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Ethyl 3,5-dinitrosalicylate

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The title compound, $C_9H_8N_2O_7$, is found to have an intramolecular hydrogen bond between the hydroxy group and the O atom of the carbonyl group. The molecule is essentially planar.

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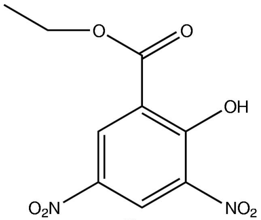
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.126
Data-to-parameter ratio = 8.7

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

Comment

The title compound, (I), is similar to methyl 3,5-dinitro salicylate (Jin *et al.*, 2004), and was first prepared by McMillan & King (1945) by the standard procedure for alkyl salicylates (Cahours, 1843). It was later found that (I) could also be prepared by the nitration of sulfides of organic compounds, from the starting materials $[4,3\text{-HO(MeO}_2\text{C)C}_6\text{H}_3]_3\text{S}$ and HNO_3 (Airan & Kulkarni, 1951). As part of our ongoing studies of the derivatives of 3,5-dinitrosalicylic acid, we prepared compound (I) and studied its crystal structure.



(I)

Compound (I) shows an intramolecular hydrogen bond between the hydroxy group and the O atom of the carbonyl group (Fig. 1), and the whole molecule is planar except for the methyl H atoms. Details of the hydrogen-bonding interaction are listed in Table 2.

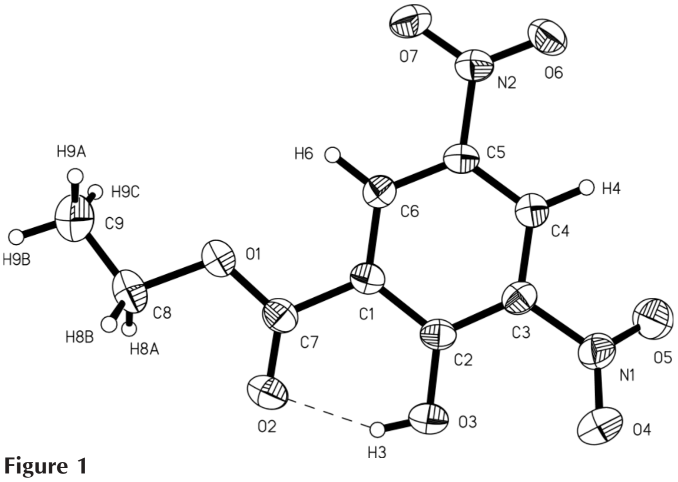


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 30% probability level. The O—H···O hydrogen bond is indicated by a dashed line.

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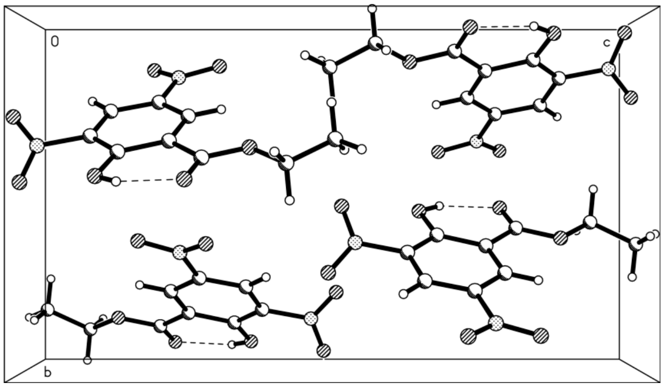

Figure 2
Packing diagram for (I). The hydrogen bonds are indicated by dashed lines.

Experimental

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

Crystal data

$C_9H_8N_2O_7$
$M_r = 256.17$
Orthorhombic, $P2_12_12_1$
$a = 6.2128$ (7) Å
$b = 10.0405$ (11) Å
$c = 17.4701$ (19) Å
$V = 1089.8$ (2) Å^3
$Z = 4$
$D_x = 1.561$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1569 reflections
$\theta = 2.3$–20.8°
$\mu = 0.14$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.24 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7236 measured reflections
1461 independent reflections
1121 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.000$
$\theta_{\rm max} = 27.5°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.126$
$S = 1.09$
1461 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.0949P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C7	1.224 (4)	C1—C7	1.480 (4)
O3—C2	1.329 (4)	N2—C5	1.462 (4)
O1—C7	1.316 (4)	N1—C3	1.471 (4)
O1—C8	1.473 (4)	C9—C8	1.466 (6)
C7—O1—C8	116.6 (3)	O1—C7—C1	113.7 (3)
O2—C7—O1	123.4 (3)	C3—C2—C1	117.1 (3)
C8—O1—C7—C1	179.4 (3)	O5—N1—C3—C4	−28.6 (4)
O7—N2—C5—C4	173.3 (3)	O4—N1—C3—C2	−28.9 (5)
O6—N2—C5—C6	173.8 (3)	C7—O1—C8—C9	−168.9 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.83 (2)	1.75 (3)	2.550 (4)	162 (7)

C-bound H atoms were included in the riding-model approximation, with C—H distances of 0.93 (aromatic H atoms), 0.96 (CH_3 H atoms) and 0.97 Å (CH_2 H atoms). The hydroxy H atom was initially located in a difference Fourier map and was then constrained to an ideal geometry (O—H = 0.82 Å) and refined isotropically. The $U_{\rm iso}$(H) values were set equal to $1.2U_{\rm eq}$ of the carrier atom for the aromatic and CH_2 H atoms, and $1.5U_{\rm eq}$ of the carrier for CH_3 H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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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