

# 1,2,3,4-Tetra-O-acetyl- $\beta$ -D-glucopyranuronic acid monohydrate at 120 K and anhydrous 1,2,3,4-tetra-O-acetyl- $\beta$ -D-glucopyranose at 292 K

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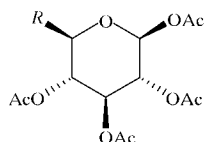
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The structure of the title acid as the monohydrate,  $C_{14}H_{18}O_{11} \cdot H_2O$ , displays hydrogen bonding which connects the molecules in layers parallel to  $(10\bar{1})$ . In the anhydrous glucopyranose,  $C_{14}H_{20}O_{10}$ , only chain connectivity is attained but, due to disorder of the OH group, only partially and in two modes, one less favoured than the other. This provides incomplete connectivity between molecules in corrugated layers parallel to  $(010)$ .

## Comment

The title compounds, namely the acid monohydrate, (I), previously reported by Fry (1955), and the anhydrous glucopyranose, (II), were prepared for use in esterifications with disaccharides.



(I)  $R = CO_2H$ , as the monohydrate

(II)  $R = CH_2OH$ , anhydrous

The asymmetric unit of (I) and the molecule of (II) are shown in Figs. 1 and 2, respectively. The atom labelling is similar and differs only for the O atoms with numeric values of 7 or greater. The compounds obviously differ in terms of the substituents attached to C5, namely  $CO_2H$  in (I) and  $CH_2OH$  in (II), and in the fact that (II) is anhydrous but (I) is the monohydrate. The values given in Table 1 show that the carboxylic acid group in (I) has the expected planar geometry, and bond lengths and angles are in the normal ranges. The hydroxyl group in (II), however, is disordered over two sites, O6A and O6B, with occupancies of 0.639 (7) and 0.361 (7),

respectively. The torsion angles given in Table 1 show that these sites are related to one another by rotation of the OH group about the C5—C6 bond by  $145.1(5)^\circ$ . The C6—O6A and C6—O6B bond lengths of 1.396 (6) and 1.338 (8) Å, respectively, are disappointingly disparate, but this is regarded as a side effect of the disorder. The disorder of the OH group has, as will be discussed later, a profound effect upon the hydrogen bonding in (II).

The pyranose rings, defined by atoms O5/C1—C5, are very similar in the two structures, with bond lengths and angles in the expected ranges and similar chair conformations with puckering parameters (Cremer & Pople, 1975) [values for (II) in square brackets] of 0.594 (2) Å [0.595 (2) Å],  $8.4(2)^\circ$  [ $2.9(2)^\circ$ ] and  $349.1(15)^\circ$  [ $323(6)^\circ$ ] for  $Q$ ,  $\theta$  and  $\varphi$ , respectively. The only significant differences between them reside in the torsion angles given in Table 2. These reflect differences in the orientation of the acetyl substituents with respect to the pyranose rings. The difference is greatest for the acetyl groups

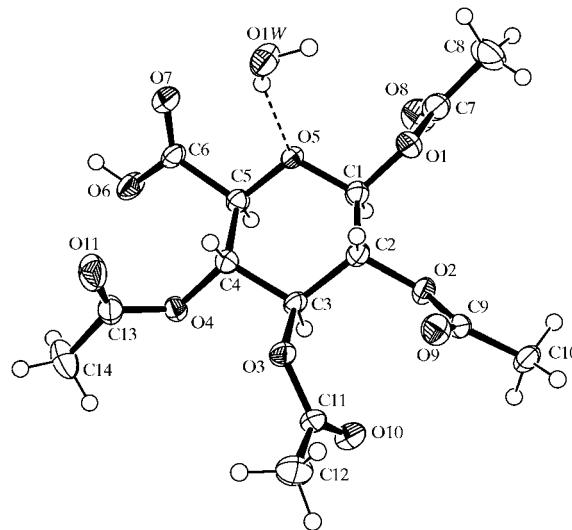


Figure 1

The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The dashed line represents an O—H...O hydrogen bond.

