

## 3-Methyl-1,5-dinitro-9-phenacyl-3-azabicyclo[3.3.1]non-7-en-6-one

Oleg Ya. Borbulevych,<sup>a,b\*</sup>  
Irina V. Shakheldyan,<sup>c</sup>  
Olga V. Leonova,<sup>c</sup> Yuriy M.  
Atroshchenko<sup>c</sup> and Elena. N.  
Alifanova<sup>c</sup><sup>a</sup>Department of Chemistry, New Mexico  
Highlands University, Las Vegas, NM 87701,  
USA, <sup>b</sup>A. N. Nesmeyanov Institute of Organoelement  
Compounds of the Russian Academy of  
Sciences, 28 Vavilov St., Moscow 119991,  
Russian Federation, and <sup>c</sup>L. N. Tolstoy Tula State  
Pedagogical University, 125 prosp. Lenina, Tula  
300600, Russian FederationCorrespondence e-mail:  
oborbulevych@yahoo.com

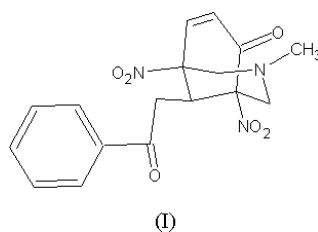
## Key indicators

Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma(\text{C}-\text{C})$  = 0.003 Å  
*R* factor = 0.061  
*wR* factor = 0.169  
Data-to-parameter ratio = 12.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

There are two molecules, *A* and *B*, in the asymmetric unit of the title compound, C<sub>34</sub>H<sub>34</sub>N<sub>6</sub>O<sub>12</sub>. In both molecules, the eight-membered ring has a conformation which is close to a boat–boat conformation and the cyclohexene ring is characterized by a sofa conformation. The nitro groups in each molecule are situated in equatorial positions. Both nitro groups of molecule *A* are considerably rotated with respect to the flattened fragment of the cyclohexene ring. The same is true for one of the nitro groups of molecule *B*, whereas the other such group in molecule *B* is almost coplanar with the corresponding fragment.

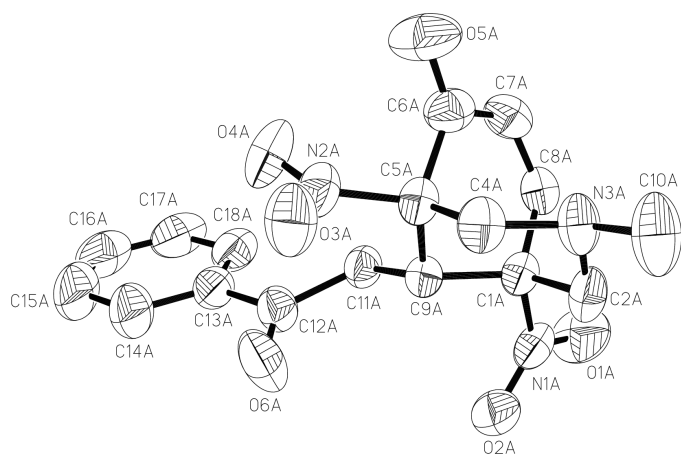
## Comment

The conformational properties of substituted bicyclo[3.3.1]-nonanes and hetero-analogs have been extensively studied by different experimental and computational methods, because of the relationship of these compounds to numerous natural compounds possessing biological activity (Jeyaraman & Avila, 1981; Zefirov & Palyulin, 1991). The influence of the stereochemical and conformational characteristics on the activity is well known. In order to gather more information about this conformationally restricted bicyclic system, the crystal structure of the title compound, (I), was studied by X-ray methods.

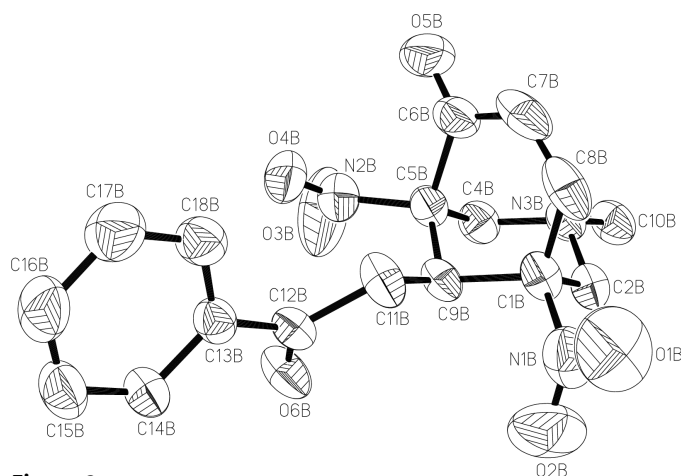


There are two molecules, *A* and *B*, in the asymmetric unit of (I). The piperidine ring adopts a chair conformation. Atoms N3 and C9 deviate from the plane of the remaining atoms of this ring by 0.689 (3) and −0.794 (3) Å, and by −0.688 (3) and 0.781 (3) Å in molecules *A* and *B*, respectively. The cyclohexene ring (C1/C8/C7/C6/C5/C9) has a sofa conformation, with atom C9 deviating by −0.783 (3) and −0.776 (3) Å from the mean plane of the remaining atoms in molecules *A* and *B*, respectively. The conformation of the eight-membered ring is close to a boat–boat conformation and can be characterized by Zefirov–Palyulin puckering parameters (Zefirov *et al.*, 1990) as follows (values are quoted for molecules *A* and *B*, respectively):  $S_2 = 1.208$  and  $1.207$  Å,  $S_3 = 0.580$  and  $0.580$  Å,  $S_4 = 0.514$  and  $-0.509$  Å,  $\varphi_2 = 176.72$  and  $357.55^\circ$ , and  $\varphi_3 = 181.65$  and  $2.07^\circ$ . The nitro groups in both molecules are in equatorial positions (Table 1). Moreover, the nitro groups of molecule *A* and the group at atom C5*B* of molecule *B* are considerably

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**Figure 1**  
A view of molecule *A* of (I). The non-H atoms are shown with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.



**Figure 2**  
A view of molecule *B* of (I). The non-H atoms are shown with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

rotated with respect to the flattened fragment of the cyclohexene ring (Table 1). In contrast, the other nitro group in molecule *B* is almost coplanar with the corresponding fragment. