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2,6-Dichlorobenzaldehyde 2,4-dinitrophenylhydrazone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.092
Data-to-parameter ratio = 14.3

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

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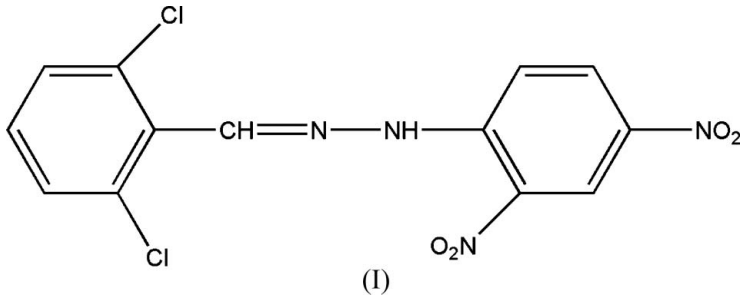
In the title compound, $C_{13}H_8Cl_2N_4O_4$, the dinitrophenylhydrazone and benzene ring planes are nearly coplanar, making a dihedral angle of 3.39 (9)°. There is an intramolecular N—H···O interaction and weak intermolecular C—H···O hydrogen-bonding interactions which stabilize the packing.

Comment

Several phenylhydrazone derivatives have been shown to be potentially DNA-damaging and are mutagenic agents (Okabe *et al.*, 1993). One of our aims is to obtain Schiff bases with a view to studying their structural conformations. We have synthesized the new Schiff base, (I), and report here the crystal structure.



(I)

The title molecule is built up from a dinitrophenylhydrazone system and a benzene ring which are nearly coplanar, making a dihedral angle of 3.39 (9)° (Fig. 1). The C1—C2 [1.402 (2) Å] and C1—C6 [1.406 (2) Å] bond lengths are significantly longer than the other C—C bond lengths in the same ring [mean value 1.378 (3) Å], which is consistent with other dinitrophenyldrazone derivatives (Ohba, 1996; Bolte *et al.*, 1998).

The two nitro groups of the dinitrophenylhydrazone system are slightly twisted with respect to the attached benzene ring, making dihedral angles of 5.42 (14) and 5.9 (3)° for O1/N3/O2 and O3/N4/O4, respectively. There is an intramolecular N—H···O hydrogen bond. Weak intermolecular C—H···O hydrogen-bonding interactions stabilize the crystal packing (Table 1 and Fig. 2).

Experimental

2,4-Dinitrophenylhydrazine (1 mmol, 0.198 g) was dissolved in anhydrous ethanol (15 ml), then H_2SO_4 (98%, 0.5 ml) was added and the mixture was stirred for several minitute at 351 K. 2,6-Dichlorobenzaldehyde (1 mmol, 0.175 g) in ethanol (5 ml) was added dropwise and the mixture was stirred at refluxing temperature for 1 h. The

product was separated and recrystallized from acetone to give brown single crystals of (I) within 4 d.

Crystal data

$C_{13}H_8Cl_2N_4O_4$
M_r = 355.13
Monoclinic, $P2_1/c$
a = 12.375 (2) Å
b = 7.928 (1) Å
c = 14.977 (2) Å
β = 101.588 (3)°
V = 1439.4 (4) Å^3
Z = 4
D_x = 1.639 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 298 (2) K
Block, yellow
0.31 × 0.28 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.866, T_{max} = 0.890
10917 measured reflections
2984 independent reflections
2290 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.07
2984 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	2.01	2.6192 (16)	127
C4—H4···O3^{i}	0.93	2.59	3.282 (2)	132
C5—H5···O4^{ii}	0.93	2.59	3.509 (2)	170
C7—H7···O2^{iii}	0.93	2.58	3.3723 (19)	144

Symmetry codes: (i) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x+1, y-1, z$; (iii) $-x, y-\frac{1}{2}, -z-\frac{1}{2}$.

H atoms were treated as riding on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,*N*).

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: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

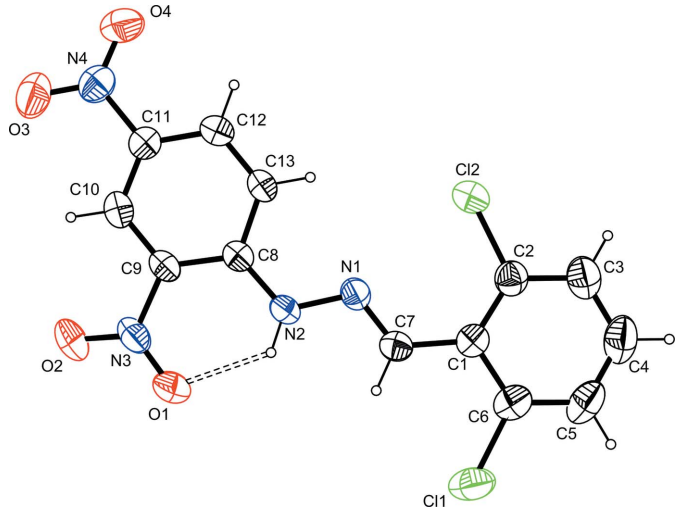


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The dashed lines indicate a hydrogen bond.

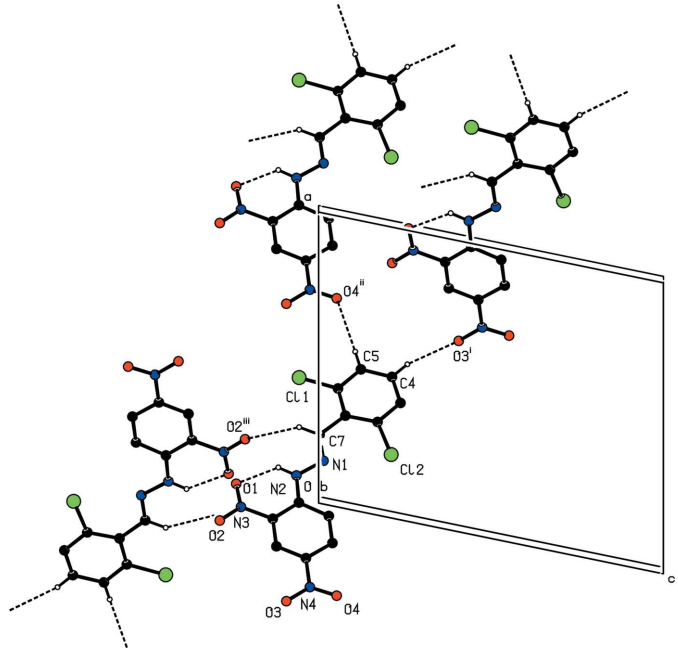


Figure 2
Partial view of the packing, showing the intermolecular hydrogen-bonding interactions as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $1+x, y-1, z$; (iii) $-x, y-\frac{1}{2}, -z-\frac{1}{2}$.]

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