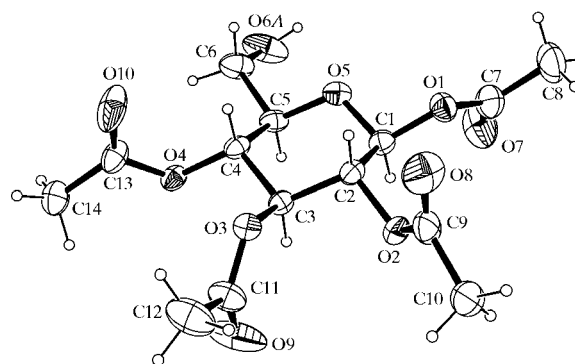


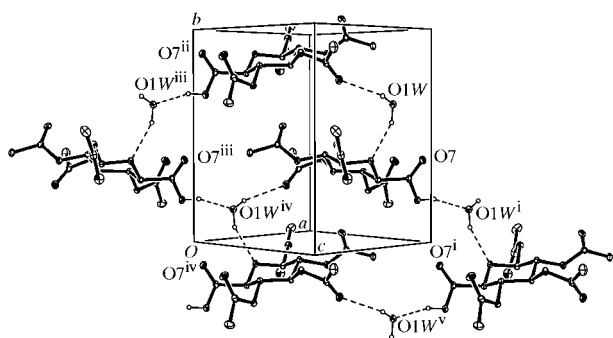
Figure 2

The molecule of (II), with displacement ellipsoids drawn at the 20% probability level and H atoms shown as small spheres of arbitrary radii. Only the major component of the twofold disorder of the OH group is shown.

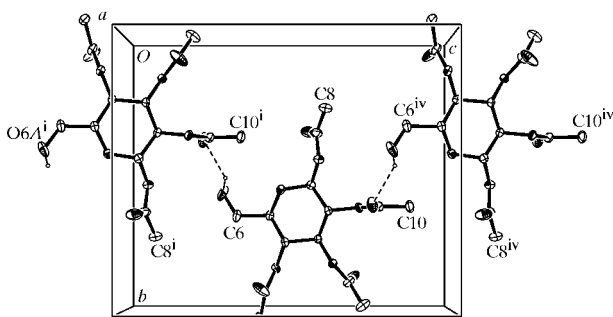
in the 2-position, less at the 3-position, still less at position 4 and least of all at position 1.

The two structures also differ significantly in their hydrogen bonding. In (I), the hydrogen bonds given in Table 3 interconnect the molecules to form layers, as shown in Fig. 3, parallel to (10 $\bar{1}$ ), within which the recurring motif is the trimolecular ring, of which two examples appear in Fig. 3. This connectivity comes about because the water molecule operates as both donor (twice) and acceptor.

In (II), with no water molecule, the possibilities for hydrogen-bond formation (Table 4) are reduced. They are, however, much influenced by the disorder of the OH group. The major component of the disorder, O6A—H6A, provides connectivity within chains propagated in the direction of (001) (Fig. 4), in which the molecules are connected edge-to-edge (type 1 chains). In contrast, the minor component, O6B—H6B, provides connectivity in chains of face-to-face molecules propagated in the direction of (100) (type 2 chains; Fig. 5). Because of the relative occupancies of the two sites, the type 1 chains are considered to be the dominant feature. Therefore, the type 2 connectivity is perceived as interconnecting and, at the same time, fragmenting the type 1 chains. The combination

