

(E)-3-Nitrobenzaldehyde O-acetyloxime

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

R factor = 0.061

wR factor = 0.158

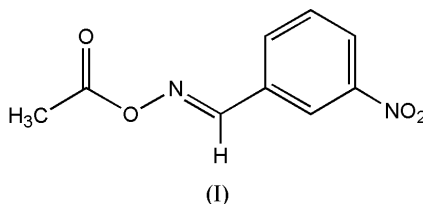
Data-to-parameter ratio = 12.4

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

In the title compound, $\text{C}_9\text{H}_8\text{N}_2\text{O}_4$, the 3-nitrobenzaldehyde oxime and acetyl group have a dihedral angle of $19.5(4)^\circ$. The acetyl carbonyl and 3-nitrobenzaldehyde oxime groups both adopt a *trans* configuration (*E*). In the crystal structure, molecules are linked by a series of weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions, forming a sheet-like structure parallel to the (303) plane.

Comment

The molecular structure of the title compound, (I), and the atom-labelling scheme are illustrated in Fig. 1. Selected bond lengths and angles are given in Table 1. The $\text{O9}-\text{N8}$ bond length of $1.429(3)$ Å is not significantly different from the $\text{N}-\text{O}$ acetyl bond length found in (*E*)-*O*-palmitoyl phenyl 2-pyridyl ketone oxime (1.439 Å; Taga & Miyasaka, 1987). The title compound exhibits a somewhat shortened $\text{N8}-\text{C7}$ bond [$1.256(3)$ Å], characteristic of an oxime group (1.273 Å; Taga & Miyasaka, 1987). The acetyloxime group is almost planar [the maximum deviation from the calculated mean plane is $0.071(2)$ Å for atom N8]. The dihedral angle between the 3-nitrobenzaldehyde oxime and acetyl groups is $19.5(4)^\circ$. The acetyl carbonyl and 3-nitrobenzaldehyde oxime groups both adopt a *trans* configuration (*E*) [torsion angle $\text{O9}-\text{N8}-\text{C7}-\text{C1} = -176.6(2)^\circ$] (see Fig. 1).



In the crystal structure, molecules of (I) are connected by a series of $\text{C}-\text{H}\cdots\text{O}$ contacts that can be considered as weak hydrogen bonds (Table 2), since all contacts have $\text{H}\cdots\text{O}$ values less than 2.6 Å. The molecules lie approximately in the (303) plane, thus forming a two-dimensional polymeric structure, as shown in Fig. 2.

Experimental

A solution of 3-nitrobenzaldehyde oxime (246 mg, 1.46 mmol) in acetic anhydride (2 ml) was refluxed for 2 h. Excess acetic anhydride was removed under vacuum, affording 261 mg (85% yield) of (I). Colourless crystals were obtained by slow evaporation of an acetic anhydride solution. IR (KBr, cm^{-1}): 1620 ($\text{C}=\text{N}$), 1699 (CO); ^1H NMR (300.08 MHz, CDCl_3 , p.p.m.): 8.5 (s, 1H), 8.4 (s, 1H), 8.2 (s, 1H), 8.1 (d, 1H), 7.2 (t, 1H), 2.2 (s, 3H); ^{13}C NMR (75.46 MHz, CDCl_3 , p.p.m.): 168.4, 153.9, 148.7, 137.9, 132.2, 130.3, 127.8, 123.3, 19.6.

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Crystal data

C₉H₈N₂O₄
M_r = 208.17
 Monoclinic, *P*2₁/*c*
a = 8.3129 (11) Å
b = 11.2119 (15) Å
c = 10.2947 (14) Å
 β = 92.769 (2)°
V = 958.4 (2) Å³
Z = 4

D_x = 1.443 Mg m⁻³
 Mo Kα radiation
 Cell parameters from 600 reflections
 θ = 20.0–25.0°
 μ = 0.12 mm⁻¹
T = 293 (2) K
 Block, colorless
 0.42 × 0.40 × 0.36 mm

Data collection

Bruker SMART area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 9042 measured reflections
 1688 independent reflections

1386 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.037
 θ_{max} = 25.0°
h = -9 → 9
k = -13 → 13
l = -12 → 12

