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(2,4-Dinitrophenyl)(1-nitrohexyl)diazene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.130
Data-to-parameter ratio = 11.5

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

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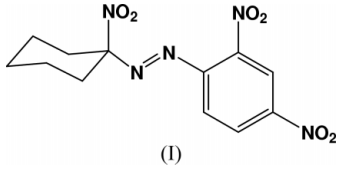
The title compound, $C_{12}H_{13}N_5O_6$, is a new azo compound. Its crystal structure shows that weak intermolecular interactions play an important role in the crystal packing. A dimer is formed *via* a weak C—H···O interaction between two molecules. Neighboring dimers are connected by weak C—H···O interactions and a three-dimensional framework is formed.

Comment

Nitric oxide (NO) is an important biological messenger with vital immunological, cardiovascular and neurological functions (Bredt & Snyder, 1994; Moncada & Higgs, 1993). Therefore, intensive research has been directed towards the reactions of NO with various organic compounds. Arylhydrazones have been utilized for the analysis of carbonyl compounds. In recent years, some of these compounds and their nitration products were found to have pharmacological properties (Morgan *et al.*, 2001, 2002). It has been reported that some compouds containing an imine bond, such as oximes, Schiff bases and hydrazones, could react with nitrosonium and nitronium cations, thereby cleaving the imine bond to produce primary carbonyl compounds (Wildsmith, 1972; Olah & Ho, 1976; Pozsgay & Jennings, 1987). A search of the Cambridge Structural Database (*CONQUEST*, Version 1.5; Allen, 2002) for the 1-azo-2,4-dinitrophenyl group gave only six hits. We report here the synthesis and crystal structure of (2,4-dinitrophenyl)(1-nitrohexyl)diazene, (I), obtained by the reaction of NO with hexanone 2,4-dinitrophenylhydrazone.



(I)

The structure of (I) (Fig. 1) consists of 2,4-dinitrophenyl and nitrohexane moieties linked by an azo group. The conformation of the cyclohexane ring is a chair and all the bond lengths and angles are normal. The O atoms of one nitro group are disordered over two positions [major orientation O3/O4 with s.o.f. = 0.57; minor orientation O3′/O4′ with s.o.f. = 0.43]. C—H···O intermolecular interactions (Table 1) play an important role in the crystal packing (Fig. 2). A dimer is formed *via* a pair of C8—H8*a*···O1^{i} (see Table 1 for symmetry codes) hydrogen bonds (Fig. 1) and C9—H9*b*···O2^{ii} and C4—H4···O6^{iii}

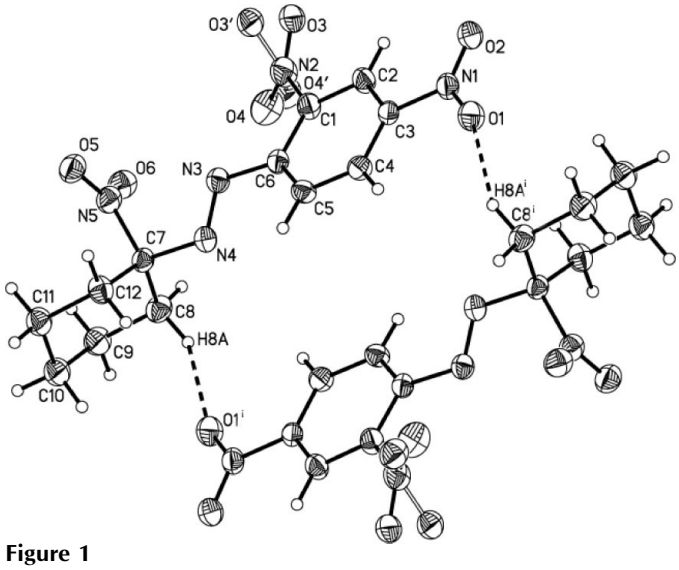


Figure 1
Details of the dimer formation in (I), drawn with 30% probability ellipsoids. The symmetry code is as in Table 1. Hydrogen bonds are shown as dashed lines.

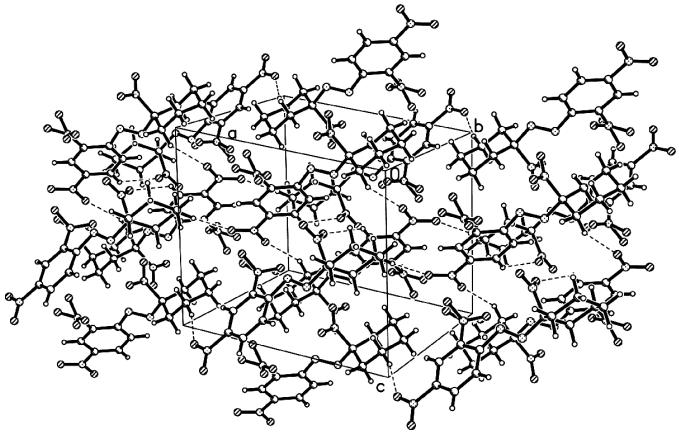


Figure 2
The packing of (I).

hydrogen bonds extend the dimers into a three-dimensional framework (Fig. 2).

Experimental

A stock solution was prepared by dissolving 0.5 mmol hexanone 2,4-dinitrophenylhydrazone in 100 ml dry CH_2Cl_2. NO was produced by the reaction of 1 *M* H_2SO_4 solution to a stirred saturated $NaNO_2$ aqueous solution under an argon atmosphere. NO was carried by argon and purified by passing it through a series of scrubbing bottles containing 4 *M* NaOH, distilled water and $CaCl_2$, in that order. The bottles were under an argon atmosphere. The purified NO was bubbled through a previously degassed stirred stock solution at room temperature for 1 h. After completion of the reaction, as indicated by thin-layer chromatography, the reaction mixture was dried with anhydrous $MgSO_4$, concentrated *in vacuo* and purified by column chromatography on silica gel (200–300 mesh, ethyl acetate–hexane), then recrystallized from hexane–ethyl acetate, giving the pure title compound.

Crystal data

$C_{12}H_{13}N_5O_6$	D_x = 1.453 Mg m^{-3}
M_r = 323.27	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 11.282 (2) Å	θ = 1.8–25.5°
b = 13.033 (2) Å	μ = 0.12 mm^{-1}
c = 10.162 (1) Å	T = 293 (2) K
β = 98.50 (1)°	Block, colorless
V = 1477.8 (4) Å^3	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

Bruker *P*4 diffractometer	θ_{max} = 25.5°
ω scans	$h = -13 \rightarrow 13$
Absorption correction: none	$k = 0 \rightarrow 15$
2748 measured reflections	$l = 0 \rightarrow 12$
2748 independent reflections	3 standard reflections
1811 reflections with $I > 2\sigma(I)$	every 97 reflections
R_{int} = 0.011	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.66P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.130	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.59 e Å^{-3}
2748 reflections	
239 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1^{i}	0.97	2.57	3.433 (4)	148
C9—H9B···O2^{ii}	0.97	2.56	3.506 (4)	165
C4—H4···O6^{iii}	0.93	2.60	3.344 (3)	138

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

All H atoms were found in difference maps and refined as riding, with C—H bond lengths in the range 0.93–0.97 Å and U_{iso} values set at $1.2U_{eq}$ of the carrier atom.

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

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