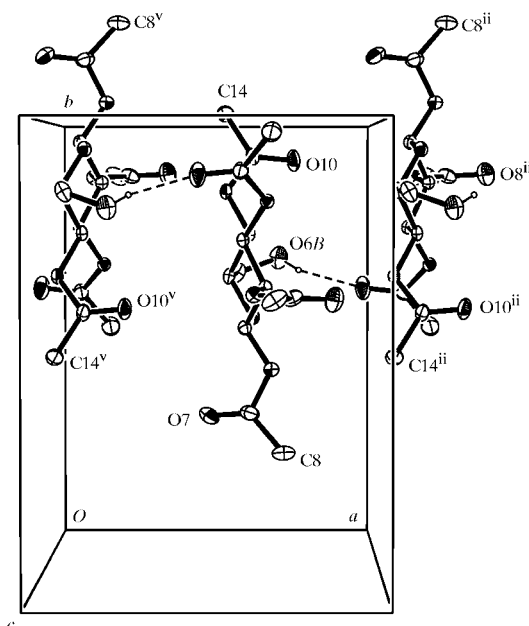


**Figure 3**  
A hydrogen-bonded layer of molecules in (I). Displacement ellipsoids are drawn at the 20% probability level and H atoms involved in hydrogen bonds (dashed lines) are shown as small spheres of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (i)  $2 - x, y - \frac{1}{2}, 2 - z$ ; (ii)  $1 - x, \frac{1}{2} + y, 1 - z$ ; (iii)  $x - 1, y, z - 1$ ; (iv)  $1 - x, y - \frac{1}{2}, 1 - z$ ; (v)  $x, y - 1, z$ .]

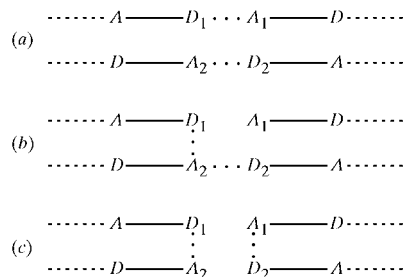


**Figure 4**  
A type 1 chain (see *Comment*) in (II). Displacement ellipsoids are drawn at the 10% probability level and H atoms involved in hydrogen bonds (dashed lines) are shown as small spheres of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (i)  $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$ ; (iv)  $\frac{3}{2} - x, 1 - y, z + \frac{1}{2}$ .]

of these two types of connection provides incomplete two-dimensional connectivity between molecules which, as can be deduced from Figs. 4 and 5, are confined to corrugated layers parallel to (010), one unit cell thick and related to one another by cell translation. If every molecule acts as acceptor for one, and only one, hydrogen bond, this has an intriguing, if conjectural, side effect upon the manner in which the type 2 interactions might be introduced into the structure of (II). The introduction of an isolated type 2 hydrogen bond, as in Fig. 6(b), leaves an acceptor, A1, unused, while A2 becomes a dual acceptor. The introduction of a second complementary and adjacent type 2 interaction, as in Fig. 6(c), permits single-acceptor functionality for all molecules.



**Figure 5**  
A type 2 chain (see *Comment*) in (II). Displacement ellipsoids are drawn at the 10% probability level and H atoms involved in hydrogen bonds (dashed lines) are shown as small spheres of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (ii)  $x + \frac{1}{2}, \frac{3}{2} - y, 1 - z$ ; (v)  $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$ .]



**Figure 6**  
Schematic representation of types 1 and 2 hydrogen bonding in (II). Molecules are represented by donor (D) and acceptor (A) centres joined by solid lines and hydrogen bonds by dashed lines. The arrangements shown are (a) a pair of type 1 chains; (b) as (a), but with the introduction of a single type 2 hydrogen bond, D1...A2; (c) as (b), but with the introduction of a second, complementary, type 2 hydrogen bond, D2...A1.

The descriptions just given consider only the strong O—H···O hydrogen bonds given in Tables 3 and 4. Weaker secondary O—H···O hydrogen bonds involving the H atoms of the water molecule are also present in (I) and still weaker C—H···O interactions are present in both structures. Many of these weaker intermolecular interactions simply parallel or reinforce some of the primary hydrogen bonds.

