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5,5′-Di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzaldehyde and its allyl-protected dialcohol and dialdehyde precursors

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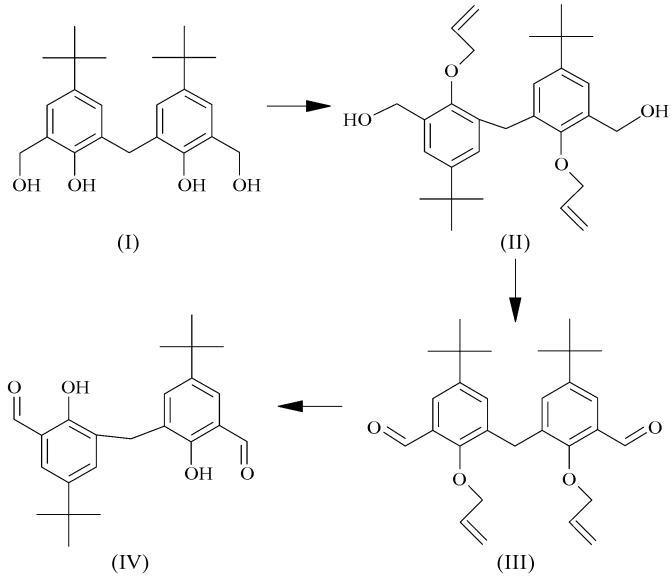
5,5′-Di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzaldehyde, $C_{23}H_{28}O_4$, (IV), has been structurally characterized in two polymorphic forms. The tetragonal form, (in $I4_1/a$) has been reported previously but is redetermined and reinterpreted here, while the monoclinic form, (in $C2/c$) is reported for the first time. In both polymorphs, the molecule lies on a crystallographic twofold axis. Two precursors in the synthesis of (IV), namely 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzenemethanol ($C_{29}H_{40}O_4$) and 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzaldehyde ($C_{29}H_{36}O_4$) have also been characterized.

Comment

The diphenolic dialdehyde 5,5′-di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzaldehyde, (IV), has been used to synthesize new polynucleating macrocycles by Schiff base condensation with diamines (Barreira Fontecha *et al.*, 2002). Compound (IV) was prepared in three steps from the known dialcohol analogue 5,5′-di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzenemethanol, (I) (Dhawan & Gutsche, 1983).

The structure of 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzenemethanol, (II), is shown in Fig. 1. The molecule is non-planar, with the two aryl rings inclined at 78.84 (9)° with respect to one another and the *tert*-butyl groups lying on opposite sides of the molecule. The apparent folding of the molecule is actually due to rotation about the C12—C4 and C12—C13 bonds, and the conformation adopted is probably a consequence of the hydrogen-bonding network throughout the lattice. Hydrogen bonding between alcohol groups generates eight-membered rings [graph-set notation $R_4^4(8)$] and links the molecules into a double chain running parallel to the *a* axis (Fig. 2 and Table 1). The H atoms on the hydroxy groups are disordered, and these atoms were modelled with 50% occupancy of two equivalent positions. As a result there are two self-consistent hydrogen-bonding patterns, which have O—H···O directions running either anticlockwise (as in Fig. 2) or clockwise around the same ring. The highest residual electron-density peak is 1.26 Å from atom C28 and 1.39 Å from atom C27, and may indicate a minor disorder of that allyl group.



The dialcohol was oxidized using MnO_2 to form the analogous dialdehyde, 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzaldehyde, (III). As Fig. 3 shows, the phenyl planes are inclined at 74.17 (5)° and the *tert*-butyl groups are on the same side of the molecule. One of the allyl groups is disordered, and this disorder was modelled as a 70:30 occupancy of two conformations. Again, the molecules are linked by hydrogen bonding into a double chain, in this case running

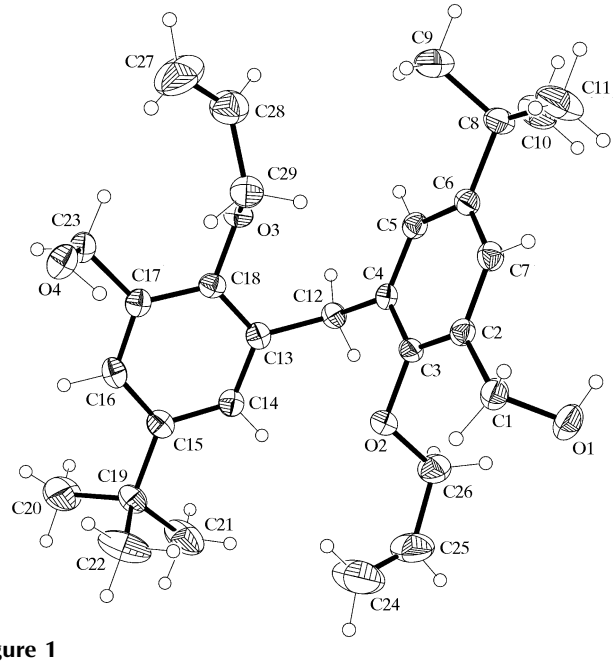


Figure 1
A view of the structure of (II). The H atoms on the two alcohol functions are disordered and only one position is shown for each. Displacement ellipsoids are drawn at the 50% probability level.

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parallel to *c* (Fig. 4); however, the interactions are all of the type C—H···O═C (Table 2). There is also some π–π stacking across the hydrogen-bonded chain involving the benzaldehyde groups. The section incorporating atoms O1/C1/C2/C3/C7 overlaps the O4/C23/C16–C18 section of a neighbouring molecule at $(x, \frac{1}{2}-y, \frac{1}{2}+z)$. The planes of the interacting benzaldehyde rings are inclined at 12.33 (7)°, while atoms O1 and C1 are 3.274 (2) and 3.357 (2) Å, respectively, from the mean plane of the interacting phenyl ring (Fig. 4).

Compound (IV) has been characterized in two polymorphic forms. We obtained the tetragonal form, (IV*a*) (space group $I4_1/a$), by recrystallization of the crude material from diethyl ether, while Masci *et al.* (2004) obtained the same polymorph by recrystallization from methanol. A second polymorph was formed as a side product in the synthesis of a macrocyclic complex; crystals of (IV*b*) in the monoclinic space group *C*2/*c* were obtained from a methanol solution containing 1,5-diaminopentan-3-ol and nickel(II) nitrate.

In the tetragonal form, (IV*a*), the asymmetric unit contains half of the molecule, with a twofold axis passing through atom C12 (Fig. 5), while in the monoclinic form, (IV*b*), the asymmetric unit contains two independent half molecules, each having twofold symmetry (Fig. 6). The molecular conformation and bond lengths are similar in the two polymorphs; the *tert*-butyl groups are on opposite sides of the linked aryl rings and the phenol H atoms are involved in intramolecular hydrogen bonds with the adjacent carbonyl groups (Tables 3 and 4). In (IV*b*), there is additional intermolecular hydrogen bonding involving one of the carbonyl groups (C13═O3). Atom C13 forms a C—H···O═C hydrogen bond to atom O3 of a neighbouring molecule at $(-x, 2-y, 1-z)$, resulting in zigzag chains parallel to *c*. The second molecule does not show a corresponding interaction. As in the precursors, the phenyl rings are inclined with respect to one another: in the tetragonal form, the phenyl rings are inclined at 61.48 (5)°, whereas in the monoclinic polymorph, the values are 73.58 (5) and 75.04 (5)°.

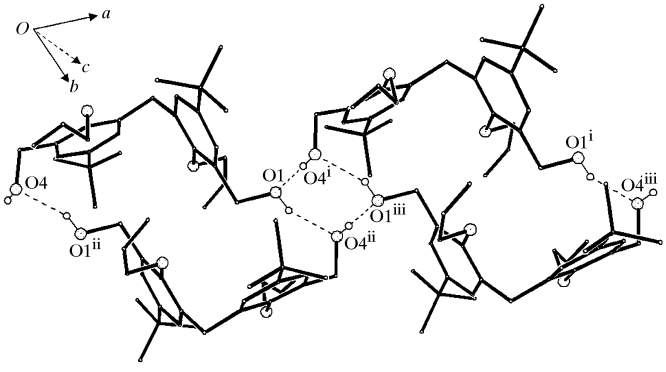

Figure 2
The hydrogen bonding in (II), producing a double chain parallel to *a*. Only one of the two orientations of the hydrogen bonding within the $R_4^4(8)$ ring is shown. [Symmetry codes: (i) $1+x, y, z$; (ii) $-x, 2-y, -z$; (iii) $1-x, 2-y, -z$.]

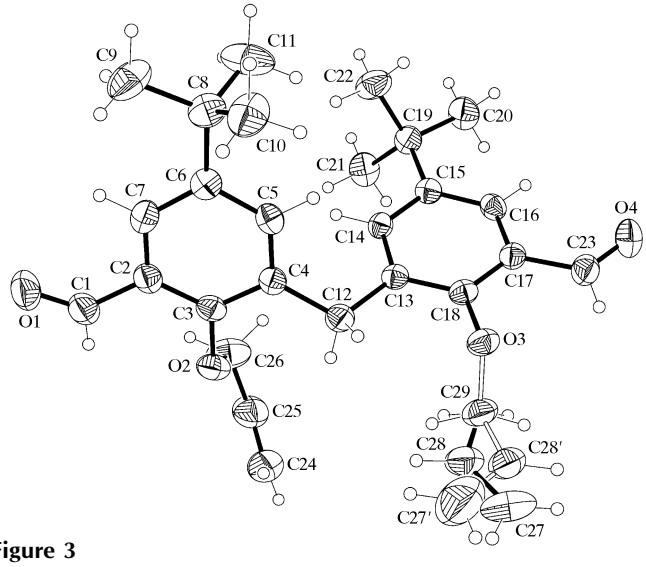

Figure 3
A view of the structure of (III), showing the disorder in one allyl group. Displacement ellipsoids are drawn at the 50% probability level.

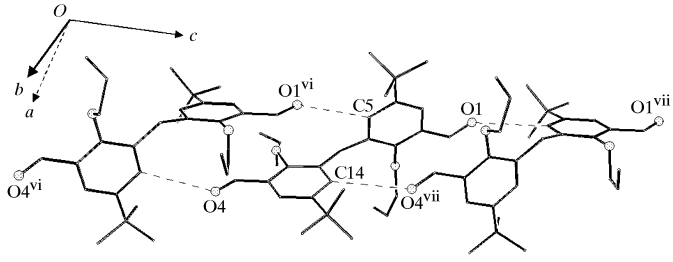

Figure 4
The C—H···O═C hydrogen-bonded chain parallel to *b* in (III). [Symmetry codes: (vi) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (vii) $x, \frac{1}{2}-y, -\frac{1}{2}+z$.]

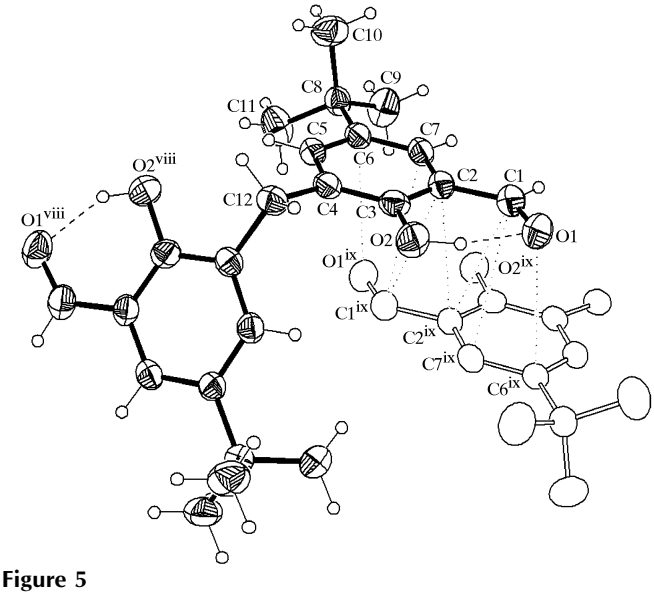

Figure 5
A view of the structure of polymorph (IV*a*); intramolecular hydrogen-bonding interactions between the phenol and aldehyde functions are shown as dashed lines. Dotted lines show interatomic distances in the range 3.42–3.47 Å in the π–π overlap region. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (viii) $2-x, \frac{3}{2}-y, z$; (ix) $2-x, 1-y, -z$.]

