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(*E*)-1-(2,4-Dinitrophenyl)-2-(2-isopropyl-5-methyl-1-nitrocyclohexyl)diazene

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The title compound, $C_{16}H_{21}N_5O_6$, is a chiral β-nitro azo compound prepared in the reaction of a chiral menthone hydrazone with nitric oxide in dichloromethane. The molecule adopts a *trans* configuration with respect to the N═N double bond. In the crystal structure, weak intermolecular C—H···O hydrogen bonds link the molecules into coupled chains extending along the *b* axis.

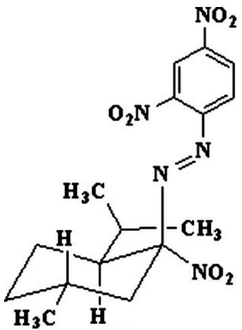
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.038
wR factor = 0.087
Data-to-parameter ratio = 8.3

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

Comment

The number of publications concerning the reactivity of NO with various organic compounds has grown rapidly in the last few years (Chiarantini *et al.*, 2003; Smith *et al.*, 2005; Masami *et al.*, 2005). Arylhydrazones have long been utilized for the analysis of carbonyl compounds. In recent years, some arylhydrazones and their complexes have been found to have anticancer properties. In particular, hydrazones have been used to build chiral configurations and certain specific skeletons (Paschalidis *et al.*, 2000; Job *et al.*, 2002). The highly selective reaction of a chiral menthone hydrazone with nitric oxide affords the title compound, (I), in a good yield. The reaction is assumed to be most likely initiated by an electrophilic addition of NO_2 to the C atom of the C═N double bond. The differences of the reaction mechanism of ketone hydrazones from aldehyde hydrazones are due to no hydrogen being eliminated on the C atom of the imine double bonds of ketone hydrazones.



(I)

In (I) (Fig. 1), there are three chiral atoms, C1, C2 and C5, in the six-membered C1–C6 ring. The bond lengths N4═N5, C1—N5 and N1—C1 are 1.231 (3), 1.472 (4) and 1.540 (4) Å, respectively. The O5/N3/O6 and O3/N2/O4 planes make dihedral angles of 6.1 (5) and 32.2 (3)°, respectively, with the benzene ring C11–C16.

Weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into coupled chains extending along the *b* axis (Fig. 2).

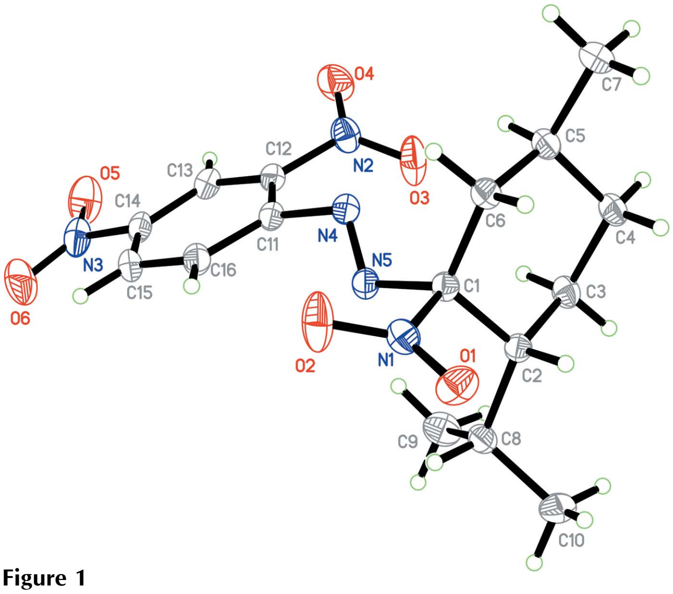


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

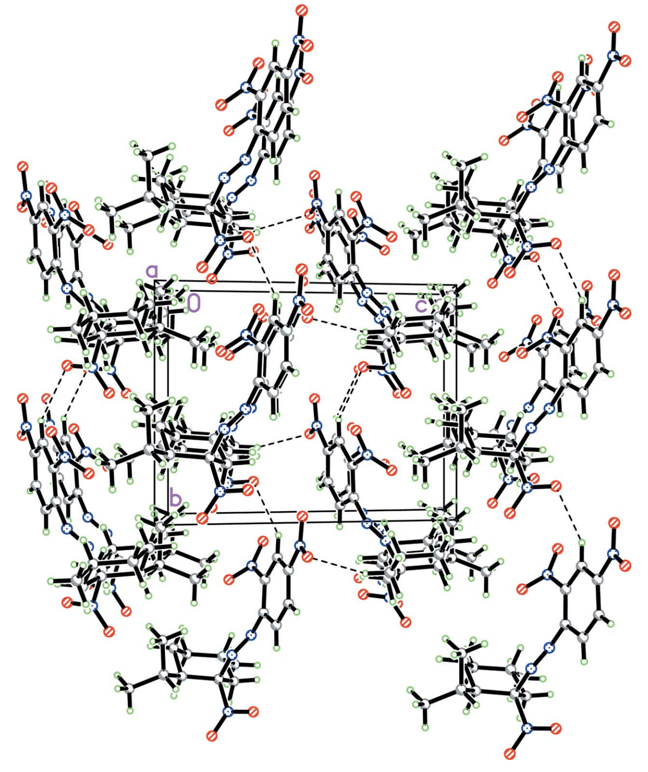


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines denote intermolecular hydrogen bonds.

Experimental

Reagents and solvents used were of commercially available quality. (*E*)-1-(2,4-Dinitrophenyl)-2-(2-isopropyl-5-methylcyclohexylidene)-hydrazine (0.5 mmol, 167 mg) was dissolved in dry CH_2Cl_2 (100 ml). Nitric oxide (NO) was produced by the reaction of a 1 *M* H_2SO_4 solution with saturated $NaNO_2$ aqueous solution. Purified and dried NO was bubbled through a previously degassed stirred stock solution at room temperature. 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 ethyl acetate–hexane (6:1 *v*/*v*) to give the title compound as orange block crystals (yield 89.1%, m.p. 369 K).

Crystal data

$C_{16}H_{21}N_5O_6$
$M_r = 379.38$
Monoclinic, $P2_1$
$a = 8.419\ (2)$ Å
$b = 9.382\ (2)$ Å
$c = 12.366\ (4)$ Å
$\beta = 107.46\ (2)^\circ$
$V = 931.8\ (4)$ Å^3
$Z = 2$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 298\ (2)$ K
Block, orange
0.49 × 0.37 × 0.20 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.950$, $T_{max} = 0.979$
2185 measured reflections
2049 independent reflections
1333 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.5^\circ$
3 standard reflections every 97 reflections intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.087$
$S = 0.88$
2049 reflections
248 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.043 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O2^{i}	0.93	2.42	3.244 (5)	147
C7—H7*B*···O6^{ii}	0.96	2.45	3.346 (5)	155

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 or $1.5U_{eq}$(C). Due to the absence of any significant anomalous scatterers in the molecule, the 1803 Friedel pairs were merged before the final refinement and the absolute configuration was assigned to correspond with that of the known chiral centres in a precursor molecule, which remained unchanged during the synthesis of the title compound.

Data collection: *XSCANS* (Siemens, 1992); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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