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3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde

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Key indicators

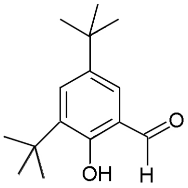
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.165
Data-to-parameter ratio = 13.3

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

The title compound, $C_{15}H_{22}O_2$, crystallizes in the monoclinic space group $P2_1/n$ with two crystallographically independent molecules in the asymmetric unit. Intramolecular O—H···O hydrogen bonds are found, forming six-membered rings.

Comment

3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde, (I), with two bulky alkyl groups, is a very useful intermediate in organic synthesis. For example, some chiral salen-type Schiff base complexes, which can be easily obtained by its condensation with chiral diamines, have exhibited excellent asymmetric catalytic properties in a variety of reactions (Tokunaga *et al.*, 1997; Canali & Sherrington, 1999; Sigman & Jacobsen, 1998; Belokon *et al.*, 2000). Until now, many metal complexes containing the 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde moiety have been structurally elucidated (Anderson & Eisenberg, 1994; Leung *et al.*, 1996; Chichak *et al.*, 2002; Wang *et al.*, 2002); this is helpful for studies of the asymmetric catalytic mechanism. Duff (1941) has established a practical formylation approach to the synthesis of (I), using 2,4-di-*tert*-butylphenol and hexamethylenetetramine as starting materials; however, its crystal structure has not yet been reported. Here, we have synthesized (I) and determined its crystal structure.



(I)

The atom-numbering scheme of (I) is shown in Fig. 1, while selected bond distances and bond angles are given in Table 1. There are two crystallographically independent molecules of (I) in the asymmetric unit of the monoclinic space group $P2_1/n$. The geometries of the phenol ring and the formyl group are in the normal ranges. However, half of the *tert*-butyl groups are found to be disordered over two positions.

Intramolecular hydrogen-bonding interactions are observed between the phenol O atoms (O2 and O4) and the carbonyl O atoms (O1 and O3) (Table 2), forming a six-membered ring in each case. In the crystal packing, all the aromatic rings are parallel to each other, forming an ordered layer structure. Nevertheless, there are no π–π stacking interactions between them, due to the steric repulsions of the *tert*-butyl groups (Fig. 2).

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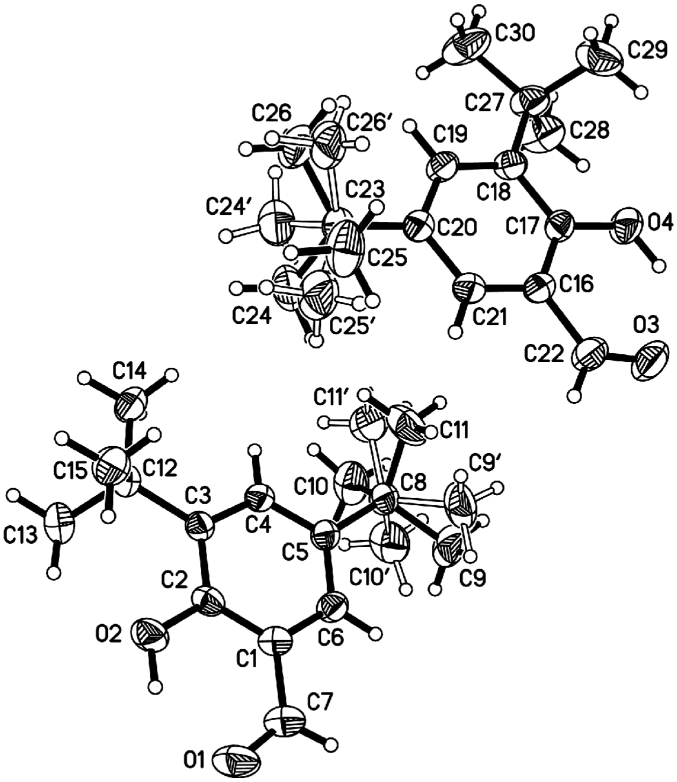

Figure 1
The structure of the asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Both components of the disordered methyl groups are shown.