In form (IV*a*), the molecules are packed as shown in Figs. 5 and 7. In contrast to the previous report of this structure (Masci *et al.*, 2004), we have identified π–π interactions (the most direct overlap being between the sections containing atoms O1/C1/C2/C7/C6; see Fig. 5) linking the molecules into sets of zigzag ribbons running parallel to either the *a* or the *b* axis. As can be seen in Fig. 5, π-stacked pairs of rings are parallel and related by inversion; the distance between the mean plane of the benzaldehyde ring containing atoms O1 and C7 and the centroid of the neighbouring phenyl ring at $(2 - x, 1 - y, -z)$ is 3.361 (4) Å.

In polymorph (IV*b*), the two independent molecules form *ABAB* π-stacked columns parallel to the *b* axis (Fig. 8). The relative rotation between adjacent layers prevents steric interference between successive *tert*-butyl groups (Fig. 6). The benzaldehyde rings are almost parallel [interplanar angle = 6.34 (10)°], with average interplanar separations of 3.48 and 3.32 Å between the ring containing atoms O1 and C7 and that containing O3 and C19 at $(-x, y, \frac{1}{2} - z)$ and $(-x, 1 + y, \frac{1}{2} - z)$, respectively. Again, the shortest π–π interactions are between the carbonyl groups and the phenyl rings of neighbouring molecules in the stack.

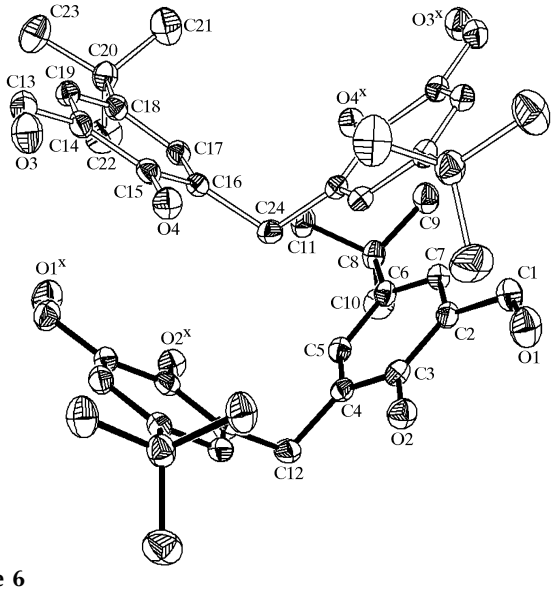

Figure 6
A view of the two independent molecules in polymorph (IV*b*). A twofold axis passes through atoms C12 and C24. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (x) $-x, y, \frac{1}{2} - z$.]

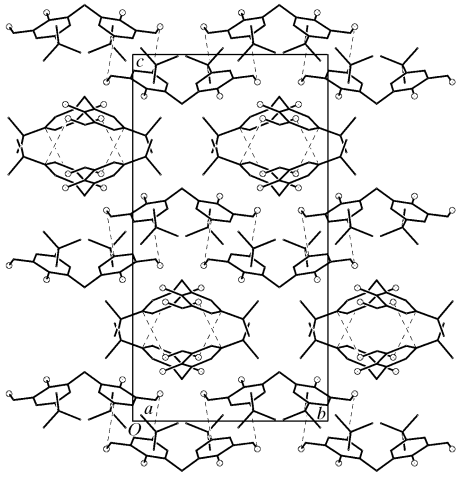

Figure 7
A unit-cell plot for polymorph (IV*a*), viewed down the *a* axis, showing the π–π-stacked ribbons running parallel to *a* and parallel to *b*. O atoms are shown as shaded circles. Dashed lines indicate the π–π overlapped sections.

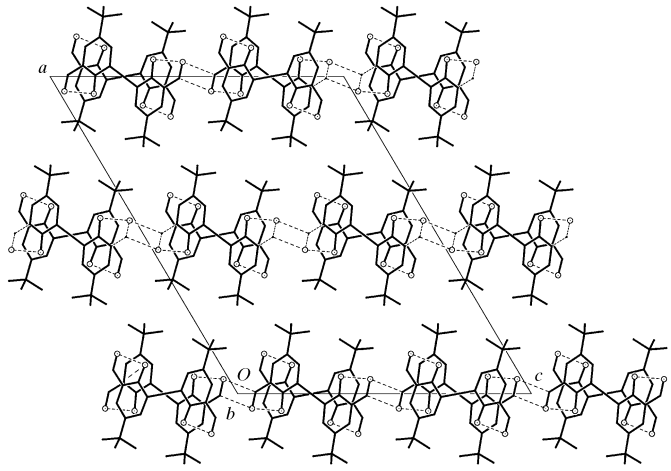

Figure 8
A unit-cell plot for polymorph (IV*b*), projected down *b*, showing π-stacked columns parallel to *b* and inter- and intramolecular hydrogen bonding (dashed lines). O atoms are shown as shaded circles.

Experimental

Compound (I) was synthesized according to the procedure of Dhawan & Gutsche (1983). For the synthesis of (II), compound (I) (10 g, 27 mmol), allyl bromide (7 g, 58 mmol), anhydrous K_2CO_3 (7.42 g) and acetone (100 ml) were placed in a 250 ml three-necked round-bottomed flask fitted with a reflux condenser and a sealed stirrer unit, and were refluxed for 20 h with stirring. The reaction mixture was then poured into water (200 ml) and the aqueous layer was extracted three times with diethyl ether. The organic layer was washed with a 2 *M* sodium hydroxide solution and dried over anhydrous K_2CO_3. The solvent was removed under vacuum, leaving a white solid, which was recrystallized from dichloromethane/*n*-hexane; the yield was 9.0 g (74%). Colourless crystals suitable for X-ray studies were obtained by slow evaporation of a solution of dichloromethane/pentane (1:5). Thin-layer chromatography (TLC) on silica gel (diethyl ether/petroleum ether 40/60, 45:55): R_F = 0.68. Analysis calculated for (II)·0.5H_2O: C 75.45, H 8.95%; found: C 75.64, H 9.06%. ^{1}H NMR ($CDCl_3$, p.p.m.): 7.24 (*d*, 2H, ArH), 7.01 (*d*, 2H, ArH), 6.07 (*m*, 2H, allyl ═CH), 5.34 (*dd*, 2H, allyl ═CH_2), 5.30 (*dd*, 2H, allyl ═CH_2), 4.73 (*d*, 4H, CH_2OH), 4.34 (*d*, 4H, allyl CH_2), 4.70 (*s*, 4H, CH_2OH), 4.07 (*s*, 2H, $ArCH_2Ar$), 1.26 [*s*, 18H, $C(CH_3)_3$]. IR (KBr, cm^{-1}): 3272 [*s*, ν(OH)], 3081 [*w*, ν(allyl ═CH_2)], 883 (*m*, 1,2,3,5 tetrasubstitution of Ar).

Compound (III) was synthesized by a method similar to that reported by Taniguchi (1984). Activated MnO_2 (50 g) was added to a solution of (II) (9 g, 20 mmol) in chloroform (200 ml). The reaction mixture was refluxed for 19–20 h, after which time MnO_2 was filtered off and the organic layer dried over anhydrous $MgSO_4$. The solvent was removed under vacuum, leaving a pale-yellow oil that crystallized under vacuum over a period of one week. The solid was then washed with cold methanol to remove the yellow impurities. Colourless

crystals suitable for X-ray studies were obtained by slow evaporation of a diethyl ether solution of the product. The yield was 7.0 g (78%). TLC on silica gel (diethyl ether/petroleum ether 40/60, 30:70): R_F = 0.50. Analysis calculated for (III)·0.5H_2O: C 76.11, H 8.15%; found: C 76.08, H 8.15%. ^{1}H NMR ($CDCl_3$, p.p.m.): 10.4 (*s*, 2H, CHO), 7.75 (*d*, 2H, ArH), 7.30 (*d*, 2H, ArH), 6.06 (*m*, 2H, allyl ═CH), 4.40 (*dd*, 2H, allyl ═CH_2), 4.44 (*dd*, 2H, allyl ═CH_2), 4.13 (*s*, 2H, $ArCH_2Ar$), 1.26 [*s*, 18H, $C(CH_3)_3$]. IR (KBr, cm^{-1}): 1660 [ν(C═O)], 3081 [*w*, ν(allyl ═CH_2)], 885 (*m*, 1,2,3,5 tetrasubstitution of Ar).

Compound (IV) was obtained using the method described by Boss & Scheffold (1976). To a solution of (III) (7 g, 15.6 mmol) in ethanol (150 ml) were added 10% Pd on activated charcoal (1.5 g) and *p*-toluenesulfonic acid (0.7 g) in water (5 ml). The stirred suspension was refluxed for 2 d, after which time the reaction mixture was filtered hot. On cooling, the product precipitated out as a pale-yellow powder, which was filtered off (yield 1 g). An additional portion of (IV) (3 g) was obtained on concentration of the resulting filtrate. Pale-yellow crystals of (IV*a*) suitable for X-ray studies were obtained by slow evaporation from a solution of the product in diethyl ether. The yield was 4 g (70%). TLC on silica gel (diethyl ether/petroleum ether 40/60, 30:70): R_F = 0.64. Analysis calculated: C 74.97, H 7.66%; found: C 74.51, H 7.86%. ^{1}H NMR ($CDCl_3$, p.p.m.): 11.19 (*s*, 2H, Ar—OH), 9.86 (*s*, 2H, CHO), 7.64 (*d*, 2H, ArH), 7.37 (*d*, 2H, ArH), 4.03 (*s*, 2H, $ArCH_2Ar$), 1.26 [*s*, 18H, $C(CH_3)_3$]. IR (KBr, cm^{-1}): 1658 [ν(C═O)], 1270 [*s*, ν(ArOH)], 1216 (*s*).

Compound (II)

Crystal data

$C_{29}H_{40}O_4$
M_r = 452.61
Triclinic, $P\overline{1}$
a = 10.6025 (11) Å
b = 11.9199 (12) Å
c = 12.4180 (13) Å
α = 64.611 (2)°
β = 82.672 (2)°
γ = 66.330 (2)°
V = 1296.7 (2) Å^3
Z = 2
D_x = 1.159 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2476 reflections
θ = 2.3–27.2°
μ = 0.08 mm^{-1}
T = 150 (2) K
Block, colourless
0.29 × 0.17 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.938, T_{max} = 1.000
9408 measured reflections
4565 independent reflections
3085 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.173
S = 1.04
4565 reflections
310 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.7234P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O*A*···O4^{i}	0.83	1.93	2.744 (3)	166
O1—H1O*B*···O4^{iv}	0.82	1.99	2.749 (2)	155
O4—H4O*A*···O1^{iv}	0.91	1.85	2.749 (2)	167
O4—H4O*B*···O1^{v}	0.84	1.90	2.744 (3)	177

Symmetry codes: (i) $1 + x, y, z$; (iv) $-x, 1 - y, -z$; (v) $x - 1, y, z$.

Table 2
Hydrogen-bonding geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{vii}	0.95	2.46	3.406 (2)	171
C14—H14···O4^{vi}	0.95	2.62	3.560 (2)	170

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

Compound (III)

Crystal data

$C_{29}H_{36}O_4$
M_r = 448.58
Monoclinic, $P2_1/c$
a = 16.2931 (10) Å
b = 10.1951 (6) Å
c = 16.4318 (10) Å
β = 108.367 (1)°
V = 2590.4 (3) Å^3
Z = 4
D_x = 1.150 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7890 reflections
θ = 2.4–23.3°
μ = 0.08 mm^{-1}
T = 150 (2) K
Block, colourless
0.26 × 0.16 × 0.14 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.900, T_{max} = 1.000
17 928 measured reflections
4559 independent reflections
3620 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°
$h = -19 \rightarrow 19$
$k = -12 \rightarrow 12$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.02
4559 reflections
317 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 1.0861P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Polymorph (IV*a*)

Crystal data

$C_{23}H_{28}O_4$
M_r = 368.45
Tetragonal, $I4_1/a$
a = 12.7930 (7) Å
c = 24.158 (2) Å
V = 3953.7 (4) Å^3
Z = 8
D_x = 1.238 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3205 reflections
θ = 2.8–22.8°
μ = 0.08 mm^{-1}
T = 150 (2) K
Tablet, yellow
0.28 × 0.20 × 0.05 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.931, T_{max} = 1.000
19 093 measured reflections
1736 independent reflections
1203 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 25.0°
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
S = 1.04
1736 reflections
125 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 4.6694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Polymorph (IV*b*)

Crystal data

$C_{23}H_{28}O_4$
M_r = 368.45
Monoclinic, $C2/c$
a = 26.809 (2) Å
b = 8.4543 (7) Å
c = 21.3720 (18) Å
β = 120.618 (1)°
V = 4168.7 (6) Å^3
Z = 8
D_x = 1.174 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3051 reflections
θ = 2.6–25.6°
μ = 0.08 mm^{-1}
T = 153 (2) K
Tablet, light brown
0.48 × 0.38 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.919, T_{max} = 1.000
14 739 measured reflections
3668 independent reflections
2235 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 25.0°
$h = -31 \rightarrow 31$
$k = -10 \rightarrow 10$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.132
S = 1.01
3668 reflections
251 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 4.8718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 3
Hydrogen-bonding geometry (Å, °) for (IV*a*).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.92 (2)	1.80 (2)	2.645 (2)	151 (2)

Table 4
Hydrogen-bonding geometry (Å, °) for (IV*b*).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.94 (2)	1.81 (2)	2.625 (3)	144 (2)
O4—H4···O3	0.88 (2)	1.85 (2)	2.631 (2)	147 (2)
C13—H13···O3^{ii}	0.95	2.57	3.454 (3)	156

Symmetry code: (ii) $-x, 2-y, 1-z$.

