

Ethyl 5-nitrosalicylate

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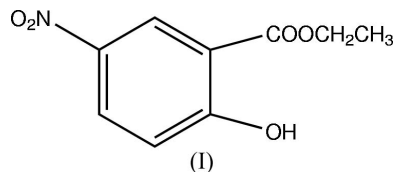
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.062
 wR factor = 0.219
Data-to-parameter ratio = 14.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_9\text{H}_9\text{NO}_5$, an intramolecular hydrogen bond exists between the hydroxyl group and the O atom of the carbonyl group. In the crystal structure, the molecules are linked to form centrosymmetrically related hydrogen-bonded dimers. The dimers are stacked along the a axis with significant $\pi-\pi$ interactions.

Comment

It is known that many compounds in the salicylate series display a broad range of biological activities (Yuan & Tsao, 1994). Also, many synthetic salicylate compounds have shown useful properties (Liu *et al.*, 1996). Furthermore, these derivatives are also interesting ligands, coordinating through the ester group to metal ions. All these properties stimulated us to find new methods to synthesize them and study their structures and activities. We report here the crystal structure of the title compound, (I).



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 0.047 Å. An intramolecular hydrogen bond

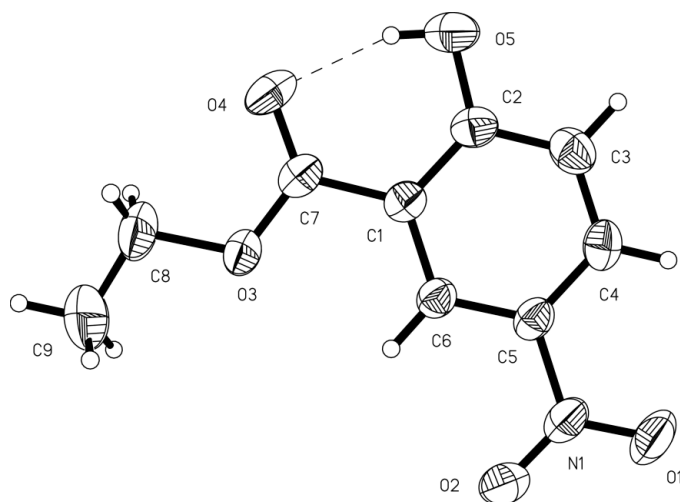


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 30% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

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bond is observed between the hydroxyl group and the O atom of the carbonyl group (Fig. 1 and Table 2). In the crystal structure, centrosymmetrically related molecules form dimeric pairs through intermolecular O—H...O hydrogen bonds (Fig. 2). Within a dimer, an O4...O4(1 - x, -y, -z) short contact of 2.827 (3) Å is observed. The dimers are stacked along the *a*-axis direction with π - π interactions, with minimum short contacts observed for C1...C5ⁱ [3.518 (3) Å], C2...C4ⁱ [3.513 (4) Å] and C7...C6ⁱ [3.513 (4) Å] [symmetry code (i): 1 + x, y, z].

Experimental

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 NO ₅	$D_x = 1.434 \text{ Mg m}^{-3}$
$M_r = 211.17$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1272 reflections
$a = 4.7424 (7) \text{ \AA}$	$\theta = 2.2\text{--}22.4^\circ$
$b = 11.3044 (17) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 18.374 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 96.618 (3)^\circ$	Prism, colourless
$V = 978.5 (3) \text{ \AA}^3$	$0.50 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2080 independent reflections
φ and ω scans	1328 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.945$, $T_{\text{max}} = 0.979$	$\theta_{\text{max}} = 27.0^\circ$
5334 measured reflections	$h = -5 \rightarrow 6$
	$k = -11 \rightarrow 14$
	$l = -22 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1024P)^2 + 0.1147P]$
$R[F^2 > 2\sigma(F^2)] = 0.062$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.219$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
2080 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
141 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected geometric parameters (Å, °).

C1—C7	1.476 (4)	C7—O3	1.319 (3)
C2—O5	1.338 (3)	C8—C9	1.470 (4)
C5—N1	1.454 (3)	N1—O1	1.215 (3)
C7—O4	1.206 (3)	N1—O2	1.216 (3)
O5—C2—C3	116.8 (3)	O1—N1—O2	122.6 (2)
O5—C2—C1	123.9 (3)	O1—N1—C5	119.0 (3)
O4—C7—C1	123.6 (3)	C7—O3—C8	114.3 (2)
O3—C7—C1	113.3 (2)	C6—C5—N1—O2	2.6 (4)
C6—C1—C7—O4	177.2 (3)	C9—C8—O3—C7	177.6 (4)
C2—C1—C7—O4	-4.1 (4)		
C4—C5—N1—O1	3.1 (4)		

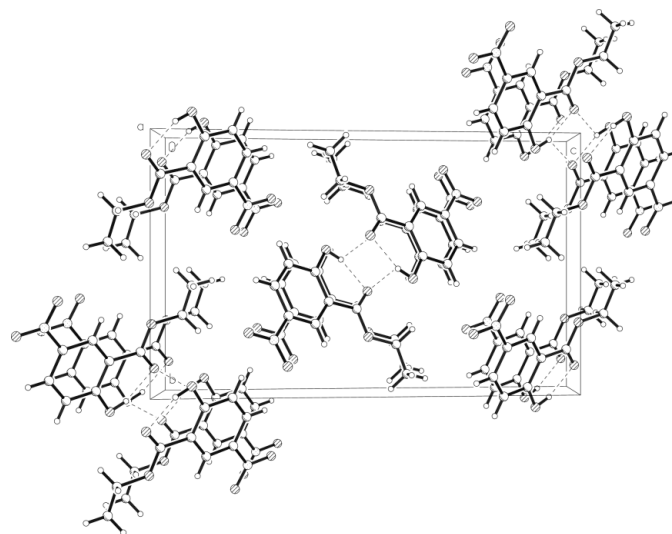


Figure 2

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5...O4 ⁱⁱ	0.86 (5)	2.51 (5)	2.981 (3)	115 (4)
O5—H5...O4	0.86 (5)	1.78 (5)	2.623 (3)	164 (4)

Symmetry code: (ii) 1 - x, -y, -z.

The hydroxyl H atom was located in a difference Fourier map and refined isotropically [O—H = 0.86 (5) 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 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. Owing to large displacement parameters for atoms C8 and C9, the C8—C9 bond distance was restrained to 1.524 (5) Å. No suitable disorder model was found for the ethyl group.

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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