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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.039
 wR factor = 0.109
Data-to-parameter ratio = 12.0

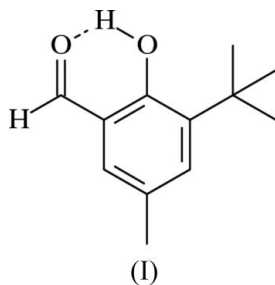
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-*tert*-Butyl-2-hydroxy-5-methylbenzaldehyde:
a redetermination

In the molecule of the title compound, $\text{C}_{12}\text{H}_{16}\text{O}_2$, all atoms except one methyl group and two H atoms are located on a crystallographic mirror plane; thus the molecule is essentially planar. An intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond and weak $\text{C}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds stabilize the packing. The structure has been previously reported in the non-centrosymmetric space group $P2_1$; our study shows that the correct space group is $P2_1/m$.

Comment

Hydroxy-substituted benzaldehyde reagents used for condensation with primary amines, hydrazines, hydroxylamine and other primary amine derivatives afford imine derivatives which can function as ligands towards a number of metal cations (Loudon, 2002; Khandar & Nejati, 2000; Khandar & Rezvani, 1999).



The structure of (I) has been previously reported [Cambridge Structural Database (Version 5.26; Allen, 2002) refcode ZOJRAR (Matilainen *et al.*, 1996)]; however, the choice of the space group $P2_1$ appears to be wrong and the correct space group is $P2_1/m$. All atoms except for one methyl group and two H atoms are located on a crystallographic mirror plane; thus the molecule except for one methyl group is essentially planar (Fig. 1). A significant intramolecular interaction is noted, involving phenol atom H1 and carbonyl atom O2, such that a six-membered ring is formed (Table 1 and Fig. 1). The aromatic ring C—C bond distances in (I) agree with those observed in bis(3-formyl-4-hydroxy-5-methoxyphenyl)methane (Odabaşoğlu *et al.*, 2006). The C7—O1 bond distance in (I) is also consistent with the value of the C=O double bond in carbonyl compounds (Loudon, 2002).

Experimental

A mixture of 2-*tert*-butyl-4-methylphenol (0.1 mol) and CHCl_3 (0.4 mol) and NaOH (0.8 mol) in ethyl alcohol was stirred at reflux temperature for 2 h. 20% H_2SO_4 solution (50 ml) was added to this

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reaction mixture and the crude product was purified with a neutral alumina column. Well shaped crystals of (I) were obtained by slow evaporation of an ethyl alcohol solution (yield 0.96 g, 5%, m.p. 345 K).

Crystal data

$C_{12}H_{16}O_2$
 $M_r = 192.25$
 Monoclinic, $P2_1/m$
 $a = 8.3633$ (8) Å
 $b = 6.6200$ (6) Å
 $c = 9.7328$ (11) Å
 $\beta = 97.170$ (9)°
 $V = 534.64$ (9) Å³
 $Z = 2$

$D_x = 1.194$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5006 reflections
 $\theta = 3.0$ – 28.8°
 $\mu = 0.08$ mm⁻¹
 $T = 100$ K
 Prism, yellow
 $0.77 \times 0.50 \times 0.20$ mm

Data collection

Stoe IPDS-II diffractometer
 φ scans
 5006 measured reflections
 1136 independent reflections
 1037 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$
 $\theta_{max} = 26.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -8 \rightarrow 8$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.109$
 $S = 1.07$
 1136 reflections
 95 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.1473P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.30$ e Å⁻³
 $\Delta\rho_{min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2–H2 \cdots O1	0.85 (1)	1.80 (1)	2.5954 (16)	155 (2)
C4–H4 \cdots O1 ⁱ	0.93	2.49	3.3717 (18)	159

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

All H atoms were located in a Fourier difference map, but the H atoms of the aromatic ring and the C9 methyl group were then placed in calculated positions and refined as riding, with C–H distances in the range 0.93 (2)–0.96 (2) Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms. The positions of the H atoms on O2 (which takes part in a hydrogen bond) and on C10 and C11 were refined using C–H and O–H restraints of 0.96 (2) and 0.85 (2) Å, respectively, and with $U_{iso}(H) = 1.5U_{eq}(methyl\ C)$ and $U_{iso}(H) = 1.2U_{eq}(O)$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

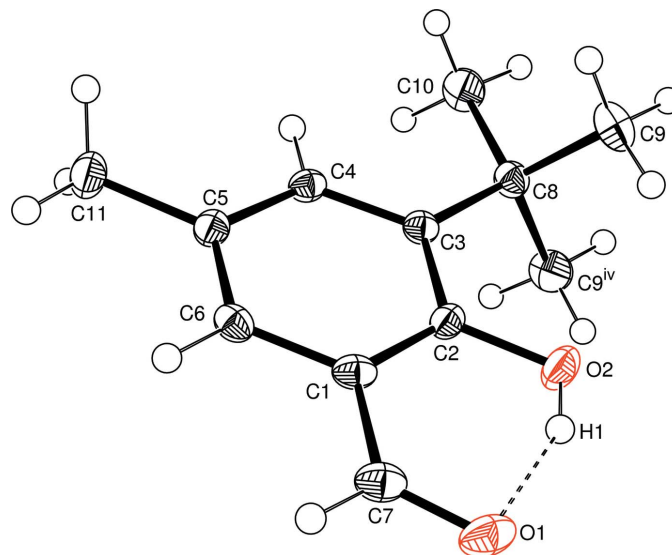


Figure 1

A view of (I), showing the atomic numbering scheme and the intramolecular hydrogen bond as a dashed line. Displacement ellipsoids are drawn at the 50% probability level.

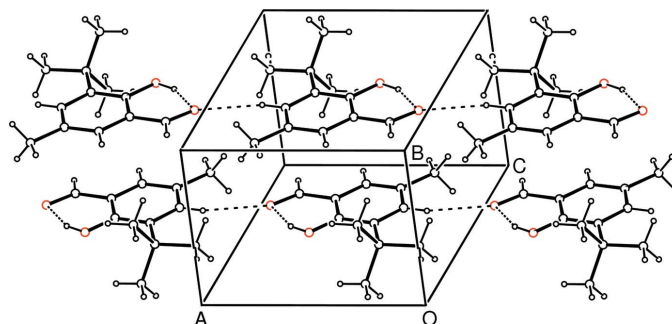


Figure 2

A packing diagram of (I), showing the hydrogen-bonding scheme (dashed lines).

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