Except as described below, H atoms bonded to C atoms were placed at calculated positions and refined using a riding model. The constrained C—H distances were 0.95, 0.98, 0.99 and 0.99 Å for aryl, methyl, methylene and ethylene H atoms, respectively. The U_{iso}(H) values were set at 1.2U_{eq}(C) for methylene and aryl H atoms, and at 1.5U_{eq}(C) for *tert*-butyl H atoms. For (II), the disordered H atoms bonded to atoms O1 and O4 were located from difference maps and were not further refined; their U_{iso}(H) values were fixed at 0.04 Å^2. The U_{iso}(H) values of the *tert*-butyl H atoms were fixed at 0.05 Å^2, and those of the H atoms on atoms C27 and C24 at 0.04 Å^2. For (IV*a*), H atoms bonded to O atoms were placed at calculated positions, with a constrained O—H distance of 0.84 Å and with U_{iso}(H) set at 1.5U_{eq} of the carrier O atom. In (IV*b*), the U_{iso} values for the *tert*-butyl H atoms were fixed at 0.05 Å^2, while the phenol H atoms were located from difference maps and their positions refined, with U_{iso}(H) values fixed at 0.05 Å^2.

For all determinations, data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1580). Services for accessing these data are described at the back of the journal.

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supporting information

Acta Cryst. (2004). C**60**, o776–o780 [doi:10.1107/S010827010402089X]

5,5′-Di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzaldehyde and its allyl-protected dialcohol and dialdehyde precursors

Sandrine Goetz, Julia Barreira Fontecha and Vickie McKee

Computing details

For all compounds, data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

(II) 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzenemethanol

Crystal data

$C_{29}H_{40}O_4$
$M_r = 452.61$
Triclinic, $P\overline{1}$
$a = 10.6025$ (11) Å
$b = 11.9199$ (12) Å
$c = 12.4180$ (13) Å
$\alpha = 64.611$ (2)°
$\beta = 82.672$ (2)°
$\gamma = 66.330$ (2)°
$V = 1296.7$ (2) Å^3
$Z = 2$
$F(000) = 492$
$D_x = 1.159$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2476 reflections
$\theta = 2.3$–27.2°
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Block, colourless
0.29 × 0.17 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.938$, $T_{max} = 1.000$
9408 measured reflections
4565 independent reflections
3085 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.173$
$S = 1.04$
4565 reflections
310 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.7234P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.34147 (18)	0.45442 (18)	0.04311 (17)	0.0350 (5)	
C1	0.2100 (3)	0.4894 (3)	−0.0094 (2)	0.0274 (6)	
H1A	0.1918	0.5682	−0.0874	0.033*	
H1B	0.1356	0.5131	0.0434	0.033*	
C2	0.2132 (2)	0.3708 (2)	−0.0259 (2)	0.0223 (5)	
C3	0.1642 (2)	0.2774 (2)	0.0588 (2)	0.0221 (5)	
O2	0.10499 (17)	0.29559 (17)	0.15977 (15)	0.0272 (4)	
C4	0.1704 (2)	0.1654 (2)	0.0439 (2)	0.0221 (5)	
C5	0.2330 (2)	0.1468 (2)	−0.0561 (2)	0.0231 (5)	
H5	0.2392	0.0703	−0.0664	0.028*	
C6	0.2872 (2)	0.2354 (2)	−0.1417 (2)	0.0227 (5)	
C7	0.2738 (2)	0.3479 (2)	−0.1249 (2)	0.0245 (6)	
H7	0.3072	0.4114	−0.1831	0.029*	
C8	0.3584 (3)	0.2077 (3)	−0.2485 (2)	0.0293 (6)	
C9	0.2579 (3)	0.1980 (4)	−0.3195 (3)	0.0512 (9)	
H9A	0.3038	0.1799	−0.3873	0.077*	
H9B	0.1772	0.2832	−0.3488	0.077*	
H9C	0.2284	0.1248	−0.2677	0.077*	
C10	0.4841 (3)	0.0739 (3)	−0.2021 (3)	0.0471 (8)	
H10A	0.5304	0.0556	−0.2697	0.071*	
H10B	0.4540	0.0010	−0.1506	0.071*	
H10C	0.5483	0.0797	−0.1563	0.071*	
C11	0.4082 (4)	0.3183 (4)	−0.3318 (3)	0.0555 (9)	
H11A	0.4536	0.2968	−0.3983	0.083*	
H11B	0.4737	0.3243	−0.2875	0.083*	
H11C	0.3291	0.4047	−0.3629	0.083*	
C12	0.1130 (2)	0.0662 (2)	0.1340 (2)	0.0232 (5)	
H12A	0.1572	0.0321	0.2135	0.028*	
H12B	0.1396	−0.0114	0.1131	0.028*	
C13	−0.0424 (2)	0.1213 (2)	0.1433 (2)	0.0223 (5)	
C14	−0.0995 (2)	0.1238 (2)	0.2502 (2)	0.0239 (6)	
H14	−0.0393	0.0936	0.3161	0.029*	
C15	−0.2413 (3)	0.1690 (2)	0.2644 (2)	0.0255 (6)	
C16	−0.3253 (3)	0.2084 (2)	0.1666 (2)	0.0266 (6)	
H16	−0.4223	0.2378	0.1741	0.032*	
C17	−0.2736 (2)	0.2066 (2)	0.0586 (2)	0.0235 (5)	
C18	−0.1309 (2)	0.1654 (2)	0.0469 (2)	0.0227 (5)	
O3	−0.07517 (17)	0.15992 (17)	−0.05911 (14)	0.0262 (4)	
C19	−0.3047 (3)	0.1796 (3)	0.3797 (2)	0.0291 (6)	

C20	−0.4174 (4)	0.1242 (5)	0.4156 (3)	0.0669 (11)	
H20A	−0.4561	0.1320	0.4892	0.100*	
H20B	−0.3785	0.0292	0.4288	0.100*	
H20C	−0.4903	0.1755	0.3518	0.100*	
C21	−0.1966 (3)	0.1030 (4)	0.4847 (2)	0.0553 (9)	
H21A	−0.2412	0.1130	0.5560	0.083*	
H21B	−0.1248	0.1399	0.4646	0.083*	
H21C	−0.1548	0.0074	0.5003	0.083*	
C22	−0.3634 (4)	0.3278 (3)	0.3585 (3)	0.0570 (10)	
H22A	−0.4037	0.3366	0.4314	0.086*	
H22B	−0.4348	0.3801	0.2933	0.086*	
H22C	−0.2893	0.3617	0.3370	0.086*	
C23	−0.3723 (3)	0.2521 (2)	−0.0429 (2)	0.0273 (6)	
H23A	−0.3207	0.2207	−0.1037	0.033*	
H23B	−0.4411	0.2104	−0.0125	0.033*	
O4	−0.44276 (18)	0.39600 (17)	−0.09784 (16)	0.0323 (5)	
C24	0.0170 (4)	0.3774 (4)	0.3440 (3)	0.0750 (13)	
H24A	−0.0451	0.4117	0.2783	0.090*	
H24B	−0.0134	0.4027	0.4086	0.090*	
C25	0.1415 (4)	0.2974 (4)	0.3455 (3)	0.0510 (9)	
H25	0.1991	0.2663	0.4134	0.061*	
C26	0.2058 (3)	0.2478 (3)	0.2528 (2)	0.0358 (7)	
H26A	0.2811	0.2796	0.2185	0.043*	
H26B	0.2461	0.1481	0.2894	0.043*	
C27	−0.2187 (4)	0.3605 (4)	−0.3517 (3)	0.0665 (11)	
H27A	−0.2885	0.4338	−0.3393	0.080*	
H27B	−0.2238	0.3480	−0.4214	0.080*	
C28	−0.1153 (3)	0.2768 (3)	−0.2729 (3)	0.0437 (8)	
H28	−0.0468	0.2043	−0.2874	0.052*	
C29	−0.0998 (3)	0.2897 (3)	−0.1610 (2)	0.0325 (6)	
H29A	−0.0214	0.3166	−0.1660	0.039*	
H29B	−0.1845	0.3598	−0.1508	0.039*	
H1OA	0.3973	0.4354	−0.0060	0.040*	0.50
H1OB	0.3456	0.5127	0.0594	0.040*	0.50
H4OA	−0.3960	0.4349	−0.0782	0.040*	0.50
H4OB	−0.5073	0.4132	−0.0527	0.040*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0306 (10)	0.0334 (11)	0.0489 (12)	−0.0147 (9)	−0.0023 (9)	−0.0206 (9)
C1	0.0243 (13)	0.0265 (14)	0.0338 (14)	−0.0101 (11)	0.0010 (11)	−0.0141 (12)
C2	0.0192 (12)	0.0231 (13)	0.0248 (13)	−0.0069 (10)	−0.0009 (10)	−0.0107 (11)
C3	0.0172 (12)	0.0281 (13)	0.0210 (12)	−0.0062 (10)	0.0015 (10)	−0.0126 (11)
O2	0.0252 (9)	0.0351 (10)	0.0263 (9)	−0.0119 (8)	0.0055 (7)	−0.0179 (8)
C4	0.0169 (12)	0.0262 (13)	0.0240 (13)	−0.0081 (10)	0.0000 (10)	−0.0110 (11)
C5	0.0211 (12)	0.0227 (13)	0.0254 (13)	−0.0073 (10)	−0.0010 (10)	−0.0105 (11)
C6	0.0186 (12)	0.0250 (13)	0.0205 (12)	−0.0056 (10)	−0.0006 (10)	−0.0081 (10)

C7	0.0241 (13)	0.0251 (13)	0.0243 (13)	−0.0118 (11)	0.0024 (10)	−0.0084 (11)
C8	0.0322 (14)	0.0328 (15)	0.0253 (14)	−0.0130 (12)	0.0070 (11)	−0.0153 (12)
C9	0.0503 (19)	0.075 (2)	0.0376 (17)	−0.0219 (18)	0.0052 (15)	−0.0346 (17)
C10	0.0416 (18)	0.0493 (19)	0.0433 (18)	−0.0079 (15)	0.0106 (14)	−0.0241 (16)
C11	0.079 (3)	0.057 (2)	0.0386 (18)	−0.0352 (19)	0.0284 (17)	−0.0254 (16)
C12	0.0222 (13)	0.0219 (13)	0.0234 (13)	−0.0077 (11)	0.0018 (10)	−0.0084 (10)
C13	0.0229 (13)	0.0175 (12)	0.0252 (13)	−0.0085 (10)	0.0021 (10)	−0.0073 (10)
C14	0.0247 (13)	0.0230 (13)	0.0213 (13)	−0.0105 (11)	0.0010 (10)	−0.0055 (10)
C15	0.0280 (14)	0.0212 (13)	0.0247 (13)	−0.0109 (11)	0.0048 (11)	−0.0069 (11)
C16	0.0221 (13)	0.0248 (13)	0.0305 (14)	−0.0099 (11)	0.0051 (11)	−0.0096 (11)
C17	0.0243 (13)	0.0193 (12)	0.0270 (13)	−0.0096 (10)	0.0004 (10)	−0.0083 (11)
C18	0.0270 (13)	0.0193 (12)	0.0240 (13)	−0.0104 (11)	0.0032 (10)	−0.0101 (10)
O3	0.0289 (10)	0.0273 (10)	0.0230 (9)	−0.0096 (8)	0.0028 (7)	−0.0128 (8)
C19	0.0291 (14)	0.0295 (14)	0.0248 (14)	−0.0112 (12)	0.0075 (11)	−0.0098 (11)
C20	0.073 (3)	0.116 (3)	0.054 (2)	−0.069 (3)	0.0407 (19)	−0.052 (2)
C21	0.0484 (19)	0.069 (2)	0.0240 (16)	−0.0070 (17)	0.0065 (14)	−0.0137 (16)
C22	0.085 (3)	0.0399 (19)	0.0326 (17)	−0.0137 (18)	0.0187 (17)	−0.0165 (15)
C23	0.0232 (13)	0.0263 (14)	0.0327 (14)	−0.0091 (11)	−0.0001 (11)	−0.0125 (11)
O4	0.0310 (10)	0.0283 (10)	0.0362 (11)	−0.0106 (8)	−0.0063 (8)	−0.0108 (8)
C24	0.066 (3)	0.093 (3)	0.059 (2)	−0.001 (2)	−0.001 (2)	−0.051 (2)
C25	0.062 (2)	0.061 (2)	0.0325 (17)	−0.0170 (18)	−0.0015 (15)	−0.0259 (16)
C26	0.0363 (16)	0.0427 (17)	0.0277 (14)	−0.0124 (13)	−0.0020 (12)	−0.0155 (13)
C27	0.082 (3)	0.081 (3)	0.0354 (19)	−0.037 (2)	−0.0150 (19)	−0.0131 (19)
C28	0.0496 (19)	0.0470 (19)	0.0339 (16)	−0.0201 (16)	0.0041 (14)	−0.0156 (14)
C29	0.0355 (15)	0.0301 (15)	0.0281 (14)	−0.0122 (12)	0.0003 (12)	−0.0089 (12)

