

Methyl 5-nitrosalicylate

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Key indicators

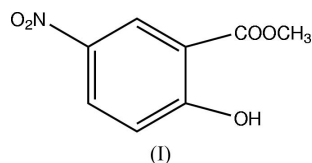
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.054
 wR factor = 0.152
Data-to-parameter ratio = 14.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The asymmetric unit of the title compound, $\text{C}_8\text{H}_7\text{NO}_5$, contains two crystallographically independent molecules. In both molecules, an intramolecular hydrogen bond exists between the hydroxyl group and the O atom of the carbonyl group. The molecules are stacked, with weak π - π interactions, along the a axis.

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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. 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 asymmetric unit of (I) consists of two crystallographically independent molecules (Fig. 1). The corresponding bond lengths and angles in the molecules agree with each other and show normal values (Table 1). In both molecules, an intramolecular hydrogen bond exists between the hydroxyl group and the O atom of the carbonyl group (Table 2). The two independent molecules are linked by a

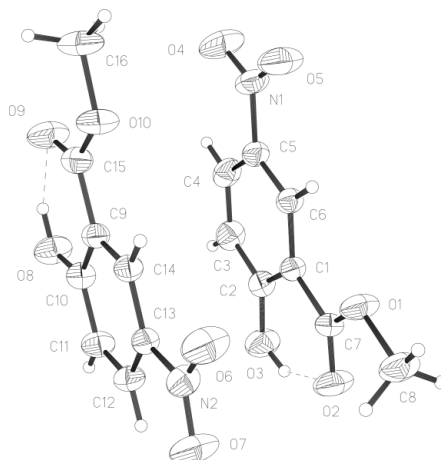


Figure 1
The asymmetric unit of (I), showing displacement ellipsoids at the 30% probability level. Hydrogen bonds are indicated by dashed lines.

weak π - π interaction involving the two benzene rings, arranged with a centroid-centroid separation of 3.6271 (14) Å. These pairs are further stacked along the *a* axis (Fig. 2).

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

$C_8H_7NO_5$	$D_x = 1.538 \text{ Mg m}^{-3}$
$M_r = 197.15$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1515 reflections
$a = 6.9648 (12) \text{ \AA}$	$\theta = 2.7\text{--}24.1^\circ$
$b = 13.162 (2) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$c = 18.579 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 91.015 (4)^\circ$	Prism, colourless
$V = 1702.9 (5) \text{ \AA}^3$	$0.30 \times 0.20 \times 0.18 \text{ mm}$
$Z = 8$	

Data collection

Bruker SMART CCD area-detector diffractometer	3689 independent reflections
φ and ω scans	2100 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.030$
$T_{\text{min}} = 0.964, T_{\text{max}} = 0.973$	$\theta_{\text{max}} = 27.0^\circ$
9850 measured reflections	$h = -8 \rightarrow 8$
	$k = -15 \rightarrow 16$
	$l = -12 \rightarrow 23$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0703P)^2]$
$wR(F^2) = 0.152$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3689 reflections	$\Delta\rho_{\text{max}} = 0.19 \text{ e \AA}^{-3}$
263 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C7	1.312 (3)	O10—C16	1.460 (3)
O1—C8	1.462 (3)	O8—C10	1.338 (3)
O2—C7	1.211 (2)	C5—N1	1.459 (3)
C13—N2	1.461 (3)	O4—N1	1.217 (2)
O9—C15	1.214 (3)	O3—C2	1.334 (3)
O5—N1	1.216 (2)	O7—N2	1.215 (2)
O10—C15	1.321 (3)	N2—O6	1.215 (2)
O2—C7—C1	122.8 (2)	O7—N2—O6	122.2 (2)
O1—C7—C1	114.9 (2)	O7—N2—C13	118.7 (2)
O9—C15—C9	123.5 (2)	O5—N1—O4	122.5 (2)
O10—C15—C9	113.6 (2)	O4—N1—C5	118.4 (2)
C8—O1—C7—C1	178.7 (2)	C14—C9—C15—O10	4.2 (3)
C2—C1—C7—O2	2.1 (3)	C12—C13—N2—O7	-4.6 (3)
C6—C1—C7—O1	1.8 (3)	C14—C13—N2—O6	-4.7 (3)
C16—O10—C15—C9	179.5 (2)	C6—C5—N1—O5	0.6 (3)
C10—C9—C15—O9	2.6 (3)	C4—C5—N1—O4	1.8 (3)

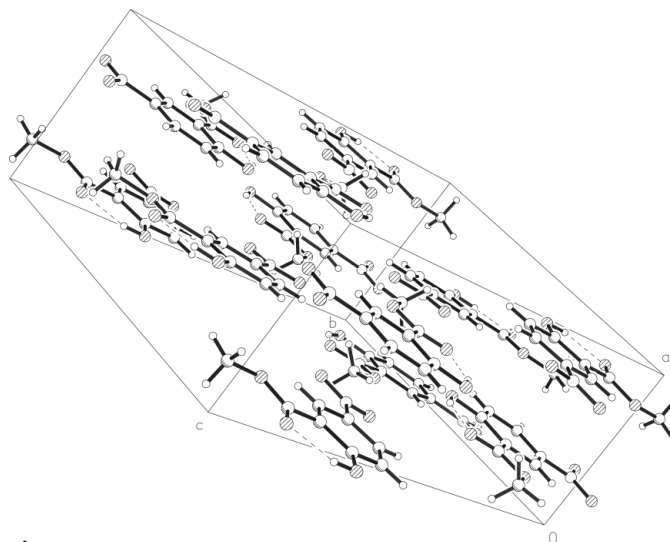


Figure 2

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3 \cdots O2	0.89 (3)	1.81 (3)	2.597 (3)	146 (3)
O8—H8 \cdots O9	0.88 (3)	1.82 (3)	2.603 (2)	147 (3)

The hydroxyl H atoms were located in a difference Fourier map and refined isotropically [$O-H = 0.88 (3)$ and $0.89 (3) \text{ \AA}$]. All other H atoms were included in the riding-model approximation, with C—H distances of 0.93 (aromatic H atoms) and 0.96 Å (methyl H atoms). The isotropic displacement parameters were set equal to $1.2U_{\text{eq}}$ of the carrier atom for the aromatic 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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