.00938 (13)	0.09860 (6)	0.0697 (7)
C21	0.1819 (3)	0.01760 (10)	0.12222 (5)	0.0498 (6)
C22	0.0421 (3)	-0.00061 (11)	0.12730 (7)	0.0646 (7)
C23	-0.0282 (4)	0.01247 (13)	0.15557 (8)	0.0816 (9)
C24	0.0368 (5)	0.04294 (13)	0.17838 (8)	0.0845 (10)
C25	0.1767 (4)	0.06142 (12)	0.17400 (7)	0.0794 (9)
C26	0.2490 (3)	0.04894 (12)	0.14583 (6)	0.0666 (7)
O2	0.4038 (2)	0.06346 (7)	-0.02004 (4)	0.0779 (6)
C2	0.4692 (3)	0.10384 (9)	0.00114 (5)	0.0566 (7)
C31	0.6313 (3)	0.09091 (9)	0.00585 (6)	0.0602 (7)
C32	0.7178 (4)	0.08023 (11)	-0.02086 (8)	0.0863 (11)
C33	0.8662 (5)	0.0702 (2)	-0.01725 (11)	0.110 (2)
C34	0.9306 (5)	0.07069 (14)	0.01205 (12)	0.1066 (13)

C35	0.8464 (4)	0.08181 (14)	0.03813 (10)	0.0903 (10)
C36	0.6980 (4)	0.09168 (11)	0.03526 (7)	0.0704 (8)
C41	0.4502 (3)	0.16177 (10)	-0.01341 (5)	0.0545 (6)
C42	0.3597 (4)	0.17166 (12)	-0.03889 (7)	0.0785 (9)
C43	0.3441 (4)	0.22488 (15)	-0.05104 (8)	0.0895 (10)
C44	0.4149 (4)	0.26802 (13)	-0.03780 (8)	0.0798 (9)
C45	0.5062 (4)	0.25932 (11)	-0.01251 (7)	0.0749 (9)
C46	0.5248 (4)	0.20639 (10)	-0.00039 (6)	0.0658 (7)
O51	0.3735 (2)	0.04959 (6)	0.04560 (3)	0.0497 (4)
C52	0.2989 (3)	0.05607 (9)	0.07396 (5)	0.0478 (5)
C53	0.2667 (3)	0.10985 (10)	0.07775 (6)	0.0669 (8)
C54	0.3213 (3)	0.13884 (11)	0.05084 (6)	0.0673 (8)
C55	0.3855 (3)	0.10154 (9)	0.03175 (5)	0.0524 (6)

Table 5. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II)

O1—C1	1.436 (3)	C2—C55	1.492 (3)
C1—C11	1.528 (3)	O51—C52	1.376 (3)
C1—C21	1.525 (3)	O51—C55	1.382 (3)
C1—C52	1.501 (3)	C52—C53	1.337 (3)
O2—C2	1.444 (3)	C53—C54	1.414 (3)
C2—C41	1.530 (3)	C54—C55	1.337 (3)
C2—C31	1.531 (4)		
O1—C1—C52	108.4 (2)	C41—C2—C31	110.3 (2)
O1—C1—C21	108.0 (2)	C52—O51—C55	107.3 (2)
C52—C1—C21	109.0 (2)	C53—C52—O51	108.7 (2)
O1—C1—C11	108.6 (2)	C53—C52—C1	135.6 (2)
C52—C1—C11	113.9 (2)	O51—C52—C1	115.6 (2)
C21—C1—C11	108.8 (2)	C52—C53—C54	107.8 (2)
O2—C2—C55	106.6 (2)	C55—C54—C53	107.4 (2)
O2—C2—C41	108.8 (2)	C54—C55—O51	108.8 (2)
C55—C2—C41	108.4 (2)	C54—C55—C2	135.4 (2)
O2—C2—C31	110.2 (2)	O51—C55—C2	115.7 (2)
C55—C2—C31	112.4 (2)		
H1—O1—C1—C11	-65	O1—C1—C52—O51	-61.9 (2)
H1—O1—C1—C21	177	C21—C1—C52—O51	-179.2 (2)
H1—O1—C1—C52	59	C11—C1—C52—O51	59.1 (2)
H2A—O2—C2—C31	58	O2—C2—C55—O51	53.8 (3)
H2A—O2—C2—C41	179	C41—C2—C55—O51	170.8 (2)
H2A—O2—C2—C55	-65	C31—C2—C55—O51	-67.0 (3)
H2B—O2—C2—C31	-40		
H2B—O2—C2—C41	81		
H2B—O2—C2—C55	-162		

Table 6. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...O2 <sup>i</sup>	0.82	2.35	3.125 (3)	159
O1—H1...O51	0.82	2.57	2.880 (3)	104
O2—H2A...O51	0.82	2.50	2.776 (3)	101
O2—H2A...O51 <sup>i</sup>	0.82	2.43	2.935 (3)	121

Symmetry code: (i)  $x, -y, -z$ .