Geometric parameters (Å, º)

O1—C1	1.440 (3)	C15—C19	1.535 (3)
O1—H1OA	0.8329	C16—C17	1.388 (3)
O1—H1OB	0.8200	C16—H16	0.9500
C1—C2	1.500 (3)	C17—C18	1.399 (3)
C1—H1A	0.9900	C17—C23	1.508 (3)
C1—H1B	0.9900	C18—O3	1.389 (3)
C2—C3	1.391 (3)	O3—C29	1.464 (3)
C2—C7	1.395 (3)	C19—C20	1.520 (4)
C3—O2	1.392 (3)	C19—C22	1.529 (4)
C3—C4	1.399 (3)	C19—C21	1.539 (4)
O2—C26	1.436 (3)	C20—H20A	0.9800
C4—C5	1.392 (3)	C20—H20B	0.9800
C4—C12	1.513 (3)	C20—H20C	0.9800
C5—C6	1.393 (3)	C21—H21A	0.9800
C5—H5	0.9500	C21—H21B	0.9800
C6—C7	1.394 (3)	C21—H21C	0.9800
C6—C8	1.536 (3)	C22—H22A	0.9800
C7—H7	0.9500	C22—H22B	0.9800
C8—C11	1.529 (4)	C22—H22C	0.9800
C8—C9	1.533 (4)	C23—O4	1.437 (3)

C8—C10	1.534 (4)	C23—H23A	0.9900
C9—H9A	0.9800	C23—H23B	0.9900
C9—H9B	0.9800	O4—H4OA	0.9127
C9—H9C	0.9800	O4—H4OB	0.8446
C10—H10A	0.9800	C24—C25	1.278 (5)
C10—H10B	0.9800	C24—H24A	0.9500
C10—H10C	0.9800	C24—H24B	0.9500
C11—H11A	0.9800	C25—C26	1.483 (4)
C11—H11B	0.9800	C25—H25	0.9500
C11—H11C	0.9800	C26—H26A	0.9900
C12—C13	1.519 (3)	C26—H26B	0.9900
C12—H12A	0.9900	C27—C28	1.309 (4)
C12—H12B	0.9900	C27—H27A	0.9500
C13—C18	1.393 (3)	C27—H27B	0.9500
C13—C14	1.394 (3)	C28—C29	1.498 (4)
C14—C15	1.395 (3)	C28—H28	0.9500
C14—H14	0.9500	C29—H29A	0.9900
C15—C16	1.393 (3)	C29—H29B	0.9900

C1—O1—H1OA	103.6	C17—C16—C15	122.9 (2)
C1—O1—H1OB	114.1	C17—C16—H16	118.5
H1OA—O1—H1OB	114.6	C15—C16—H16	118.5
O1—C1—C2	108.52 (19)	C16—C17—C18	118.5 (2)
O1—C1—H1A	110.0	C16—C17—C23	119.3 (2)
C2—C1—H1A	110.0	C18—C17—C23	122.2 (2)
O1—C1—H1B	110.0	O3—C18—C13	119.1 (2)
C2—C1—H1B	110.0	O3—C18—C17	120.2 (2)
H1A—C1—H1B	108.4	C13—C18—C17	120.6 (2)
C3—C2—C7	118.4 (2)	C18—O3—C29	116.10 (18)
C3—C2—C1	121.8 (2)	C20—C19—C22	110.3 (3)
C7—C2—C1	119.7 (2)	C20—C19—C15	111.0 (2)
C2—C3—O2	120.1 (2)	C22—C19—C15	108.2 (2)
C2—C3—C4	121.4 (2)	C20—C19—C21	108.2 (3)
O2—C3—C4	118.5 (2)	C22—C19—C21	106.8 (3)
C3—O2—C26	112.68 (18)	C15—C19—C21	112.2 (2)
C5—C4—C3	117.8 (2)	C19—C20—H20A	109.5
C5—C4—C12	120.8 (2)	C19—C20—H20B	109.5
C3—C4—C12	121.4 (2)	H20A—C20—H20B	109.5
C4—C5—C6	122.9 (2)	C19—C20—H20C	109.5
C4—C5—H5	118.5	H20A—C20—H20C	109.5
C6—C5—H5	118.5	H20B—C20—H20C	109.5
C5—C6—C7	116.9 (2)	C19—C21—H21A	109.5
C5—C6—C8	120.6 (2)	C19—C21—H21B	109.5
C7—C6—C8	122.5 (2)	H21A—C21—H21B	109.5
C6—C7—C2	122.5 (2)	C19—C21—H21C	109.5
C6—C7—H7	118.8	H21A—C21—H21C	109.5
C2—C7—H7	118.8	H21B—C21—H21C	109.5
C11—C8—C9	108.9 (2)	C19—C22—H22A	109.5

C11—C8—C10	108.2 (2)	C19—C22—H22B	109.5
C9—C8—C10	109.3 (2)	H22A—C22—H22B	109.5
C11—C8—C6	111.8 (2)	C19—C22—H22C	109.5
C9—C8—C6	109.7 (2)	H22A—C22—H22C	109.5
C10—C8—C6	108.9 (2)	H22B—C22—H22C	109.5
C8—C9—H9A	109.5	O4—C23—C17	112.0 (2)
C8—C9—H9B	109.5	O4—C23—H23A	109.2
H9A—C9—H9B	109.5	C17—C23—H23A	109.2
C8—C9—H9C	109.5	O4—C23—H23B	109.2
H9A—C9—H9C	109.5	C17—C23—H23B	109.2
H9B—C9—H9C	109.5	H23A—C23—H23B	107.9
C8—C10—H10A	109.5	C23—O4—H4OA	109.5
C8—C10—H10B	109.5	C23—O4—H4OB	104.6
H10A—C10—H10B	109.5	H4OA—O4—H4OB	93.5
C8—C10—H10C	109.5	C25—C24—H24A	120.0
H10A—C10—H10C	109.5	C25—C24—H24B	120.0
H10B—C10—H10C	109.5	H24A—C24—H24B	120.0
C8—C11—H11A	109.5	C24—C25—C26	127.1 (3)
C8—C11—H11B	109.5	C24—C25—H25	116.5
H11A—C11—H11B	109.5	C26—C25—H25	116.5
C8—C11—H11C	109.5	O2—C26—C25	110.4 (2)
H11A—C11—H11C	109.5	O2—C26—H26A	109.6
H11B—C11—H11C	109.5	C25—C26—H26A	109.6
C4—C12—C13	115.50 (19)	O2—C26—H26B	109.6
C4—C12—H12A	108.4	C25—C26—H26B	109.6
C13—C12—H12A	108.4	H26A—C26—H26B	108.1
C4—C12—H12B	108.4	C28—C27—H27A	120.0
C13—C12—H12B	108.4	C28—C27—H27B	120.0
H12A—C12—H12B	107.5	H27A—C27—H27B	120.0
C18—C13—C14	118.5 (2)	C27—C28—C29	123.2 (3)
C18—C13—C12	120.8 (2)	C27—C28—H28	118.4
C14—C13—C12	120.6 (2)	C29—C28—H28	118.4
C13—C14—C15	122.8 (2)	O3—C29—C28	108.9 (2)
C13—C14—H14	118.6	O3—C29—H29A	109.9
C15—C14—H14	118.6	C28—C29—H29A	109.9
C16—C15—C14	116.6 (2)	O3—C29—H29B	109.9
C16—C15—C19	120.6 (2)	C28—C29—H29B	109.9
C14—C15—C19	122.8 (2)	H29A—C29—H29B	108.3

O1—C1—C2—C3	94.7 (3)	C18—C13—C14—C15	−0.2 (4)
O1—C1—C2—C7	−81.6 (3)	C12—C13—C14—C15	−177.7 (2)
C7—C2—C3—O2	179.0 (2)	C13—C14—C15—C16	2.0 (4)
C1—C2—C3—O2	2.7 (3)	C13—C14—C15—C19	−176.4 (2)
C7—C2—C3—C4	−2.3 (3)	C14—C15—C16—C17	−1.4 (4)
C1—C2—C3—C4	−178.5 (2)	C19—C15—C16—C17	177.0 (2)
C2—C3—O2—C26	−83.1 (3)	C15—C16—C17—C18	−1.0 (4)
C4—C3—O2—C26	98.2 (3)	C15—C16—C17—C23	−179.5 (2)
C2—C3—C4—C5	2.8 (3)	C14—C13—C18—O3	−178.3 (2)

O2—C3—C4—C5	−178.4 (2)	C12—C13—C18—O3	−0.8 (3)
C2—C3—C4—C12	−178.0 (2)	C14—C13—C18—C17	−2.3 (3)
O2—C3—C4—C12	0.7 (3)	C12—C13—C18—C17	175.2 (2)
C3—C4—C5—C6	−1.0 (3)	C16—C17—C18—O3	178.8 (2)
C12—C4—C5—C6	179.9 (2)	C23—C17—C18—O3	−2.7 (3)
C4—C5—C6—C7	−1.3 (3)	C16—C17—C18—C13	2.9 (3)
C4—C5—C6—C8	178.5 (2)	C23—C17—C18—C13	−178.6 (2)
C5—C6—C7—C2	1.9 (3)	C13—C18—O3—C29	−112.4 (2)
C8—C6—C7—C2	−177.8 (2)	C17—C18—O3—C29	71.6 (3)
C3—C2—C7—C6	−0.2 (3)	C16—C15—C19—C20	46.1 (3)
C1—C2—C7—C6	176.2 (2)	C14—C15—C19—C20	−135.6 (3)
C5—C6—C8—C11	179.2 (2)	C16—C15—C19—C22	−75.1 (3)
C7—C6—C8—C11	−1.0 (3)	C14—C15—C19—C22	103.2 (3)
C5—C6—C8—C9	58.4 (3)	C16—C15—C19—C21	167.3 (3)
C7—C6—C8—C9	−121.9 (3)	C14—C15—C19—C21	−14.4 (4)
C5—C6—C8—C10	−61.3 (3)	C16—C17—C23—O4	75.1 (3)
C7—C6—C8—C10	118.5 (3)	C18—C17—C23—O4	−103.3 (3)
C5—C4—C12—C13	−114.7 (2)	C3—O2—C26—C25	166.8 (2)
C3—C4—C12—C13	66.2 (3)	C24—C25—C26—O2	−3.8 (5)
C4—C12—C13—C18	63.8 (3)	C18—O3—C29—C28	−145.7 (2)
C4—C12—C13—C14	−118.7 (2)	C27—C28—C29—O3	129.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*OB*···O4^{i}	0.82	1.99	2.749 (2)	155
O1—H1*OA*···O4^{ii}	0.83	1.93	2.744 (3)	166
O4—H4*OA*···O1^{i}	0.91	1.85	2.749 (2)	167
O4—H4*OB*···O1^{iii}	0.84	1.90	2.744 (3)	177

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) *x*+1, *y*, *z*; (iii) *x*−1, *y*, *z*.

