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Acetaldehyde 2,4-dinitrophenylhydrazone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.109
Data-to-parameter ratio = 14.1

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

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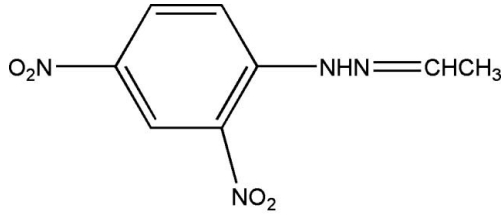
The molecule of the title Schiff base compound, $C_8H_8N_4O_4$, is not planar, with a dihedral angle of 9.3 (2)° between the N—N=C—C plane and the benzene ring. There is an intramolecular N—H···O hydrogen bond, and N—O···π interactions, which help to stabilize the structure.

Comment

2,4-Dinitrophenylhydrazine is a reagent which is widely used for condensation with aldehydes and ketones. Several phenylhydrazone derivatives have been shown to be potentially DNA-damaging and are mutagenic agents (Okabe *et al.*, 1993). Some phenylhydrazone derivatives have been synthesized in our laboratory (Qian, Liu, Yin & Ng, 2006, Qian, Liu & Yin, 2006). As part of our work, we have synthesized the title compound, (I), and report the crystal structure here.



(I)

In (I), there is a dihedral angle of 9.3 (2)° between the benzene ring and the N3/N4/C7/C8 plane (Fig.1). The dihedral angles between the benzene ring and the O1/O2/N2/C4/C5/C6 and O1/O2/N2/C4/C3/C2 planes are 4.70 (11) and 4.43 (11)°, respectively. The C1—C2 and C1—C6 bond lengths are slightly longer than the other C—C bond lengths in the benzene ring (Table 1).

There is an intramolecular N—H···O hydrogen bond (Fig. 1 and Table 2), while the molecules are linked by N—O···π interactions between N1—O4 and the benzene ring (Steiner *et al.*, 1995) [O4···*Cg*1^{i} = 3.35 Å and N1—O4···*Cg*1^{i} = 94.04°, where *Cg*1 is the centroid of the benzene ring; symmetry code: (i) −1 + *x*, *y*, *z*].

Experimental

2,4-Dinitrophenylhydrazine (1 mmol, 0.198 g) was dissolved in anhydrous ethanol (15 ml). H_2SO_4 (98% 0.5 ml) was added and the mixture stirred for several minutes at 351 K. Acetaldehyde (1 mmol, 0.044 g) in ethanol (5 ml) was added dropwise and the mixture was stirred at refluxing temperature for 3 h. The product was separated and recrystallized from dichloromethane. Yellow single crystals of (I) were obtained after 2 d.

Crystal data

$C_8H_8N_4O_4$
M_r = 224.18
Monoclinic, $P2_1/c$
a = 5.353 (1) Å
b = 10.872 (2) Å
c = 17.725 (3) Å
β = 96.160 (4)°
V = 1025.6 (3) Å^3
Z = 4
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
Block, yellow
0.23 × 0.21 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.973, T_{max} = 0.979
8012 measured reflections
2120 independent reflections
931 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 0.82
2120 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0029 (11)

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.411 (3)	C3—C4	1.370 (3)
C1—C6	1.428 (3)	C4—C5	1.379 (3)
C2—C3	1.385 (3)	C5—C6	1.363 (3)
O4—N1—C2—C3	177.78 (18)	O1—N2—C4—C3	−174.4 (2)
O3—N1—C2—C1	177.16 (18)	O2—N2—C4—C5	−175.4 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O4	0.899 (9)	1.927 (16)	2.622 (2)	132.7 (18)

H3*A* attached to N3 was located in a difference map and refined freely; N—H = 0.899 (9) Å. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and refined with U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions, with C—H = 0.93 Å, N—H = 0.89 Å and U_{iso}(H) = 1.2U_{eq}(C).

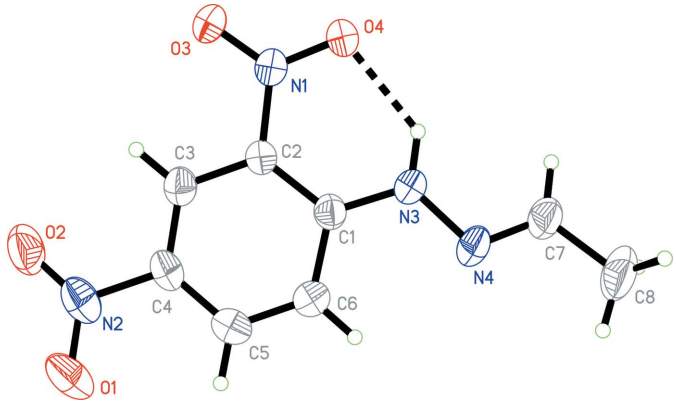


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXTL*.

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