## Experimental

For the preparation of compound (I), glucuronic acid (5.00 g, 26 mmol) was added to a stirred solution of acetic anhydride (25 ml, 245 mmol) and concentrated sulfuric acid (3 drops). The temperature was allowed to reach 323 K and extra glucuronic acid (5.00 g, 26 mmol) was added. The reaction mixture was maintained at 323–333 K for 1 h with stirring and then cooled to room temperature. Water (75 ml) was added to the stirred solution. After 20 min, crystals of the monohydrate, (I), which had separated out of the solution, were collected and washed with water (yield: 9.76 g, 49.8%); m.p. 369–372 K;  $[\alpha]_D^{24}$  ( $c = 3$ , CHCl<sub>3</sub>) 18.5; literature values for material recrystallized from toluene: m.p. 425–427 K;  $[\alpha]_D$  (CHCl<sub>3</sub>) 16.3 (Fry, 1955). <sup>1</sup>H NMR (250 MHz, CDCl<sub>3</sub>):  $\delta$  1.99 (*s*, 3H, Me), 2.01 (*s*, 3H, Me), 2.03 (*s*, 3H, Me), 2.07 (*s*, 3H, Me), 4.62 (*d*, 1H,  $J = 9.16$  Hz, H6), 5.11 (*dd*, 1H,  $J = 6.8$  and 8.5 Hz, H2), 5.25 (*t*, 1H,  $J = 8.5$  Hz, H3), 5.41 (*t*, 1H,  $J = 8.5$  Hz, H4), 5.83 (*d*, 1H,  $J = 6.8$  Hz, H1); <sup>13</sup>C NMR (63 MHz, CDCl<sub>3</sub>):  $\delta$  20.5, 20.8, 68.9, 70.2, 72.0, 73.1, 97.4, 166.4, 168.8, 169.2, 169.3, 169.9; IR (KBr,  $\nu$ , cm<sup>-1</sup>): 3608–3401, 1761, 1747, 1618. MS (ES<sup>+</sup>): 385.2 [100%,  $M + Na$ ], 401.1 [8%,  $M + K$ ]. The monohydrate, (I), used in the X-ray determination was recrystallized from an acetone–water (1:1 *v/v*) solution.

Compound (II) was prepared in two stages. Firstly, simultaneous acetylation at positions 1–4 and protection of O6 with a trityl group was carried out on commercially available  $\beta$ -D-glucose in the manner described by Talley (1963) with procedural details as follows. To a hot solution of anhydrous D-glucose (20.0 g, 110 mmol) and trityl chloride (33.5 g, 120 mmol) in pyridine (100 ml) was added acetic anhydride (50 ml, 490 mmol). After stirring for 24 h, the reaction mixture was evaporated at reduced pressure to give a syrup. The syrup was added to water, stirred and the resulting precipitate filtered and washed with water. The precipitate was dried and yielded 25.8 g (39%) of the precursor of (II) (m.p. 438–439 K). Thereafter, Amberlite IR 20 resin (20 g) and water (1 ml) were added to a solution of the precursor (20 g, 37 mmol) in CH<sub>3</sub>CN (100 ml). The stirred solution was heated at 333 K for 20 h. The reaction mixture was hot-filtered to remove the resin and, on cooling, a white precipitate formed from the reaction mixture. The precipitate was filtered off, washed with CH<sub>3</sub>CN, and the filtrate and washings combined. The solvent was removed at reduced pressure. A solution of the resulting solid in CH<sub>2</sub>Cl<sub>2</sub> was dried by the addition of anhydrous CaCl<sub>2</sub>, which was then removed by filtration; the solvent was removed from the filtrate under reduced pressure. The solid (II) obtained was crystallized initially from methyl *tert*-butyl ether. Further recrystallization from diethyl ether yielded (II) in its final form (yield: 4.1 g, 37%); m.p. 403–404 K;  $[\alpha]_D$  ( $c = 4$ , CHCl<sub>3</sub>) 11.63; literature value:  $[\alpha]_D$  (CHCl<sub>3</sub>) 12 (Ding *et al.*, 1997; Horrobin *et al.*, 1998). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  1.99 (*s*, 3H, Me), 2.00 (*s*, 3H, Me), 2.03 (*s*, 3H, Me), 2.08 (*s*, 3H, Me), 3.55 (*dd*, 1H,  $J = 4.2$  and 12.5 Hz, H6), 3.62 (*ddd*, 1H,  $J = 2.3$ , 4.2 and 9.7 Hz, H5), 3.73 (*dd*, 1H,  $J = 2.3$  and 12.5 Hz, H6), 5.07 (*dd* and *t*, 2H,  $J = 8.4$  and 9.7 Hz, H2, 4), 5.27 (*t*, 1H,  $J = 9.7$  Hz, H3), 5.70 (*d*, 1H,  $J = 8.4$  Hz, H1); <sup>13</sup>C NMR (63 MHz, CDCl<sub>3</sub>):  $\delta$  20.6, 20.8, 60.8, 68.2, 70.4, 72.6, 77.6, 91.7, 169.1, 169.3, 170.1, 170.3; IR (KBr,  $\nu$ , cm<sup>-1</sup>): 3540, 2954, 1749. MS (ES<sup>+</sup>): 371.2 [100%,  $M + Na$ ], 387.1 [25%,  $M + K$ ].