(III) 2,2′-bis(allyloxy)-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzaldehyde

Crystal data

$C_{29}H_{36}O_4$
M_r = 448.58
Monoclinic, $P2_1/c$
a = 16.2931 (10) Å
b = 10.1951 (6) Å
c = 16.4318 (10) Å
β = 108.367 (1)°
V = 2590.4 (3) Å^3
Z = 4
$F(000)$ = 968
D_x = 1.150 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7890 reflections
θ = 2.4–23.3°
μ = 0.08 mm^{-1}
T = 150 K
Block, pale yellow
0.26 × 0.16 × 0.14 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.900, T_{max} = 1.000
17928 measured reflections
4559 independent reflections

3620 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.3°$
$h = -19 \rightarrow 19$
$k = -12 \rightarrow 12$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.123$
$S = 1.02$
4559 reflections
317 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 1.0861P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.63607 (10)	0.33380 (15)	1.08524 (9)	0.0608 (4)	
O4	0.86478 (9)	0.34770 (14)	0.45857 (8)	0.0534 (4)	
C1	0.67472 (13)	0.38088 (19)	1.04065 (11)	0.0462 (5)	
H1	0.7123	0.4526	1.0628	0.055*	
C2	0.66705 (10)	0.33351 (16)	0.95351 (10)	0.0342 (4)	
C3	0.70349 (10)	0.40326 (15)	0.90068 (10)	0.0317 (4)	
C4	0.69296 (9)	0.36070 (15)	0.81739 (10)	0.0288 (3)	
C5	0.64915 (9)	0.24344 (16)	0.79061 (10)	0.0310 (4)	
H5	0.6429	0.2128	0.7344	0.037*	
C6	0.61386 (9)	0.16860 (16)	0.84255 (10)	0.0323 (4)	
C7	0.62221 (10)	0.21718 (17)	0.92370 (10)	0.0351 (4)	
H7	0.5969	0.1704	0.9598	0.042*	
C8	0.56911 (11)	0.03898 (18)	0.80846 (12)	0.0428 (4)	
C9	0.52995 (14)	−0.0256 (2)	0.87163 (14)	0.0608 (6)	
H9A	0.5015	−0.1078	0.8471	0.091*	
H9B	0.4874	0.0336	0.8828	0.091*	
H9C	0.5758	−0.0441	0.9255	0.091*	
C10	0.49717 (13)	0.0616 (2)	0.72365 (12)	0.0570 (5)	
H10A	0.4690	−0.0221	0.7022	0.085*	
H10B	0.5219	0.0989	0.6816	0.085*	
H10C	0.4544	0.1226	0.7328	0.085*	
C11	0.63634 (14)	−0.0536 (2)	0.79162 (18)	0.0707 (7)	
H11A	0.6087	−0.1370	0.7689	0.106*	
H11B	0.6830	−0.0694	0.8454	0.106*	
H11C	0.6603	−0.0134	0.7498	0.106*	

C12	0.72087 (10)	0.44424 (16)	0.75498 (11)	0.0342 (4)	
H12A	0.7519	0.5218	0.7861	0.041*	
H12B	0.6684	0.4764	0.7104	0.041*	
C13	0.77824 (9)	0.37788 (14)	0.71076 (9)	0.0272 (3)	
C14	0.83411 (9)	0.27642 (14)	0.74857 (9)	0.0273 (3)	
H14	0.8350	0.2463	0.8036	0.033*	
C15	0.88930 (9)	0.21633 (15)	0.70929 (10)	0.0291 (3)	
C16	0.88543 (10)	0.26107 (15)	0.62858 (10)	0.0307 (3)	
H16	0.9225	0.2230	0.6005	0.037*	
C17	0.82869 (10)	0.36046 (15)	0.58740 (10)	0.0302 (3)	
C18	0.77561 (9)	0.41923 (14)	0.62884 (10)	0.0285 (3)	
C19	0.94943 (10)	0.10569 (16)	0.75587 (11)	0.0358 (4)	
C20	1.00889 (12)	0.05833 (19)	0.70575 (12)	0.0476 (5)	
H20A	1.0459	−0.0126	0.7375	0.071*	
H20B	1.0451	0.1313	0.6984	0.071*	
H20C	0.9737	0.0261	0.6494	0.071*	
C21	1.00683 (11)	0.1551 (2)	0.84346 (11)	0.0465 (5)	
H21A	0.9703	0.1866	0.8767	0.070*	
H21B	1.0433	0.2271	0.8352	0.070*	
H21C	1.0435	0.0833	0.8744	0.070*	
C22	0.89451 (13)	−0.00976 (18)	0.76842 (14)	0.0501 (5)	
H22A	0.8563	0.0197	0.8004	0.075*	
H22B	0.9325	−0.0795	0.8005	0.075*	
H22C	0.8596	−0.0434	0.7123	0.075*	
C23	0.82405 (11)	0.40009 (17)	0.49972 (11)	0.0392 (4)	
H23	0.7869	0.4708	0.4741	0.047*	
O2	0.74638 (8)	0.52087 (11)	0.92911 (7)	0.0401 (3)	
C24	0.84626 (18)	0.7420 (2)	0.93001 (16)	0.0698 (7)	
H24A	0.7858	0.7440	0.9009	0.084*	
H24B	0.8796	0.8198	0.9350	0.084*	
C25	0.88268 (15)	0.6348 (2)	0.96244 (14)	0.0573 (5)	
H25	0.9432	0.6375	0.9910	0.069*	
C26	0.83900 (13)	0.50676 (19)	0.95959 (15)	0.0577 (6)	
H26A	0.8572	0.4464	0.9213	0.069*	
H26B	0.8568	0.4678	1.0177	0.069*	
O3	0.71578 (7)	0.51227 (10)	0.58584 (7)	0.0357 (3)	
C27	0.6204 (3)	0.8014 (4)	0.5349 (3)	0.0694 (10)	0.70
H27A	0.6445	0.8179	0.4902	0.083*	0.70
H27B	0.5682	0.8436	0.5344	0.083*	0.70
C28	0.6583 (3)	0.7218 (4)	0.5960 (3)	0.0572 (9)	0.70
H28	0.6312	0.7092	0.6389	0.069*	0.70
C29	0.73933 (12)	0.64767 (16)	0.60730 (12)	0.0444 (4)	0.70
H29A	0.7704	0.6834	0.5692	0.053*	0.70
H29B	0.7776	0.6545	0.6674	0.053*	0.70
C27′	0.6173 (11)	0.7853 (16)	0.5874 (11)	0.113 (5)	0.30
H27C	0.6230	0.7673	0.6456	0.136*	0.30
H27D	0.5724	0.8411	0.5545	0.136*	0.30
C28′	0.6708 (5)	0.7338 (8)	0.5528 (7)	0.0506 (19)	0.30

H28′	0.6662	0.7506	0.4947	0.061*	0.30
C29′	0.73933 (12)	0.64767 (16)	0.60730 (12)	0.0444 (4)	0.30
H29C	0.7951	0.6672	0.5977	0.053*	0.30
H29D	0.7462	0.6635	0.6685	0.053*	0.30

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0847 (10)	0.0650 (9)	0.0419 (7)	0.0187 (8)	0.0331 (7)	0.0085 (7)
O4	0.0657 (9)	0.0631 (9)	0.0403 (7)	−0.0012 (7)	0.0293 (7)	0.0014 (6)
C1	0.0606 (12)	0.0427 (10)	0.0356 (9)	0.0168 (9)	0.0158 (9)	0.0044 (8)
C2	0.0355 (8)	0.0363 (9)	0.0302 (8)	0.0101 (7)	0.0093 (7)	0.0024 (7)
C3	0.0300 (8)	0.0286 (8)	0.0345 (8)	0.0064 (7)	0.0072 (7)	−0.0006 (7)
C4	0.0233 (7)	0.0316 (8)	0.0324 (8)	0.0063 (6)	0.0100 (6)	0.0023 (7)
C5	0.0253 (8)	0.0377 (9)	0.0292 (8)	0.0035 (7)	0.0077 (6)	−0.0024 (7)
C6	0.0225 (7)	0.0363 (9)	0.0365 (9)	0.0022 (6)	0.0070 (7)	0.0021 (7)
C7	0.0303 (8)	0.0409 (9)	0.0358 (9)	0.0062 (7)	0.0128 (7)	0.0090 (7)
C8	0.0337 (9)	0.0420 (10)	0.0498 (10)	−0.0076 (8)	0.0092 (8)	−0.0008 (8)
C9	0.0595 (13)	0.0555 (13)	0.0609 (13)	−0.0213 (10)	0.0099 (10)	0.0109 (10)
C10	0.0489 (11)	0.0653 (13)	0.0498 (11)	−0.0255 (10)	0.0056 (9)	−0.0017 (10)
C11	0.0572 (13)	0.0431 (12)	0.109 (2)	−0.0097 (10)	0.0217 (13)	−0.0260 (12)
C12	0.0342 (8)	0.0317 (9)	0.0400 (9)	0.0062 (7)	0.0165 (7)	0.0034 (7)
C13	0.0243 (7)	0.0261 (8)	0.0309 (8)	−0.0034 (6)	0.0084 (6)	−0.0014 (6)
C14	0.0255 (7)	0.0292 (8)	0.0276 (7)	−0.0019 (6)	0.0089 (6)	0.0010 (6)
C15	0.0252 (7)	0.0295 (8)	0.0329 (8)	−0.0017 (6)	0.0095 (6)	−0.0010 (6)
C16	0.0295 (8)	0.0321 (8)	0.0336 (8)	−0.0019 (7)	0.0144 (7)	−0.0047 (7)
C17	0.0316 (8)	0.0294 (8)	0.0293 (8)	−0.0065 (6)	0.0091 (7)	−0.0014 (6)
C18	0.0257 (7)	0.0254 (8)	0.0314 (8)	−0.0034 (6)	0.0046 (6)	−0.0002 (6)
C19	0.0335 (8)	0.0368 (9)	0.0401 (9)	0.0075 (7)	0.0158 (7)	0.0050 (7)
C20	0.0442 (10)	0.0514 (11)	0.0510 (11)	0.0208 (9)	0.0204 (9)	0.0065 (9)
C21	0.0377 (9)	0.0572 (12)	0.0416 (10)	0.0153 (9)	0.0085 (8)	0.0071 (9)
C22	0.0515 (11)	0.0352 (10)	0.0674 (13)	0.0097 (8)	0.0244 (10)	0.0117 (9)
C23	0.0450 (10)	0.0392 (9)	0.0340 (9)	−0.0080 (8)	0.0135 (8)	−0.0002 (7)
O2	0.0458 (7)	0.0293 (6)	0.0416 (7)	0.0021 (5)	0.0086 (5)	−0.0048 (5)
C24	0.0963 (18)	0.0447 (12)	0.0846 (17)	−0.0022 (12)	0.0518 (15)	0.0034 (12)
C25	0.0648 (13)	0.0463 (12)	0.0629 (13)	−0.0108 (10)	0.0232 (11)	−0.0115 (10)
C26	0.0475 (11)	0.0402 (11)	0.0708 (14)	−0.0048 (9)	−0.0022 (10)	−0.0021 (10)
O3	0.0379 (6)	0.0285 (6)	0.0364 (6)	0.0024 (5)	0.0055 (5)	0.0039 (5)
C27	0.060 (2)	0.0344 (18)	0.103 (3)	0.0072 (17)	0.011 (2)	0.008 (2)
C28	0.076 (3)	0.0338 (18)	0.074 (3)	0.0125 (16)	0.041 (2)	0.0073 (19)
C29	0.0538 (11)	0.0277 (9)	0.0513 (11)	−0.0033 (8)	0.0161 (9)	0.0038 (8)
C27′	0.145 (12)	0.088 (9)	0.138 (13)	0.024 (8)	0.091 (11)	0.042 (9)
C28′	0.060 (5)	0.036 (4)	0.063 (5)	0.004 (4)	0.029 (4)	0.010 (4)
C29′	0.0538 (11)	0.0277 (9)	0.0513 (11)	−0.0033 (8)	0.0161 (9)	0.0038 (8)

Geometric parameters (Å, º)

