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(2*E*,6*E*)-2,6-Bis(3,5-di-*tert*-butyl-4-hydroxybenzylidene)cyclohexanone acetic acid solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.155
Data-to-parameter ratio = 18.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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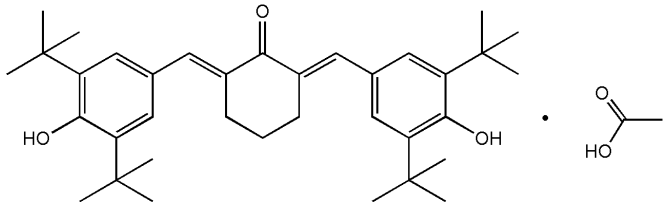
The title compound, $C_{36}H_{50}O_3\cdot C_2H_4O_2$, crystallizes with acetic acid solvent molecules in a 1.1 stoichiometric molar ratio. The molecules are connected by intermolecular O—H···O hydrogen bonds, resulting in zigzag chains running along the [101] axis.

Comment

The title compound, (I) (Fig. 1), exhibits antioxidative properties (Sardjiman *et al.*, 1997). We report here its crystal structure.



(I)

The X-ray study confirms the previously proposed molecular structure based on spectroscopic data. The C—O and C—C distances show no unusual values (Table 1). In the crystal structure there are intramolecular O—H···O hydrogen bonds between the hydroxyl groups and the carboxylic O atoms of the acetic acid molecule, and O—H···O hydrogen bonds between the hydroxyl groups and the carbonyl O atom (Table 2), resulting in zigzag chains along the [101] axis (Fig. 2).

Experimental

The title compound was synthesized using a published procedure (Sardjiman *et al.*, 1997; Youssef *et al.*, 2004). Crystals were obtained by recrystallization from acetic acid and water.

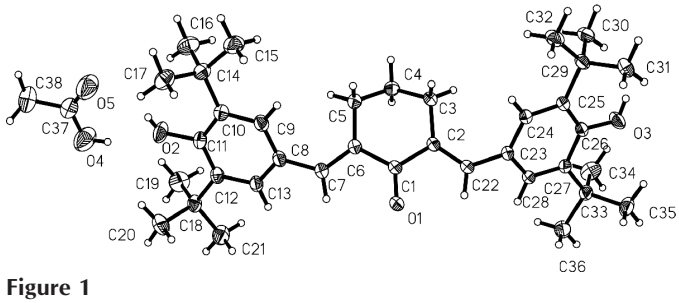


Figure 1
Perspective view of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Crystal data

$C_{36}H_{50}O_3\cdot C_2H_4O_2$
M_r = 590.81
Monoclinic, $P2_1/n$
a = 12.838 (7) Å
b = 16.250 (9) Å
c = 17.221 (9) Å
β = 102.376 (11)°
V = 3509 (3) Å^3
Z = 4
D_x = 1.118 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 970 reflections
θ = 2.2–27.0°
μ = 0.07 mm^{-1}
T = 293 (2) K
Block, brown
0.50 × 0.46 × 0.41 mm

Data collection

Bruker AXS SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.968, T_{max} = 0.970
19669 measured reflections
7661 independent reflections
5405 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 27.1°
$h = -16 \rightarrow 15$
$k = -12 \rightarrow 20$
$l = -18 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.155
S = 1.03
7661 reflections
416 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 1.1965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.2239 (19)	C4—C5	1.515 (2)
O2—C11	1.376 (2)	C5—C6	1.504 (2)
O3—C26	1.3634 (19)	C6—C7	1.337 (2)
C2—C22	1.338 (2)	C7—C8	1.464 (2)
C3—C4	1.510 (2)	C22—C23	1.464 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*C*···O1^{i}	0.82	2.21	2.801 (2)	129
O2—H2···O5	0.71 (4)	2.25 (4)	2.832 (3)	140 (5)
O4—H4*C*···O2	0.82	2.06	2.851 (3)	162

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$.

Atom H2, H16*A*–*C*, and H17*A*–*C* H atoms were refined without constraints. All other H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å, O—H = 0.82 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

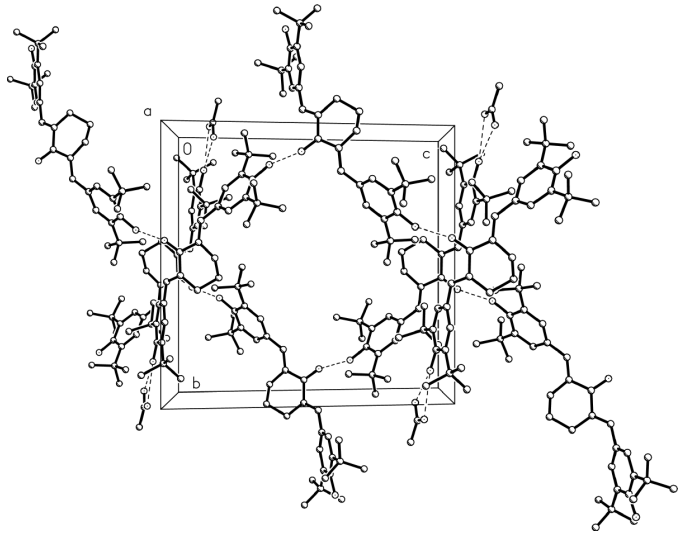


Figure 2
The molecular packing of (I), viewed along the *b* axis. All H atoms attached to C atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

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