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Methyl 3,5-dinitrosalicylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.133
Data-to-parameter ratio = 12.0

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

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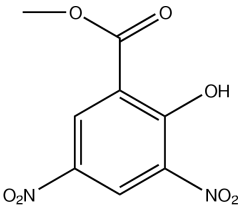
The title compound, $C_8H_6N_2O_7$, crystallizes with an intramolecular hydrogen bond between the hydroxyl group and the O atom of the carbonyl group. A weak intermolecular hydrogen bond between the hydroxyl group and a nitro O atom forms planes parallel to the *b* axis.

Comment

The title compound, (I), was first prepared in 1945 (Freeman & John, 1945) by the standard procedure of alkyl salicylate (Cahours, 1843). It was later found that (I) could also be prepared by the nitration of sulfides of organic compounds, the starting materials being $[4,3\text{-HO}(\text{MeO}_2\text{C})\text{C}_6\text{H}_3]_3\text{S}$ and HNO_3 (Airan & Kulkarni, 1951). In the framework of a systematic search for new synthetic routes to derivatives of 3,5-dinitrosalicylic acid, such as 3,5-dinitrosalicylhydroximate, we characterized the crystal structure of (I).



(I)

Compound (I) exists with an intramolecular hydrogen bond between the hydroxyl group and the O atom of the carbonyl group (Fig. 1) and packs in a hydrogen-bonded plane, parallel to the *b* axis, through a weak intermolecular hydrogen bond between the hydroxyl group and a nitro O atom (Fig. 2 and Table 2).

Experimental

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

Crystal data

$C_8H_6N_2O_7$	D_x = 1.692 Mg m^{-3}
M_r = 242.16	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1976 reflections
a = 8.459 (2) Å	θ = 2.4–28.2°
b = 16.942 (5) Å	μ = 0.15 mm^{-1}
c = 6.6832 (18) Å	T = 293 (2) K
β = 96.929 (4)°	Block, colorless
V = 950.8 (4) Å^3	0.38 × 0.22 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
4905 measured reflections
1869 independent reflections
1520 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 26.0°$
$h = -10 \rightarrow 7$
$k = -20 \rightarrow 19$
$l = -7 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.133$
$S = 1.06$
1869 reflections
156 parameters
Hatom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.018$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.486 (3)	C7—O1	1.312 (3)
C2—O3	1.329 (2)	C8—O1	1.455 (3)
C5—N2	1.463 (3)	N2—O6	1.217 (2)
C7—O2	1.211 (3)	N2—O7	1.220 (2)
O3—C2—C1	122.08 (18)	O1—C7—C1	111.72 (18)
O2—C7—O1	124.7 (2)	C7—O1—C8	118.25 (18)
C2—C3—N1—O4	−34.9 (3)	C6—C5—N2—O7	13.6 (3)
C2—C3—N1—O5	145.5 (2)	O2—C7—O1—C8	−3.0 (4)
C6—C5—N2—O6	−167.0 (2)	C1—C7—O1—C8	176.2 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3$\cdots$O6$^{\rm i}$	0.82	2.40	2.910 (2)	121
O3—H3$\cdots$O2	0.82	1.88	2.602 (2)	147

Symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All 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), and an O—H distance of 0.82 Å. The hydroxyl H atoms were initially located in a difference Fourier map, and were then constrained to an ideal geometry. The isotropic displacement parameters were set equal to $1.2U_{\rm eq}$ of the carrier atom for the aromatic H atoms and to $1.5U_{\rm eq}$ of the carrier for methyl and hydroxyl H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT–Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Bruker, 2001); software used to prepare material for publication: *SHELXTL-NT*.

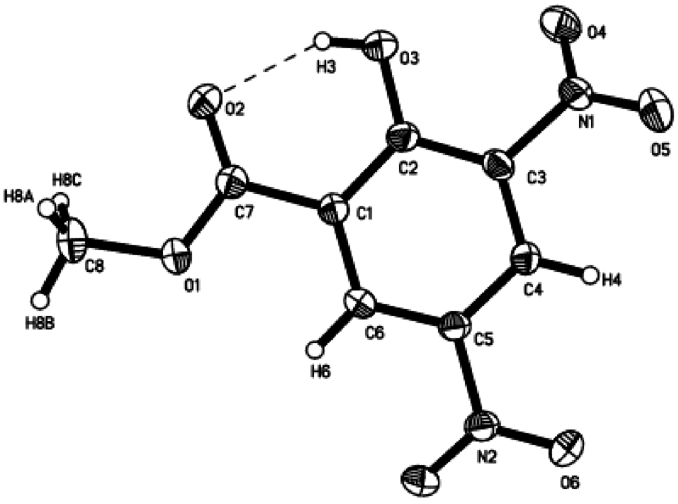


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 30% probability level. H atoms are drawn as small spheres of arbitrary radii.

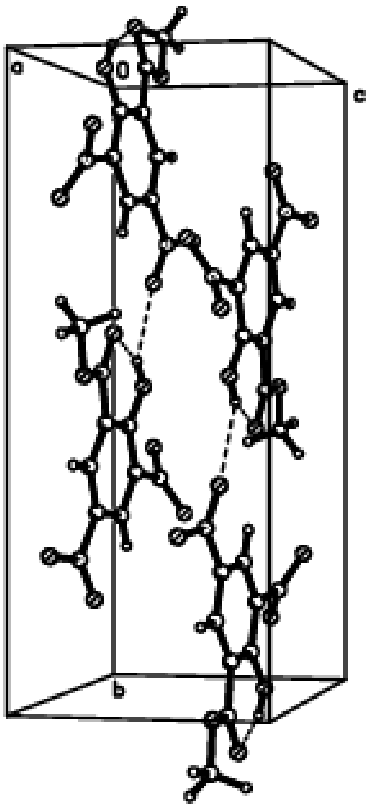


Figure 2
Packing diagram for (I). Hydrogen bonds are indicated by dashed lines.

References

Airan, J. W. & Kulkarni, D. M. (1951). *J. Indian Chem. Soc.* **28**, 595–596.

Bruker (2001). *SMART* (Version 5.0), *SAINT-Plus* (Version 5.0) and *SHELXTL-NT* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.

Cahours, A. (1843). *Annales*, **48**, 63–67.

Freeman, H. M. & John, A. K. (1945). *J. Am. Chem. Soc.* **67**, 2271–2272.

Paul, D. B. & Edward, N. T. (1958). *J. Am. Chem. Soc.* **80**, 5808–5812.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

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