O1—C1	1.206 (2)	C16—H16	0.9500
O4—C23	1.211 (2)	C17—C18	1.394 (2)
C1—C2	1.479 (2)	C17—C23	1.475 (2)
C1—H1	0.9500	C18—O3	1.3839 (18)
C2—C3	1.392 (2)	C19—C22	1.531 (2)
C2—C7	1.398 (2)	C19—C21	1.534 (2)
C3—O2	1.3923 (19)	C19—C20	1.534 (2)
C3—C4	1.394 (2)	C20—H20A	0.9800
C4—C5	1.391 (2)	C20—H20B	0.9800
C4—C12	1.509 (2)	C20—H20C	0.9800
C5—C6	1.397 (2)	C21—H21A	0.9800
C5—H5	0.9500	C21—H21B	0.9800
C6—C7	1.388 (2)	C21—H21C	0.9800
C6—C8	1.528 (2)	C22—H22A	0.9800
C7—H7	0.9500	C22—H22B	0.9800
C8—C9	1.528 (3)	C22—H22C	0.9800
C8—C10	1.530 (3)	C23—H23	0.9500
C8—C11	1.535 (3)	O2—C26	1.440 (2)
C9—H9A	0.9800	C26—C25	1.481 (3)
C9—H9B	0.9800	C26—H26A	0.9900
C9—H9C	0.9800	C26—H26B	0.9900
C10—H10A	0.9800	C25—C24	1.277 (3)
C10—H10B	0.9800	C25—H25	0.9500
C10—H10C	0.9800	C24—H24A	0.9500
C11—H11A	0.9800	C24—H24B	0.9500
C11—H11B	0.9800	O3—C29	1.446 (2)
C11—H11C	0.9800	C29—C28	1.481 (5)
C12—C13	1.513 (2)	C29—H29A	0.9900
C12—H12A	0.9900	C29—H29B	0.9900
C12—H12B	0.9900	C28—C27	1.289 (6)
C13—C14	1.389 (2)	C28—H28	0.9500
C13—C18	1.398 (2)	C27—H27A	0.9500
C14—C15	1.402 (2)	C27—H27B	0.9500
C14—H14	0.9500	C28′—C27′	1.291 (17)
C15—C16	1.385 (2)	C28′—H28′	0.9500
C15—C19	1.531 (2)	C27′—H27C	0.9500
C16—C17	1.395 (2)	C27′—H27D	0.9500
O1—C1—C2	123.87 (19)	C18—C17—C16	119.59 (14)
O1—C1—H1	118.1	C18—C17—C23	120.72 (15)
C2—C1—H1	118.1	C16—C17—C23	119.67 (14)
C3—C2—C7	119.57 (15)	O3—C18—C17	119.40 (13)
C3—C2—C1	120.97 (16)	O3—C18—C13	120.08 (13)
C7—C2—C1	119.45 (16)	C17—C18—C13	120.35 (14)
C2—C3—O2	119.90 (14)	C22—C19—C15	108.93 (13)
C2—C3—C4	120.58 (15)	C22—C19—C21	109.61 (15)

O2—C3—C4	119.40 (14)	C15—C19—C21	109.38 (14)
C5—C4—C3	118.05 (14)	C22—C19—C20	108.97 (15)
C5—C4—C12	120.31 (14)	C15—C19—C20	112.11 (14)
C3—C4—C12	121.42 (14)	C21—C19—C20	107.81 (14)
C4—C5—C6	123.05 (14)	C19—C20—H20A	109.5
C4—C5—H5	118.5	C19—C20—H20B	109.5
C6—C5—H5	118.5	H20A—C20—H20B	109.5
C7—C6—C5	117.22 (15)	C19—C20—H20C	109.5
C7—C6—C8	123.49 (15)	H20A—C20—H20C	109.5
C5—C6—C8	119.29 (15)	H20B—C20—H20C	109.5
C6—C7—C2	121.42 (15)	C19—C21—H21A	109.5
C6—C7—H7	119.3	C19—C21—H21B	109.5
C2—C7—H7	119.3	H21A—C21—H21B	109.5
C6—C8—C9	112.13 (16)	C19—C21—H21C	109.5
C6—C8—C10	110.10 (15)	H21A—C21—H21C	109.5
C9—C8—C10	108.64 (15)	H21B—C21—H21C	109.5
C6—C8—C11	108.22 (14)	C19—C22—H22A	109.5
C9—C8—C11	109.38 (18)	C19—C22—H22B	109.5
C10—C8—C11	108.31 (18)	H22A—C22—H22B	109.5
C8—C9—H9A	109.5	C19—C22—H22C	109.5
C8—C9—H9B	109.5	H22A—C22—H22C	109.5
H9A—C9—H9B	109.5	H22B—C22—H22C	109.5
C8—C9—H9C	109.5	O4—C23—C17	123.79 (17)
H9A—C9—H9C	109.5	O4—C23—H23	118.1
H9B—C9—H9C	109.5	C17—C23—H23	118.1
C8—C10—H10A	109.5	C3—O2—C26	113.02 (12)
C8—C10—H10B	109.5	O2—C26—C25	111.32 (17)
H10A—C10—H10B	109.5	O2—C26—H26A	109.4
C8—C10—H10C	109.5	C25—C26—H26A	109.4
H10A—C10—H10C	109.5	O2—C26—H26B	109.4
H10B—C10—H10C	109.5	C25—C26—H26B	109.4
C8—C11—H11A	109.5	H26A—C26—H26B	108.0
C8—C11—H11B	109.5	C24—C25—C26	125.9 (2)
H11A—C11—H11B	109.5	C24—C25—H25	117.1
C8—C11—H11C	109.5	C26—C25—H25	117.1
H11A—C11—H11C	109.5	C25—C24—H24A	120.0
H11B—C11—H11C	109.5	C25—C24—H24B	120.0
C4—C12—C13	115.99 (13)	H24A—C24—H24B	120.0
C4—C12—H12A	108.3	C18—O3—C29	116.08 (12)
C13—C12—H12A	108.3	O3—C29—C28	107.39 (19)
C4—C12—H12B	108.3	O3—C29—H29A	110.2
C13—C12—H12B	108.3	C28—C29—H29A	110.2
H12A—C12—H12B	107.4	O3—C29—H29B	110.2
C14—C13—C18	118.18 (14)	C28—C29—H29B	110.2
C14—C13—C12	122.38 (13)	H29A—C29—H29B	108.5
C18—C13—C12	119.44 (13)	C27—C28—C29	127.5 (6)
C13—C14—C15	122.99 (14)	C27—C28—H28	116.3
C13—C14—H14	118.5	C29—C28—H28	116.3

C15—C14—H14	118.5	C28—C27—H27A	120.0
C16—C15—C14	117.04 (14)	C28—C27—H27B	120.0
C16—C15—C19	123.37 (14)	H27A—C27—H27B	120.0
C14—C15—C19	119.59 (13)	C27′—C28′—H28′	121.3
C15—C16—C17	121.81 (14)	C28′—C27′—H27C	120.0
C15—C16—H16	119.1	C28′—C27′—H27D	120.0
C17—C16—H16	119.1	H27C—C27′—H27D	120.0

O1—C1—C2—C3	170.72 (16)	C13—C14—C15—C16	−1.0 (2)
O1—C1—C2—C7	−9.0 (3)	C13—C14—C15—C19	179.52 (14)
C7—C2—C3—O2	178.41 (13)	C14—C15—C16—C17	−0.7 (2)
C1—C2—C3—O2	−1.3 (2)	C19—C15—C16—C17	178.78 (14)
C7—C2—C3—C4	2.3 (2)	C15—C16—C17—C18	1.6 (2)
C1—C2—C3—C4	−177.39 (14)	C15—C16—C17—C23	−177.01 (14)
C2—C3—C4—C5	−3.3 (2)	C16—C17—C18—O3	−176.18 (13)
O2—C3—C4—C5	−179.43 (13)	C23—C17—C18—O3	2.4 (2)
C2—C3—C4—C12	171.27 (14)	C16—C17—C18—C13	−0.8 (2)
O2—C3—C4—C12	−4.8 (2)	C23—C17—C18—C13	177.74 (14)
C3—C4—C5—C6	1.5 (2)	C14—C13—C18—O3	174.56 (13)
C12—C4—C5—C6	−173.22 (14)	C12—C13—C18—O3	−5.5 (2)
C4—C5—C6—C7	1.4 (2)	C14—C13—C18—C17	−0.7 (2)
C4—C5—C6—C8	−178.22 (14)	C12—C13—C18—C17	179.24 (14)
C5—C6—C7—C2	−2.5 (2)	C16—C15—C19—C22	−116.42 (17)
C8—C6—C7—C2	177.14 (14)	C14—C15—C19—C22	63.02 (19)
C3—C2—C7—C6	0.7 (2)	C16—C15—C19—C21	123.80 (16)
C1—C2—C7—C6	−179.59 (15)	C14—C15—C19—C21	−56.76 (19)
C7—C6—C8—C9	3.7 (2)	C16—C15—C19—C20	4.3 (2)
C5—C6—C8—C9	−176.71 (15)	C14—C15—C19—C20	−176.29 (15)
C7—C6—C8—C10	124.76 (18)	C18—C17—C23—O4	−175.62 (16)
C5—C6—C8—C10	−55.6 (2)	C16—C17—C23—O4	3.0 (3)
C7—C6—C8—C11	−117.05 (19)	C2—C3—O2—C26	100.36 (18)
C5—C6—C8—C11	62.6 (2)	C4—C3—O2—C26	−83.54 (18)
C5—C4—C12—C13	−56.86 (19)	C3—O2—C26—C25	161.55 (16)
C3—C4—C12—C13	128.65 (15)	O2—C26—C25—C24	−10.7 (3)
C4—C12—C13—C14	−29.0 (2)	C17—C18—O3—C29	−101.49 (17)
C4—C12—C13—C18	150.97 (14)	C13—C18—O3—C29	83.16 (18)
C18—C13—C14—C15	1.7 (2)	C18—O3—C29—C28	−149.5 (2)
C12—C13—C14—C15	−178.28 (14)	O3—C29—C28—C27	−105.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{i}	0.95	2.46	3.406 (2)	171
C14—H14···O4^{ii}	0.95	2.62	3.560 (2)	170

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*, −*y*+1/2, *z*+1/2.

(IVa) 5,5′-di-*tert*-butyl-2,2′-dihydroxy-3,3′-methanediyldibenzaldehyde

Crystal data

$C_{23}H_{28}O_4$
$M_r = 368.45$
Tetragonal, $I4_1/a$
$a = 12.7930$ (7) Å
$c = 24.158$ (2) Å
$V = 3953.7$ (4) Å^3
$Z = 8$
$F(000) = 1584$
$D_x = 1.238$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3205 reflections
$\theta = 2.8$–$22.8°$
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Block ## AUTHOR: Tablet?, yellow
0.28 × 0.20 × 0.05 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.931$, $T_{max} = 1.000$
19093 measured reflections
1736 independent reflections
1203 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.04$
1736 reflections
125 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 4.6694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.91517 (13)	0.36217 (12)	0.07535 (7)	0.0529 (5)	
C1	1.00238 (18)	0.38549 (17)	0.05825 (10)	0.0448 (6)	
H1	1.0406	0.3332	0.0390	0.054*	
C2	1.05164 (16)	0.48672 (16)	0.06515 (9)	0.0359 (5)	
C3	0.99989 (15)	0.56794 (16)	0.09323 (9)	0.0359 (5)	
O2	0.90144 (11)	0.55533 (13)	0.11373 (7)	0.0472 (4)	
H2	0.8850 (17)	0.4864 (18)	0.1065 (9)	0.040*	
C4	1.05026 (15)	0.66379 (15)	0.10117 (8)	0.0332 (5)	
C5	1.14976 (15)	0.67573 (16)	0.07960 (8)	0.0342 (5)	

