Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Formaldehyde 2,4-dinitrophenylhydrazone

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Received 22 November 2006
Accepted 25 November 2006

In the title compound, $C_7H_6N_4O_4$, the molecule is essentially planar and the nitro groups are nearly coplanar with the benzene ring. The crystal structure is stabilized by intermolecular C—H···O, N—H···O and C—H···N interactions, as well as by short O···O and O···N contacts.

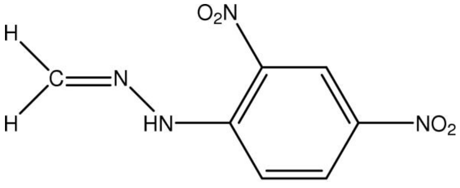
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 17.5

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

Comment

2,4-Dinitrophenylhydrazone derivatives are widely used in various forms of analytical chemistry (Lamberton *et al.*, 1974; Pötter *et al.*, 1997; Zegota, 1999; Cordis *et al.*, 1998; Zlotorzynska & Lai, 1999) and are also used as dyes (Guillaumont & Nakamura, 2000). Structurally, they are found to exist as both *E*- and *Z*-stereoisomers (Uchiyama *et al.*, 2003). The title compound, (I), was first reported by Clark *et al.* (1946) but its single-crystal X-ray structure has not been established. Continuing our investigations into the complexation properties of dinitrophenylhydrazine, DNPH, with carbonyl compounds (Tameem *et al.*, 2006), we report here the structure of compound (I), the product of the reaction of DNPH with formaldehyde.


(I)

Bond lengths and angles in (I) (Fig. 1) have normal values (Allen *et al.*, 1987) and agree well with those found in related

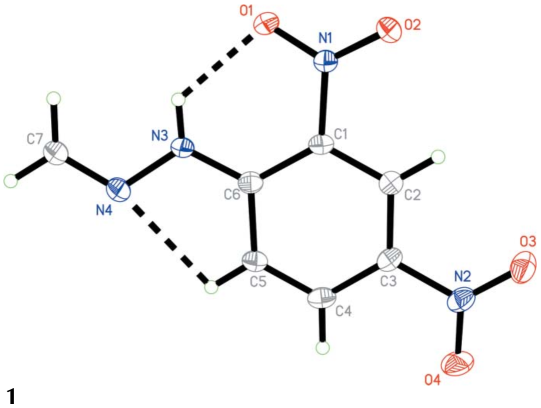


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Dashed lines indicate intramolecular hydrogen bonds.

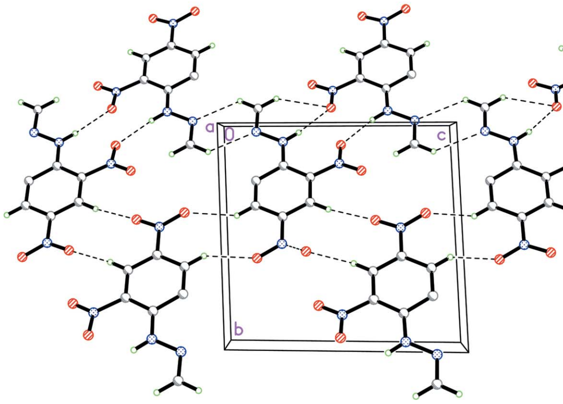


Figure 2
The crystal packing of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

structures (Fronczek, 1994). The molecule is approximately planar, with a maximum deviation of 0.270 (1) Å from the molecular plane for atom O1. The nitro groups at C1 and C3 are almost coplanar with the attached ring [O1—N1—C1—C6 = −12.3 (2)° and O3—N2—C3—C2 = 7.2 (2)°], as is the hydrazine group, with out-of-plane distances for atoms N3, N4 and C7 of −0.031 (1), 0.040 (1) and 0.157 (1) Å, respectively.

In the crystal structure of (I), intermolecular N—H⋯O and C—H⋯O hydrogen bonds (Fig. 2, Table 1) form sheets parallel to the (120) plane. In addition, relatively short O1⋯O1^{i} [2.7258 (12) Å], O1⋯O1^{v} [2.8819 (12) Å], O2⋯O3^{vi} [2.8779 (12) Å] and O1⋯N1^{i} [2.9467 (12) Å] contacts further stabilize the crystal packing [symmetry codes: (i) $-x, -y, 1-z$; (v) $1-x, -y, 1-z$; (vi) $1-x, 1-y, 1-z$].

Experimental

Compound (I) was prepared as described by Clark *et al.* (1946). Crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a saturated solution of compound (I) in an ethanol–acetonitrile solvent mixture (1:1).

Crystal data

$C_7H_6N_4O_4$
$M_r = 210.16$
Triclinic, $P\overline{1}$
$a = 4.2482$ (1) Å
$b = 9.7774$ (2) Å
$c = 10.3407$ (2) Å
$\alpha = 86.825$ (1)°
$\beta = 83.966$ (1)°
$\gamma = 82.413$ (1)°
$V = 423.02$ (2) Å^3
$Z = 2$
$D_x = 1.650$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 100.0$ (1) K
Block, yellow
0.35 × 0.18 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.953$, $T_{\max} = 0.987$
14315 measured reflections
2447 independent reflections
2027 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.06$
2447 reflections
140 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.0932P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3A⋯O1	0.849 (17)	2.03 (2)	2.623 (1)	126 (1)
C5—H5A⋯N4	0.93	2.41	2.743 (2)	101
N3—H3A⋯O1^{i}	0.849 (17)	2.49 (2)	3.271 (1)	153 (2)
C2—H2A⋯O3^{ii}	0.93	2.59	3.498 (1)	164
C4—H4A⋯O4^{iii}	0.93	2.54	3.346 (2)	145
C7—H7A⋯O1^{i}	0.97	2.60	3.425 (1)	143
C7—H7B⋯N4^{iv}	0.96	2.59 (2)	3.373 (2)	138

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+2, -y+1, -z$; (iv) $-x, -y, -z$.

The H atom on N3 was located in a difference map and refined isotropically, with N—H = 0.849 (17) Å. The remaining H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government for providing a SAGA grant (No. 304/PKIMIA/653010/A118), and Universiti Sains Malaysia for a Short-Term Grant, which have made this work possible.

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