**Table 1**

Selected geometric parameters for (I) and (II) (Å, °).

(I)		(II)	
C6—O6	1.313 (3)	C6—O6A	1.396 (6)
C6—O7	1.206 (3)	C6—O6B	1.338 (8)
C5—C6—O6	110.1 (2)	C5—C6—O6A	113.1 (4)
C5—C6—O7	123.0 (2)	C5—C6—O6B	116.0 (4)
O6—C6—O7	126.9 (2)		
O5—C5—C6—O6	162.00 (19)	O5—C5—C6—O6A	67.6 (3)
O5—C5—C6—O7	−17.8 (3)	O5—C5—C6—O6B	−77.5 (6)
C4—C5—C6—O6	−80.3 (3)	C4—C5—C6—O6A	−174.5 (3)
C4—C5—C6—O7	99.8 (3)	C4—C5—C6—O6B	40.4 (6)

**Table 2**

Selected torsion angles (°) for (I) and (II).

(I)	(II)	
C7—O1—C1—O5	−90.4 (2)	−89.9 (3)
C7—O1—C1—C2	152.7 (2)	153.7 (2)
C9—O2—C2—C3	−93.0 (2)	−119.2 (2)
C9—O2—C2—C1	148.7 (2)	121.4 (2)
C11—O3—C3—C2	−94.6 (3)	−122.0 (2)
C11—O3—C3—C4	145.0 (2)	119.0 (3)
C13—O4—C4—C3	133.2 (2)	112.7 (2)
C13—O4—C4—C5	−107.2 (2)	−127.4 (2)

## Compound (I)

### Crystal data

C<sub>14</sub>H<sub>18</sub>O<sub>11</sub>·H<sub>2</sub>O  
 $M_r = 380.30$   
 Monoclinic,  $P2_1$   
 $a = 9.0644$  (4) Å  
 $b = 10.4661$  (6) Å  
 $c = 9.7703$  (5) Å  
 $\beta = 103.131$  (3)°  
 $V = 902.66$  (8) Å<sup>3</sup>  
 $Z = 2$

$D_x = 1.399$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 20602 reflections  
 $\theta = 2.9$ – $27.5^\circ$   
 $\mu = 0.13$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 Block, colourless  
 $0.26 \times 0.24 \times 0.12$  mm

### Data collection

Nonius KappaCCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)  
 $T_{\min} = 0.842$ ,  $T_{\max} = 0.988$   
 6414 measured reflections

2131 independent reflections  
 1848 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.047$   
 $\theta_{\max} = 27.5^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -12 \rightarrow 13$   
 $l = -12 \rightarrow 12$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.094$   
 $S = 1.09$   
 2131 reflections  
 248 parameters  
 H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.0412P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

**Table 3**

Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O1W <sup>i</sup>	0.84	1.79	2.592 (3)	159
O1W—H1W1···O5	0.91 (5)	2.12 (5)	2.970 (3)	155 (4)
O1W—H1W2···O10 <sup>ii</sup>	0.84 (5)	2.27 (5)	3.003 (3)	146 (4)

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

Compound (II)

Crystal data

C<sub>14</sub>H<sub>20</sub>O<sub>10</sub> Mo K $\alpha$  radiation  
 M<sub>r</sub> = 348.30 Cell parameters from 4864 reflections  
 Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
 a = 9.4830 (8) Å  $\theta$  = 2.7–24.3°  
 b = 12.6536 (12) Å  $\mu$  = 0.11 mm<sup>-1</sup>  
 c = 15.1455 (14) Å T = 292 (2) K  
 V = 1817.4 (3) Å<sup>3</sup> Block, colourless  
 Z = 4 0.50 × 0.50 × 0.27 mm  
 D<sub>x</sub> = 1.273 Mg m<sup>-3</sup>