Compound (I) crystallized in the orthorhombic system; space group  $P2_1cn$  or  $Pm\bar{c}n$  from the systematic absences. The structure solution was only possible in  $P2_1cn$  (non-standard setting for  $Pna2_1$  such that  $a < b < c$ ). The hydroxy H atoms were disordered over two sites in each case, with occupancies of 0.62 and 0.38 from the difference maps. Compound (II) also crystallized in the orthorhombic system, in space group  $F2dd$  (a non-standard setting of  $Fdd2$  such that  $a < b < c$ ) from the systematic absences. Difference maps showed that the hydroxy H atom on O2 was equally disordered over two sites. For both structures, the data do not allow the direction of the chiral axis to be determined. The diagrams were prepared using ORTEPII (Johnson, 1976) as implemented in PLATON (Spek, 1995a). Examination of the structures with the SOLV option in PLATON showed that there were no solvent accessible voids in the crystal lattices.

For both compounds, data collection: CAD-4/PC Software (Enraf-Nonius, 1992); cell refinement: CAD-4/PC Software; data reduction: DATRD2 in NRCVAX94 (Gabe, Le Page,

Charland, Lee & White, 1989); program(s) used to solve structures: SHELXS86 (Sheldrick, 1985); program(s) used to refine structures: NRCVAX94 and SHELXL93 (Sheldrick, 1993); software used to prepare material for publication: PLUTON (Spek 1995b), NRCVAX94 and SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Baur, W. H. (1972). *Acta Cryst. B28*, 1456–1465.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem.* In the press.
- Bondi, A. (1964). *J. Chem. Phys.* **68**, 441–451.
- Bourne, S. A., Johnson, L., Marais, C., Nassimbeni, L. R., Weber, E., Skobridis, K. & Toda, F. (1991). *J. Chem. Soc. Perkin Trans. 2*, pp. 1707–1713.
- Enraf-Nonius (1992). *CAD-4/PC Software*. Version 1.1. Enraf-Nonius, Delft, The Netherlands.
- Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst. B46*, 256–262.
- Ferguson, G., Carroll, C. D., Glidewell, C., Zakaria, C. M. & Lough, A. J. (1995). *Acta Cryst. B51*, 367–377.
- Ferguson, G., Gallagher, J. F., Glidewell, C., Low, J. N. & Scrimgeour, S. N. (1992). *Acta Cryst. C48*, 1272–1275.
- Ferguson, G., Gallagher, J. F., Glidewell, C. & Zakaria, C. M. (1993). *J. Chem. Soc. Dalton Trans.* pp. 3499–3506.
- Ferguson, G., Glidewell, C., Oromolla, G., Zakaria, C. M. & Zanello, P. (1995). *J. Organomet. Chem.* Accepted for publication.
- Flack, H. (1983). *Acta Cryst. A39*, 876–881.
- Fourme, R. (1972). *Acta Cryst. B28*, 2984–2991.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Glidewell, C., Ferguson, G., Lough, A. J. & Zakaria, C. M. (1994). *J. Chem. Soc. Dalton Trans.* pp. 1971–1982.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Johnson, L., Nassimbeni, L. R. & Toda, F. (1992). *Acta Cryst. B48*, 827–832.
- Martuscelli, E. & Pedone, C. (1968). *Acta Cryst. B24*, 175–179.
- Mazaki, Y., Hayashi, N. & Kobayashi, K. (1992). *J. Chem. Soc. Chem. Commun.* pp. 1381–1383.
- Semmlingsen, D., Nordenson, S. & Aasen, A. (1986). *Acta Chem. Scand. Ser A*, **40**, 559–565.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Spek, A. L. (1995a). *PLATON. Molecular Geometry Program*. Version of July 1995. University of Utrecht, The Netherlands.
- Spek, A. L. (1995b). *PLUTON. Molecular Graphics Program*. Version of July 1995. University of Utrecht, The Netherlands.
- Taylor, R., Kennard, O. & Versichel, W. (1984). *J. Am. Chem. Soc.* **106**, 244–248.
- Toda, F., Kai, A., Toyotaka, R., Yip, W.-H. & Mak, T. C. W. (1989). *Chem. Lett.* pp. 1921–1924.