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.061
wR (*F*²) = 0.158
S = 1.12
 1688 reflections
 136 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0771*P*)² + 0.3126*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.26 e Å⁻³
 Δρ_{min} = -0.23 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O9–N8	1.429 (3)	O13–N12	1.215 (3)
O9–C10	1.363 (3)	N8–C7	1.256 (3)
O10–C10	1.180 (3)	N12–C5	1.471 (3)
O12–N12	1.212 (3)		
N8–O9–C10	112.18 (17)	N12–C5–C6	118.2 (2)
O9–N8–C7	109.66 (19)	N8–C7–C1	119.7 (2)
O12–N12–O13	123.1 (2)	O9–C10–O10	123.8 (3)
O12–N12–C5	118.2 (2)	O9–C10–C11	109.8 (2)
O13–N12–C5	118.7 (2)	O10–C10–C11	126.4 (3)
N12–C5–C4	118.8 (2)		
C10–O9–N8–C7	-175.8 (2)	O9–N8–C7–C1	-176.6 (2)
N8–O9–C10–C11	-174.3 (2)	O13–N12–C5–C6	-5.5 (4)
N8–O9–C10–O10	6.3 (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>
C4–H4...O13 ⁱ	0.93	2.56	3.387 (3)	148
C6–H6...O10 ⁱⁱ	0.93	2.41	3.264 (3)	152
C7–H7...O10 ⁱⁱ	0.93	2.58	3.385 (3)	146

Symmetry codes: (i) 1 - *x*, ½ + *y*, -½ - *z*; (ii) -*x*, *y* - ½, ½ - *z*.

H atoms were included in calculated positions and treated as riding atoms, with C–H distances in the range 0.93–0.96 Å, and with *U*_{iso}(H) = 1.5*U*_{eq}(C) for the methyl H atoms and 1.2*U*_{eq}(C) for the other H atoms.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2000); software used to prepare material for publication: SHELXL97 and WinGX2003 (Farrugia, 1999).

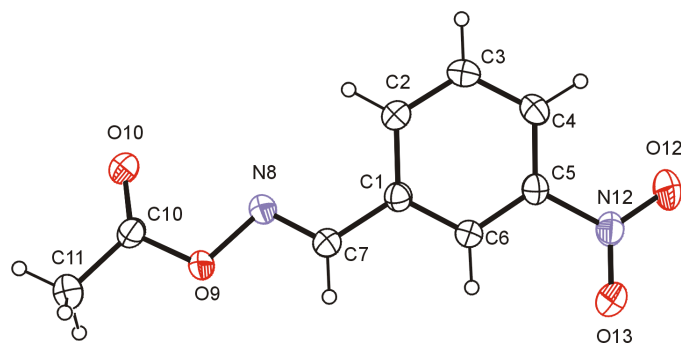


Figure 1
 The molecular structure of the title compound, (I), with displacement ellipsoids drawn at the 30% probability level.

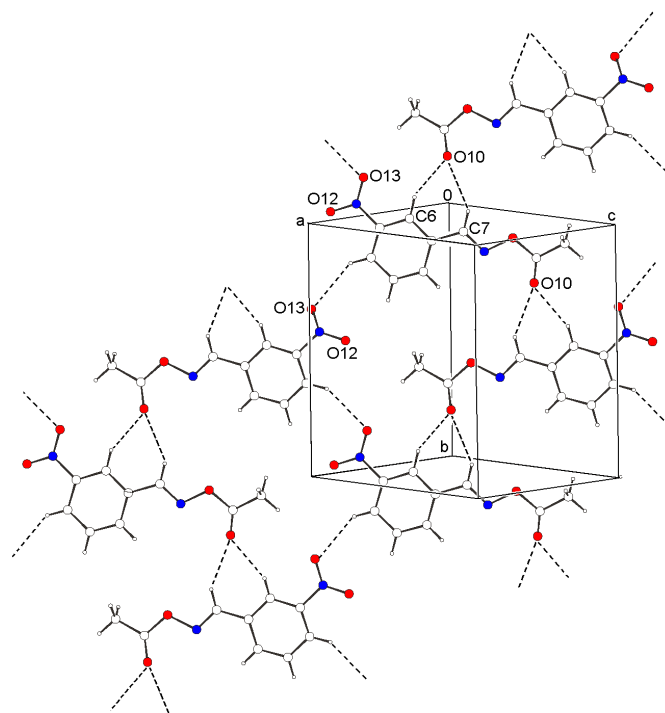


Figure 2

The crystal packing of (I), showing the formation of the two-dimensional C–H...O hydrogen-bonded polymer in the (303) plane. Hydrogen bonds are shown as dashed lines.

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