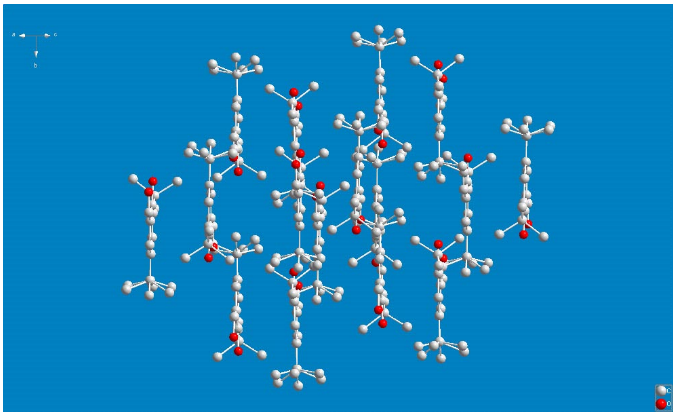

Figure 2
The layer structure of the title complex. H atoms have been omitted for clarity. Both components of the disordered methyl groups are shown.

Experimental

3,5-Di-*tert*-butyl-2-hydroxybenzaldehyde was prepared according to the literature method of Larrow *et al.* (1994). The resulting yellow solid was collected in 40% yield [m.p. 330–331 K; literature 331–333 K (Claus *et al.*, 1972)]. Analysis calculated for $C_{15}H_{22}O_2$: C 76.87, H 9.47%; found: C 76.80, H,9.57%. IR (KBr, cm^{-1}): 2959 (*s*), 2905 (*s*), 2870 (*s*), 2843 (*s*), 2739 (*m*), 1650 (*s*), 1612 (*s*), 1439 (*w*), 1381 (*s*), 1246 (*m*), 1170 (*s*). ^1H NMR ($CDCl_3$): 1.26 (*s*, 9H, ^{t}Bu), 1.36 (*s*, 9H, ^{t}Bu), 7.28 (*d*, 1H, ArH), 7.53 (*d*, 1H, ArH), 9.80 (*s*, 1H, OH), 11.57 (*s*, 1H, CHO). The single-crystal sample suitable for X-ray diffraction was obtained by slow evaporation in air of a solution in methanol.

Crystal data

$C_{15}H_{22}O_2$
$M_r = 234.33$
Monoclinic, $P2_1/n$
$a = 11.820\ (3)$ Å
$b = 10.175\ (3)$ Å
$c = 24.169\ (7)$ Å
$\beta = 96.512\ (4)°$
$V = 2887.9\ (13)$ Å^3
$Z = 8$

$D_x = 1.078$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 933 reflections
$\theta = 2.6$–$27.8°$
$\mu = 0.07$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
$0.50 \times 0.40 \times 0.30$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.968$, $T_{\max} = 0.979$
13 667 measured reflections

5071 independent reflections
3241 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 25.0°$
$h = -14 \rightarrow 13$
$k = -12 \rightarrow 5$
$l = -28 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.165$
$S = 1.01$
5071 reflections
381 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.453 (2)	C16—C22	1.450 (2)
C2—O2	1.3555 (19)	C17—O4	1.354 (2)
C7—O1	1.218 (2)	C22—O3	1.217 (2)
O1—C7—C1	125.5 (2)	O3—C22—C16	125.87 (19)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1	0.93	1.89	2.6045 (19)	132
O4—H4*B*···O3	0.93	1.89	2.6029 (19)	132

Atoms C9/C10/C11 and C24/C25/C26 were found to be disordered over two positions, with site-occupancy factors 0.724 (4):0.276 (4) and 0.750 (5):0.250 (5), respectively. All H atoms were treated as riding atoms, with methyl C—H = 0.96 Å, and other C—H and O—H = 0.93 Å. $U_{\rm iso}$(H) values were set equal to $1.5U_{\rm eq}$ of the carrier atom for methyl H atoms and H atoms attached to O; for other H atoms attached to C atoms, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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