

2-Hydroxy-3,5-diisopropylbenzoic acid

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Key indicators

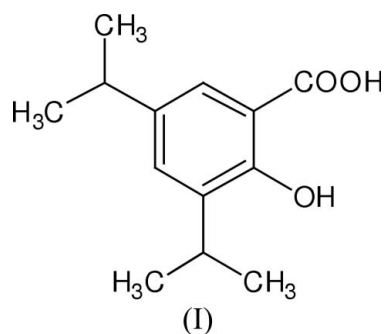
Single-crystal X-ray study
 $T = 173$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 Disorder in main residue
 R factor = 0.050
 wR factor = 0.136
 Data-to-parameter ratio = 15.6

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

The title compound, $\text{C}_{13}\text{H}_{18}\text{O}_3$, crystallizes with three independent molecules in the asymmetric unit. In the crystal structure, two of the molecules form a hydrogen-bonded dimer, while the third forms a centrosymmetric hydrogen-bonded dimer with its symmetry-equivalent molecule. In each independent molecule, there is an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond.

Comment

Substituted hydroxybenzoic acids are used for the preparation of coumarin derivatives, which are known antibacterials (Friedman *et al.*, 2003). Benzoic acid was among the first organic crystal structures to be examined by the application of X-ray crystallography (Bragg, 1921, 1922). Since then, numerous structures of benzoic acid and its derivatives have been reported (Sim *et al.*, 1955; Parkin *et al.*, 2005), owing to their importance in synthetic medicinal chemistry. The intermolecular association of carboxylic acid dimers in the supramolecular assembly of 2-(2-phenylethyl)benzoic acid has been reported (Anilkumar *et al.*, 2005). In view of the importance of the title compound, (I), its crystal structure is reported here.



In the structure of (I), there are three independent molecules in the asymmetric unit. A view of one of the independent molecules is shown in Fig. 1. In each molecule, bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27, November 2005, updated August 2006; *MOGUL*, Version 1.1; Allen, 2002). The geometries of the three molecules in the asymmetric unit are almost identical and their molecular conformations are stabilized by intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1).

In the crystal structure of (I), two symmetry-independent molecules form a hydrogen-bonded dimer, while the third molecule forms a centrosymmetric hydrogen-bonded dimer with its symmetry-equivalent molecule (Fig. 2).

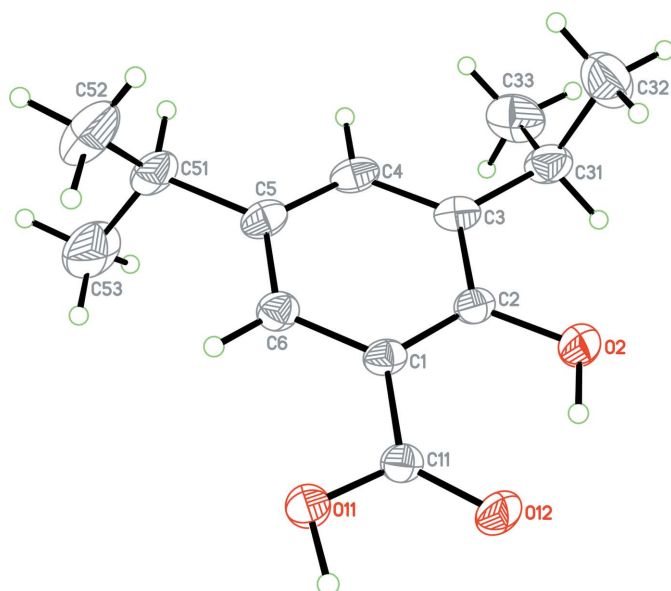


Figure 1
A view of one of the independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level.