Data collection

Bruker SMART 1000 CCD area 3702 independent reflections  
 detector diffractometer 1921 reflections with I > 2σ(I)  
 φ and ω scans R<sub>int</sub> = 0.040  
 Absorption correction: multi-scan θ<sub>max</sub> = 32.6°  
 (SADABS; Sheldrick, 1999) h = -14 → 10  
 T<sub>min</sub> = 0.876, T<sub>max</sub> = 0.928 k = -18 → 19  
 21264 measured reflections l = -22 → 22

Refinement

Refinement on F<sup>2</sup> w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0739P)<sup>2</sup>  
 R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.046 + 0.0408P]  
 wR(F<sup>2</sup>) = 0.148 where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
 S = 1.00 (Δ/σ)<sub>max</sub> < 0.001  
 3702 reflections Δρ<sub>max</sub> = 0.17 e Å<sup>-3</sup>  
 233 parameters Δρ<sub>min</sub> = -0.17 e Å<sup>-3</sup>  
 H-atom parameters constrained

Table 4

Hydrogen-bond geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
O6A—H6A...O8 <sup>i</sup>	0.82	2.15	2.851 (5)	144
O6B—H6B...O9 <sup>ii</sup>	0.82	2.10	2.656 (8)	125
C8—H8B...O6B <sup>iii</sup>	0.96	2.32	3.280 (7)	178

Symmetry codes: (i) -x + 3/2, -y + 1, z - 1/2; (ii) x + 1/2, -y + 3/2, -z + 1; (iii) -x + 3/2, -y + 1, z + 1/2.

In the absence of any atom with Z > 8, the refinements were carried out on merged data. The absolute structures are therefore indeterminate on the basis of the X-ray data and the Flack asymmetry parameters (Flack, 1983) are meaningless. The structural models were, however, set up in accord with the known D configuration of the pyranose ring. A recurrent problem in structures like those of (I) and (II) is considerable freedom of movement for some of the atoms of the substituent groups. For the acetyl groups, this primarily affects the oxo O atom and the methyl group and is attributable, therefore, to oscillation of the group by means of back and fore partial rotation about the C—O bond, e.g. C7—O1. A similar oscillatory motion about the C5—C6 bond applies to the carboxylic acid group in (I). These phenomena are particularly evident in the large and highly anisotropic displacement parameters associated with the motile atoms in the 292 K structure of (II). In the 120 K structure of (I), movement of the atoms is much reduced and the anisotropic displacement parameters are more reasonable.

In the final stages of refinement, H atoms of methyl groups and those attached to tertiary C atoms were placed in calculated positions,

with C—H = 0.98 and 1.00 Å, respectively, for (I) and 0.96 and 0.98 Å for (II), and refined with a riding model, with U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(C<sub>methyl</sub>) or 1.2U<sub>eq</sub>(C<sub>tertiary</sub>). The H atoms of the methylene group in (II) were created as two pairs of atoms, one pair for each component of the disorder of the OH group, and with corresponding occupancy factors, with C—H set to 0.97 Å, and they were refined with a riding model, with U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C). The H atoms of the carboxylic acid group of (I) and the OH group of (II) were placed in calculated positions such as to provide idealized geometry and reasonable hydrogen bonding, with O—H = 0.84 and 0.82 Å for (I) and (II), respectively, and refined, along with the torsion angle about the C—O bond, with a riding model, with U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(O) in both cases. Peaks in a difference map provided approximate coordinates for the H atoms of the water molecule of (I). These H atoms were then refined with isotropic displacement parameters.

Data collection: DENZO (Otwinowski & Minor, 1997) and COLLECT (Nonius, 1998) for (I); SMART (Bruker, 1999) for (II). Cell refinement: DENZO and COLLECT for (I); SAINT (Bruker, 1999) for (II). Data reduction: DENZO and COLLECT for (I); SAINT for (II). For both compounds, program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG1287). Services for accessing these data are described at the back of the journal.

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