H5	1.1840	0.7408	0.0851	0.041*	
C6	1.20321 (15)	0.59785 (15)	0.05009 (8)	0.0332 (5)	
C7	1.15190 (16)	0.50302 (16)	0.04417 (9)	0.0364 (5)	
H7	1.1860	0.4476	0.0253	0.044*	
C8	1.31390 (16)	0.61729 (16)	0.02875 (9)	0.0379 (5)	
C9	1.3448 (2)	0.5401 (2)	−0.01681 (11)	0.0575 (7)	
H9A	1.4161	0.5554	−0.0293	0.086*	
H9B	1.2964	0.5469	−0.0481	0.086*	
H9C	1.3418	0.4687	−0.0022	0.086*	
C10	1.39030 (18)	0.6054 (2)	0.07729 (11)	0.0560 (7)	
H10A	1.3713	0.6544	0.1068	0.084*	
H10B	1.4615	0.6204	0.0646	0.084*	
H10C	1.3870	0.5337	0.0915	0.084*	
C11	1.3234 (2)	0.72779 (19)	0.00507 (9)	0.0584 (7)	
H11A	1.3039	0.7788	0.0335	0.088*	
H11B	1.2768	0.7350	−0.0269	0.088*	
H11C	1.3958	0.7403	−0.0066	0.088*	
C12	1.0000	0.75000	0.13460 (9)	0.0367 (7)	
H12A	0.9461	0.7186	0.1589	0.044*	0.50
H12B	1.0539	0.7814	0.1589	0.044*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0454 (10)	0.0437 (10)	0.0695 (12)	−0.0126 (7)	−0.0040 (8)	0.0090 (8)
C1	0.0448 (14)	0.0360 (13)	0.0535 (14)	−0.0042 (10)	−0.0042 (11)	0.0074 (10)
C2	0.0349 (11)	0.0302 (11)	0.0425 (12)	−0.0024 (9)	−0.0028 (9)	0.0062 (9)
C3	0.0276 (11)	0.0394 (12)	0.0408 (12)	0.0022 (9)	−0.0014 (9)	0.0069 (9)
O2	0.0334 (9)	0.0452 (10)	0.0629 (11)	−0.0046 (7)	0.0061 (7)	0.0008 (8)
C4	0.0335 (11)	0.0312 (11)	0.0350 (11)	0.0047 (9)	−0.0025 (9)	0.0020 (9)
C5	0.0345 (11)	0.0293 (11)	0.0390 (12)	0.0003 (9)	−0.0015 (9)	0.0029 (9)
C6	0.0303 (11)	0.0304 (11)	0.0388 (12)	0.0016 (8)	−0.0005 (9)	0.0038 (9)
C7	0.0378 (12)	0.0285 (11)	0.0428 (12)	0.0042 (9)	−0.0001 (9)	0.0014 (9)
C8	0.0330 (11)	0.0328 (11)	0.0478 (12)	0.0013 (9)	0.0046 (10)	−0.0002 (10)
C9	0.0472 (14)	0.0562 (16)	0.0691 (18)	−0.0016 (12)	0.0199 (13)	−0.0177 (13)
C10	0.0350 (13)	0.0681 (17)	0.0649 (17)	−0.0004 (12)	−0.0030 (12)	0.0025 (14)
C11	0.0546 (16)	0.0455 (14)	0.0750 (18)	−0.0014 (11)	0.0242 (14)	0.0121 (13)
C12	0.0348 (16)	0.0377 (17)	0.0377 (16)	0.0049 (13)	0.000	0.000

Geometric parameters (Å, º)

O1—C1	1.227 (3)	C8—C9	1.530 (3)
C1—C2	1.450 (3)	C8—C11	1.530 (3)
C1—H1	0.9500	C8—C10	1.534 (3)
C2—C7	1.395 (3)	C9—H9A	0.9800
C2—C3	1.406 (3)	C9—H9B	0.9800
C3—O2	1.363 (2)	C9—H9C	0.9800
C3—C4	1.399 (3)	C10—H10A	0.9800

O2—H2	0.92 (2)	C10—H10B	0.9800
C4—C5	1.384 (3)	C10—H10C	0.9800
C4—C12	1.511 (3)	C11—H11A	0.9800
C5—C6	1.403 (3)	C11—H11B	0.9800
C5—H5	0.9500	C11—H11C	0.9800
C6—C7	1.387 (3)	C12—C4^{i}	1.510 (3)
C6—C8	1.527 (3)	C12—H12A	0.9900
C7—H7	0.9500	C12—H12B	0.9900

O1—C1—C2	125.0 (2)	C9—C8—C10	108.7 (2)
O1—C1—H1	117.5	C11—C8—C10	109.05 (19)
C2—C1—H1	117.5	C8—C9—H9A	109.5
C7—C2—C3	119.83 (19)	C8—C9—H9B	109.5
C7—C2—C1	119.4 (2)	H9A—C9—H9B	109.5
C3—C2—C1	120.7 (2)	C8—C9—H9C	109.5
O2—C3—C4	118.67 (19)	H9A—C9—H9C	109.5
O2—C3—C2	121.52 (19)	H9B—C9—H9C	109.5
C4—C3—C2	119.81 (18)	C8—C10—H10A	109.5
C3—O2—H2	104.7 (14)	C8—C10—H10B	109.5
C5—C4—C3	117.98 (19)	H10A—C10—H10B	109.5
C5—C4—C12	120.80 (19)	C8—C10—H10C	109.5
C3—C4—C12	121.16 (19)	H10A—C10—H10C	109.5
C4—C5—C6	124.13 (19)	H10B—C10—H10C	109.5
C4—C5—H5	117.9	C8—C11—H11A	109.5
C6—C5—H5	117.9	C8—C11—H11B	109.5
C7—C6—C5	116.30 (19)	H11A—C11—H11B	109.5
C7—C6—C8	123.13 (18)	C8—C11—H11C	109.5
C5—C6—C8	120.51 (18)	H11A—C11—H11C	109.5
C6—C7—C2	121.9 (2)	H11B—C11—H11C	109.5
C6—C7—H7	119.0	C4^{i}—C12—C4	115.4 (2)
C2—C7—H7	119.0	C4^{i}—C12—H12A	108.4
C6—C8—C9	112.16 (18)	C4—C12—H12A	108.4
C6—C8—C11	110.52 (17)	C4^{i}—C12—H12B	108.4
C9—C8—C11	107.88 (19)	C4—C12—H12B	108.4
C6—C8—C10	108.48 (19)	H12A—C12—H12B	107.5

O1—C1—C2—C7	179.3 (2)	C4—C5—C6—C8	−178.80 (19)
O1—C1—C2—C3	0.1 (4)	C5—C6—C7—C2	1.6 (3)
C7—C2—C3—O2	179.23 (19)	C8—C6—C7—C2	178.56 (19)
C1—C2—C3—O2	−1.5 (3)	C3—C2—C7—C6	0.0 (3)
C7—C2—C3—C4	−1.6 (3)	C1—C2—C7—C6	−179.3 (2)
C1—C2—C3—C4	177.7 (2)	C7—C6—C8—C9	21.4 (3)
O2—C3—C4—C5	−179.34 (18)	C5—C6—C8—C9	−161.8 (2)
C2—C3—C4—C5	1.4 (3)	C7—C6—C8—C11	141.8 (2)
O2—C3—C4—C12	3.6 (3)	C5—C6—C8—C11	−41.4 (3)
C2—C3—C4—C12	−175.65 (19)	C7—C6—C8—C10	−98.7 (2)
C3—C4—C5—C6	0.2 (3)	C5—C6—C8—C10	78.1 (2)

C12—C4—C5—C6	177.35 (19)	C5—C4—C12—C4^{i}	81.9 (2)
C4—C5—C6—C7	−1.7 (3)	C3—C4—C12—C4^{i}	−101.1 (2)

Symmetry code: (i) −*x*+2, −*y*+3/2, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.92 (2)	1.80 (2)	2.645 (2)	151 (2)

(IVb) 2,2′–Dihydroxy–5,5′–di–tert–butyl–3,3′– methanediyldibenzaldehyde

Crystal data

$C_{23}H_{28}O_4$
M_r = 368.45
Monoclinic, *C*2/*c*
a = 26.809 (2) Å
b = 8.4543 (7) Å
c = 21.3720 (18) Å
β = 120.618 (1)°
V = 4168.7 (6) Å^3
Z = 8
F(000) = 1584
D_x = 1.174 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 3051 reflections
θ = 2.6–25.6°
μ = 0.08 mm^{-1}
T = 153 K
Tablet, light brown
0.48 × 0.38 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.919, T_{max} = 1.000
14739 measured reflections
3668 independent reflections
2235 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 25.0°, θ_{min} = 1.8°
h = −31→31
k = −10→10
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.132
S = 1.01
3668 reflections
251 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 4.8718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.12612 (7)	0.3427 (2)	0.17034 (10)	0.0614 (5)	
C1	0.07491 (11)	0.3578 (3)	0.12349 (14)	0.0472 (6)	
H1	0.0660	0.4217	0.0825	0.057*	
C2	0.02703 (9)	0.2858 (3)	0.12591 (12)	0.0347 (5)	
C3	0.03676 (9)	0.1916 (3)	0.18513 (12)	0.0352 (5)	
C4	−0.00989 (10)	0.1207 (3)	0.18602 (12)	0.0338 (5)	
C5	−0.06496 (10)	0.1474 (3)	0.12771 (12)	0.0356 (6)	
H5	−0.0966	0.0972	0.1278	0.043*	
C6	−0.07678 (9)	0.2444 (3)	0.06828 (12)	0.0350 (5)	
C7	−0.02974 (9)	0.3110 (3)	0.06866 (12)	0.0367 (6)	
H7	−0.0360	0.3757	0.0290	0.044*	
O2	0.09102 (7)	0.1694 (2)	0.24251 (9)	0.0448 (4)	
H2	0.1181 (10)	0.220 (3)	0.2342 (13)	0.050*	
C8	−0.13972 (10)	0.2741 (3)	0.00882 (13)	0.0424 (6)	
C9	−0.14402 (11)	0.3823 (3)	−0.05135 (13)	0.0528 (7)	
H9A	−0.1253	0.4838	−0.0302	0.079*	
H9B	−0.1849	0.4002	−0.0877	0.079*	
H9C	−0.1246	0.3321	−0.0746	0.079*	
C10	−0.16911 (11)	0.1174 (3)	−0.02543 (15)	0.0604 (8)	
H10A	−0.2094	0.1371	−0.0635	0.091*	
H10B	−0.1684	0.0486	0.0119	0.091*	
H10C	−0.1485	0.0659	−0.0468	0.091*	
C11	−0.17152 (10)	0.3556 (3)	0.04259 (14)	0.0526 (7)	
H11A	−0.1524	0.4562	0.0645	0.079*	
H11B	−0.1707	0.2872	0.0801	0.079*	
H11C	−0.2118	0.3757	0.0048	0.079*	
C12	0.0000	0.0212 (4)	0.2500	0.0396 (8)	
H12A	−0.0341	−0.0477	0.2350	0.048*	0.50
H12B	0.0341	−0.0477	0.2650	0.048*	0.50
O3	0.04622 (7)	0.8544 (2)	0.48082 (9)	0.0523 (5)	
C13	−0.00570 (11)	0.8664 (3)	0.43552 (13)	0.0432 (6)	
H13	−0.0284	0.9321	0.4475	0.052*	
C14	−0.03482 (9)	0.7891 (3)	0.36593 (12)	0.0342 (5)	
C15	−0.00390 (9)	0.6939 (3)	0.34339 (12)	0.0326 (5)	
C16	−0.03278 (9)	0.6202 (2)	0.27591 (12)	0.0315 (5)	
C17	−0.09193 (9)	0.6446 (3)	0.23255 (12)	0.0342 (5)	
H17	−0.1117	0.5928	0.1868	0.041*	
C18	−0.12444 (9)	0.7414 (3)	0.25209 (12)	0.0348 (5)	
C19	−0.09454 (9)	0.8120 (3)	0.31941 (12)	0.0358 (5)	
H19	−0.1149	0.8782	0.3348	0.043*	
O4	0.05409 (7)	0.6738 (2)	0.38596 (9)	0.0417 (4)	
H4	0.0659 (10)	0.725 (3)	0.4270 (13)	0.050*	
C20	−0.18896 (10)	0.7661 (3)	0.19921 (13)	0.0430 (6)	
C21	−0.19675 (12)	0.8506 (4)	0.13167 (15)	0.0688 (9)	
H21A	−0.1772	0.9535	0.1456	0.103*	