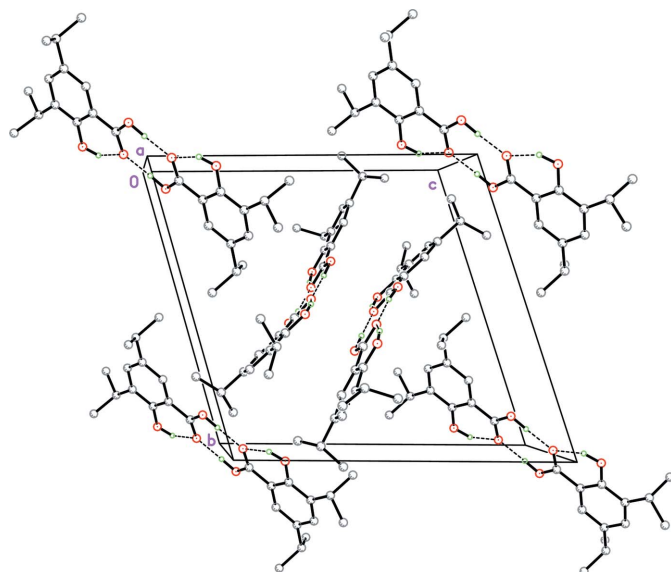


Figure 2
Part of the crystal structure of (I), viewed approximately perpendicular to the *bc* plane. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted. Only one disorder component is shown.

Experimental

Salicylic acid (13.8 g, 0.1 mol) and propan-2-ol (12 g, 0.2 mol) were heated with concentrated sulfuric acid (25 ml) at 343–353 K for 1 h, and the resulting mixture was then cooled and added to crushed ice. The resulting mass was filtered off and crystallized from ethanol. X-ray diffraction quality crystals were obtained by slow evaporation of an acetone solution of (I) (yield 50%; m.p. 391 K). Analysis for $C_{13}H_{18}O_3$, found (calculated): C 70.10 (70.24%), H 8.11 (8.16%).

Crystal data

$C_{13}H_{18}O_3$
 $M_r = 222.27$
 Triclinic, $P\bar{1}$
 $a = 9.2545$ (7) Å
 $b = 14.7627$ (13) Å
 $c = 15.2767$ (12) Å
 $\alpha = 72.823$ (7)°
 $\beta = 78.217$ (6)°
 $\gamma = 76.792$ (7)°

$V = 1920.2$ (3) Å³
 $Z = 6$
 $D_x = 1.153$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 173$ (2) K
 Block, colourless
 0.43 × 0.41 × 0.38 mm

Data collection

Stoe IPDSII two-circle
 diffractometer
 ω scans
 Absorption correction: none
 17863 measured reflections

7147 independent reflections
 5551 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.049$
 $\theta_{max} = 25.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.136$
 $S = 1.05$
 7147 reflections
 457 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.3735P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.31$ e Å⁻³
 $\Delta\rho_{min} = -0.38$ e Å⁻³
 Extinction correction: *SHELXL97*
 (Sheldrick, 1997)
 Extinction coefficient: 0.0136 (19)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2...O12	0.97 (3)	1.76 (3)	2.6162 (19)	146 (3)
O11—H11...O12 ⁱ	0.96 (3)	1.68 (3)	2.6374 (17)	174 (2)
O2A—H2A...O12A	0.87 (3)	1.87 (3)	2.6417 (16)	147 (3)
O11A—H11A...O12B	0.96 (3)	1.74 (3)	2.6891 (15)	175 (2)
O2B—H2B...O12B	0.89 (3)	1.83 (3)	2.6420 (16)	150 (2)
O11B—H11B...O12A	0.89 (2)	1.79 (2)	2.6724 (15)	172 (2)

Symmetry code: (i) $-x + 2, -y, -z$.

H atoms were found in a difference map, but those bonded to C atoms were positioned geometrically and allowed to ride on their parent C atoms at distances ranging from 0.95 to 1.00 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$. H atoms bonded to O atoms were refined independently with isotropic displacement parameters. In one independent molecule, two of the methyl groups of an isopropyl group are disordered over two sites with refined occupancy factors of 0.502 (10) and 0.498 (10). The disordered C atoms were refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL* and *PLATON* (Spek, 2003).

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