

Ethyl 5-bromosalicylate

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Key indicators

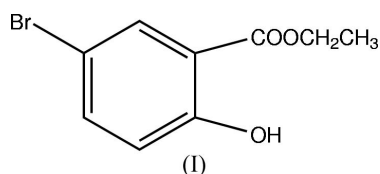
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.041
 wR factor = 0.116
Data-to-parameter ratio = 16.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_9\text{H}_9\text{BrO}_3$, an intramolecular hydrogen bond exists between the hydroxyl group and the O atom of the carbonyl group. The molecules are stacked along the b axis with significant π - π interactions.

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Comment

Salicylate compounds have displayed a broad range of biological activities (Yuan & Tsao, 1994) and useful properties (Liu *et al.*, 1996). As part of our ongoing studies (Jin *et al.*, 2004) on active salicylate and its complexes, the preparation and crystal structure determination of the title compound, (I), was undertaken.



The bond lengths and angles in (I) show normal values (Table 1). The non-H atoms of the molecule lie in a plane with an r.m.s. deviation of 0.061 \AA . An intramolecular hydrogen bond is observed between the hydroxyl group and the O atom of the carbonyl group (Fig. 1 and Table 2). The molecules

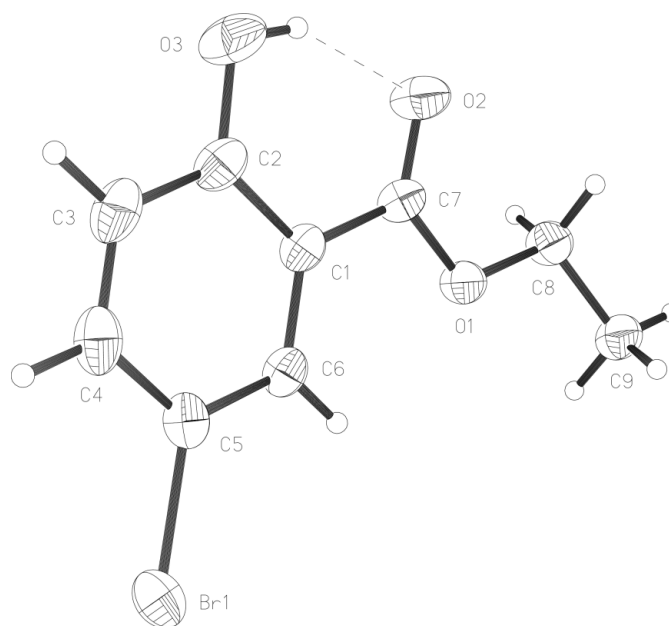


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 30% probability level. The hydrogen bond is indicated by a dashed line.

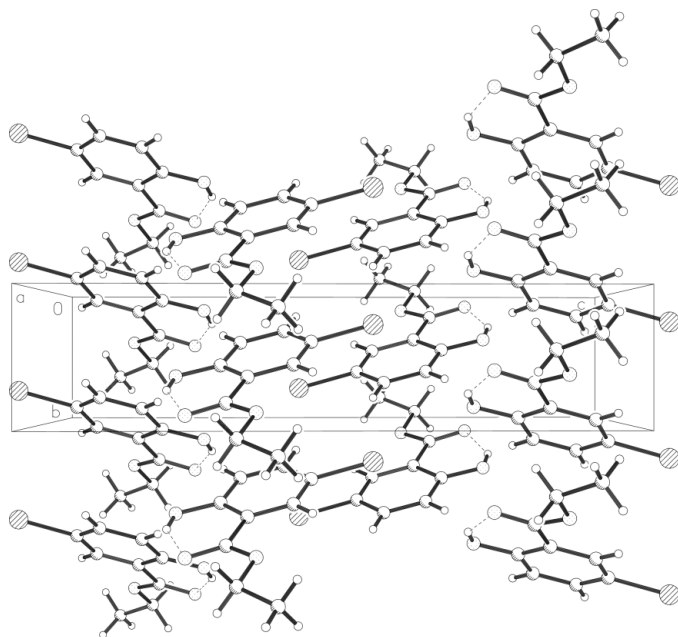


Figure 2
The packing of (I). Hydrogen bonds are indicated by dashed lines.

translated one unit cell along the *b*-axis direction are stacked with π - π interactions, with minimum short contacts for C2...C3ⁱ [3.592 (5) Å], C6...C5ⁱ [3.596 (4) Å] and C7...C7ⁱ [3.558 (5) Å] [symmetry code (i): *x*, 1 + *y*, *z*].

Experimental

The title compound, (I), was synthesized according to the literature procedure of Bartlett & Trachtenberg (1958). A crystal suitable for X-ray analysis was grown from a solution in methanol at room temperature by slow evaporation.

Crystal data

C ₉ H ₉ BrO ₃	$D_x = 1.727 \text{ Mg m}^{-3}$
$M_r = 245.07$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1695 reflections
$a = 13.169 (2) \text{ \AA}$	$\theta = 2.3\text{--}25.5^\circ$
$b = 4.0582 (6) \text{ \AA}$	$\mu = 4.33 \text{ mm}^{-1}$
$c = 17.651 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 92.121 (2)^\circ$	Prism, colourless
$V = 942.6 (3) \text{ \AA}^3$	0.40 × 0.20 × 0.10 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2047 independent reflections
φ and ω scans	1459 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.024$
$T_{\text{min}} = 0.388$, $T_{\text{max}} = 0.647$	$\theta_{\text{max}} = 27.0^\circ$
5088 measured reflections	$h = -15 \rightarrow 16$
	$k = -5 \rightarrow 4$
	$l = -22 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.117$
 $S = 1.01$
 2047 reflections
 123 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0704P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.96 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

Table 1
Selected geometric parameters (Å, °).

Br1—C5	1.902 (3)	O2—C7	1.213 (4)
O1—C7	1.324 (4)	C8—C9	1.481 (4)
O1—C8	1.462 (3)	C2—O3	1.360 (4)
C1—C7	1.481 (4)		
O2—C7—O1	122.9 (3)	O2—C7—C1	124.1 (3)
C7—O1—C8—C9	167.5 (3)	C2—C1—C7—O2	−0.2 (5)
C8—O1—C7—C1	178.1 (3)	C6—C1—C7—O1	0.9 (4)

Table 2
Hydrogen-bonding 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>
O3—H3...O2	0.89 (4)	1.84 (4)	2.625 (4)	146 (4)

The hydroxyl H atom was located in a difference Fourier map and refined isotropically [O—H = 0.89 (4) Å]. All other H atoms were included in the riding-model approximation, with C—H distances of 0.93 (aromatic H atoms), 0.96 (methyl H atoms) and 0.97 Å (methylene H atoms). The isotropic displacement parameters were set equal to $1.2U_{\text{eq}}$ of the carrier atom for the aromatic and methylene H atoms and to $1.5U_{\text{eq}}$ of the carrier for methyl H atoms.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL-NT (Bruker, 2001); software used to prepare material for publication: SHELXTL-NT.

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