H21B	−0.2382	0.8663	0.0971	0.103*	
H21C	−0.1799	0.7863	0.1090	0.103*	
C22	−0.21941 (11)	0.6067 (4)	0.17775 (18)	0.0700 (9)	
H22A	−0.2609	0.6227	0.1437	0.105*	
H22B	−0.2140	0.5522	0.2213	0.105*	
H22C	−0.2030	0.5424	0.1545	0.105*	
C23	−0.21703 (11)	0.8669 (4)	0.23221 (16)	0.0645 (8)	
H23A	−0.1979	0.9703	0.2461	0.097*	
H23B	−0.2130	0.8137	0.2753	0.097*	
H23C	−0.2582	0.8814	0.1964	0.097*	
C24	0.0000	0.5208 (4)	0.2500	0.0362 (8)	
H24A	0.0277	0.4518	0.2902	0.043*	0.50
H24B	−0.0277	0.4518	0.2098	0.043*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0376 (11)	0.0847 (15)	0.0625 (12)	−0.0085 (10)	0.0259 (10)	−0.0015 (11)
C1	0.0446 (16)	0.0551 (17)	0.0491 (15)	−0.0064 (13)	0.0290 (14)	−0.0014 (13)
C2	0.0363 (13)	0.0363 (13)	0.0377 (13)	−0.0013 (10)	0.0234 (11)	−0.0038 (10)
C3	0.0356 (13)	0.0336 (13)	0.0373 (13)	0.0014 (10)	0.0193 (11)	−0.0060 (11)
C4	0.0412 (14)	0.0278 (12)	0.0365 (13)	−0.0002 (10)	0.0227 (11)	−0.0057 (10)
C5	0.0376 (13)	0.0333 (13)	0.0411 (14)	−0.0068 (10)	0.0238 (12)	−0.0078 (11)
C6	0.0371 (13)	0.0377 (13)	0.0334 (13)	−0.0020 (10)	0.0202 (11)	−0.0052 (10)
C7	0.0405 (14)	0.0394 (14)	0.0353 (13)	0.0013 (11)	0.0230 (12)	−0.0003 (11)
O2	0.0366 (10)	0.0507 (11)	0.0447 (10)	0.0028 (8)	0.0189 (8)	0.0008 (8)
C8	0.0352 (13)	0.0547 (16)	0.0392 (14)	−0.0014 (12)	0.0203 (11)	−0.0043 (12)
C9	0.0445 (15)	0.072 (2)	0.0403 (15)	0.0080 (14)	0.0204 (12)	0.0077 (14)
C10	0.0465 (16)	0.068 (2)	0.0548 (17)	−0.0080 (14)	0.0174 (14)	−0.0129 (15)
C11	0.0404 (15)	0.0721 (19)	0.0497 (16)	0.0072 (13)	0.0262 (13)	0.0012 (14)
C12	0.049 (2)	0.0293 (18)	0.042 (2)	0.000	0.0250 (17)	0.000
O3	0.0485 (11)	0.0675 (13)	0.0395 (10)	−0.0082 (9)	0.0214 (9)	−0.0079 (9)
C13	0.0490 (16)	0.0464 (15)	0.0411 (14)	−0.0042 (12)	0.0280 (13)	−0.0036 (12)
C14	0.0397 (13)	0.0352 (13)	0.0329 (12)	−0.0042 (10)	0.0223 (11)	0.0003 (10)
C15	0.0332 (13)	0.0332 (13)	0.0363 (13)	−0.0021 (10)	0.0214 (11)	0.0048 (10)
C16	0.0379 (13)	0.0267 (12)	0.0373 (13)	−0.0019 (10)	0.0246 (11)	0.0015 (10)
C17	0.0386 (13)	0.0309 (13)	0.0389 (13)	−0.0076 (10)	0.0239 (11)	−0.0026 (10)
C18	0.0345 (13)	0.0361 (13)	0.0393 (13)	−0.0040 (10)	0.0228 (11)	−0.0004 (11)
C19	0.0398 (13)	0.0332 (13)	0.0429 (14)	−0.0011 (10)	0.0273 (12)	−0.0003 (11)
O4	0.0357 (9)	0.0492 (11)	0.0382 (9)	0.0016 (8)	0.0175 (8)	0.0007 (8)
C20	0.0340 (13)	0.0520 (16)	0.0455 (14)	−0.0022 (11)	0.0221 (12)	−0.0032 (12)
C21	0.0496 (17)	0.097 (2)	0.0554 (18)	0.0130 (16)	0.0237 (15)	0.0161 (17)
C22	0.0390 (16)	0.071 (2)	0.093 (2)	−0.0143 (15)	0.0291 (16)	−0.0139 (18)
C23	0.0382 (15)	0.085 (2)	0.070 (2)	0.0089 (15)	0.0274 (14)	−0.0112 (17)
C24	0.0411 (19)	0.0281 (17)	0.046 (2)	0.000	0.0270 (16)	0.000

Geometric parameters (Å, º)

O1—C1	1.226 (3)	O3—C13	1.230 (3)
C1—C2	1.445 (3)	C13—C14	1.437 (3)
C1—H1	0.9500	C13—H13	0.9500
C2—C7	1.401 (3)	C14—C19	1.403 (3)
C2—C3	1.403 (3)	C14—C15	1.405 (3)
C3—O2	1.356 (3)	C15—O4	1.354 (3)
C3—C4	1.396 (3)	C15—C16	1.389 (3)
C4—C5	1.382 (3)	C16—C17	1.385 (3)
C4—C12	1.509 (3)	C16—C24	1.510 (3)
C5—C6	1.406 (3)	C17—C18	1.405 (3)
C5—H5	0.9500	C17—H17	0.9500
C6—C7	1.377 (3)	C18—C19	1.377 (3)
C6—C8	1.530 (3)	C18—C20	1.524 (3)
C7—H7	0.9500	C19—H19	0.9500
O2—H2	0.94 (2)	O4—H4	0.88 (2)
C8—C10	1.524 (4)	C20—C22	1.520 (4)
C8—C11	1.533 (3)	C20—C23	1.527 (3)
C8—C9	1.534 (3)	C20—C21	1.526 (4)
C9—H9A	0.9800	C21—H21A	0.9800
C9—H9B	0.9800	C21—H21B	0.9800
C9—H9C	0.9800	C21—H21C	0.9800
C10—H10A	0.9800	C22—H22A	0.9800
C10—H10B	0.9800	C22—H22B	0.9800
C10—H10C	0.9800	C22—H22C	0.9800
C11—H11A	0.9800	C23—H23A	0.9800
C11—H11B	0.9800	C23—H23B	0.9800
C11—H11C	0.9800	C23—H23C	0.9800
C12—C4^{i}	1.509 (3)	C24—C16^{i}	1.510 (3)
C12—H12A	0.9900	C24—H24A	0.9900
C12—H12B	0.9900	C24—H24B	0.9900
O1—C1—C2	124.9 (2)	O3—C13—C14	125.3 (2)
O1—C1—H1	117.5	O3—C13—H13	117.4
C2—C1—H1	117.5	C14—C13—H13	117.4
C7—C2—C3	119.6 (2)	C19—C14—C15	119.7 (2)
C7—C2—C1	119.6 (2)	C19—C14—C13	119.5 (2)
C3—C2—C1	120.7 (2)	C15—C14—C13	120.7 (2)
O2—C3—C4	119.0 (2)	O4—C15—C16	118.93 (19)
O2—C3—C2	121.0 (2)	O4—C15—C14	121.2 (2)
C4—C3—C2	120.0 (2)	C16—C15—C14	119.9 (2)
C5—C4—C3	118.1 (2)	C17—C16—C15	118.1 (2)
C5—C4—C12	121.46 (18)	C17—C16—C24	121.28 (18)
C3—C4—C12	120.42 (18)	C15—C16—C24	120.63 (18)
C4—C5—C6	123.8 (2)	C16—C17—C18	124.1 (2)
C4—C5—H5	118.1	C16—C17—H17	118.0
C6—C5—H5	118.1	C18—C17—H17	118.0

C7—C6—C5	116.6 (2)	C19—C18—C17	116.4 (2)
C7—C6—C8	123.9 (2)	C19—C18—C20	123.5 (2)
C5—C6—C8	119.5 (2)	C17—C18—C20	120.1 (2)
C6—C7—C2	121.8 (2)	C18—C19—C14	121.8 (2)
C6—C7—H7	119.1	C18—C19—H19	119.1
C2—C7—H7	119.1	C14—C19—H19	119.1
C3—O2—H2	110.1 (15)	C15—O4—H4	108.5 (16)
C10—C8—C6	109.8 (2)	C22—C20—C18	109.5 (2)
C10—C8—C11	110.0 (2)	C22—C20—C23	108.6 (2)
C6—C8—C11	108.98 (19)	C18—C20—C23	112.0 (2)
C10—C8—C9	108.4 (2)	C22—C20—C21	109.6 (2)
C6—C8—C9	111.98 (19)	C18—C20—C21	108.81 (19)
C11—C8—C9	107.7 (2)	C23—C20—C21	108.3 (2)
C8—C9—H9A	109.5	C20—C21—H21A	109.5
C8—C9—H9B	109.5	C20—C21—H21B	109.5
H9A—C9—H9B	109.5	H21A—C21—H21B	109.5
C8—C9—H9C	109.5	C20—C21—H21C	109.5
H9A—C9—H9C	109.5	H21A—C21—H21C	109.5
H9B—C9—H9C	109.5	H21B—C21—H21C	109.5
C8—C10—H10A	109.5	C20—C22—H22A	109.5
C8—C10—H10B	109.5	C20—C22—H22B	109.5
H10A—C10—H10B	109.5	H22A—C22—H22B	109.5
C8—C10—H10C	109.5	C20—C22—H22C	109.5
H10A—C10—H10C	109.5	H22A—C22—H22C	109.5
H10B—C10—H10C	109.5	H22B—C22—H22C	109.5
C8—C11—H11A	109.5	C20—C23—H23A	109.5
C8—C11—H11B	109.5	C20—C23—H23B	109.5
H11A—C11—H11B	109.5	H23A—C23—H23B	109.5
C8—C11—H11C	109.5	C20—C23—H23C	109.5
H11A—C11—H11C	109.5	H23A—C23—H23C	109.5
H11B—C11—H11C	109.5	H23B—C23—H23C	109.5
C4^{i}—C12—C4	112.3 (3)	C16^{i}—C24—C16	112.4 (2)
C4^{i}—C12—H12A	109.2	C16^{i}—C24—H24A	109.1
C4—C12—H12A	109.2	C16—C24—H24A	109.1
C4^{i}—C12—H12B	109.2	C16^{i}—C24—H24B	109.1
C4—C12—H12B	109.2	C16—C24—H24B	109.1
H12A—C12—H12B	107.9	H24A—C24—H24B	107.9

O1—C1—C2—C7	−179.5 (2)	O3—C13—C14—C19	179.5 (2)
O1—C1—C2—C3	0.8 (4)	O3—C13—C14—C15	−2.0 (4)
C7—C2—C3—O2	−177.7 (2)	C19—C14—C15—O4	177.66 (19)
C1—C2—C3—O2	2.0 (3)	C13—C14—C15—O4	−0.8 (3)
C7—C2—C3—C4	1.4 (3)	C19—C14—C15—C16	−1.4 (3)
C1—C2—C3—C4	−178.9 (2)	C13—C14—C15—C16	−179.9 (2)
O2—C3—C4—C5	178.70 (19)	O4—C15—C16—C17	−178.75 (19)
C2—C3—C4—C5	−0.4 (3)	C14—C15—C16—C17	0.4 (3)
O2—C3—C4—C12	0.1 (3)	O4—C15—C16—C24	−0.7 (3)
C2—C3—C4—C12	−179.0 (2)	C14—C15—C16—C24	178.5 (2)

C3—C4—C5—C6	−1.4 (3)	C15—C16—C17—C18	1.0 (3)
C12—C4—C5—C6	177.2 (2)	C24—C16—C17—C18	−177.0 (2)
C4—C5—C6—C7	2.1 (3)	C16—C17—C18—C19	−1.3 (3)
C4—C5—C6—C8	−176.2 (2)	C16—C17—C18—C20	177.5 (2)
C5—C6—C7—C2	−1.1 (3)	C17—C18—C19—C14	0.1 (3)
C8—C6—C7—C2	177.2 (2)	C20—C18—C19—C14	−178.6 (2)
C3—C2—C7—C6	−0.6 (3)	C15—C14—C19—C18	1.2 (3)
C1—C2—C7—C6	179.7 (2)	C13—C14—C19—C18	179.7 (2)
C7—C6—C8—C10	122.3 (2)	C19—C18—C20—C22	−125.2 (3)
C5—C6—C8—C10	−59.5 (3)	C17—C18—C20—C22	56.1 (3)
C7—C6—C8—C11	−117.2 (2)	C19—C18—C20—C23	−4.6 (3)
C5—C6—C8—C11	61.0 (3)	C17—C18—C20—C23	176.7 (2)
C7—C6—C8—C9	1.8 (3)	C19—C18—C20—C21	115.0 (3)
C5—C6—C8—C9	−180.0 (2)	C17—C18—C20—C21	−63.7 (3)
C5—C4—C12—C4^{i}	−100.4 (2)	C17—C16—C24—C16^{i}	101.4 (2)
C3—C4—C12—C4^{i}	78.14 (18)	C15—C16—C24—C16^{i}	−76.67 (18)

Symmetry code: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.94 (2)	1.81 (2)	2.625 (3)	144 (2)
O4—H4···O3	0.88 (2)	1.85 (2)	2.631 (2)	147 (2)
C13—H13···O3^{ii}	0.95	2.57	3.454 (3)	156

Symmetry code: (ii) −*x*, −*y*+2, −*z*+1.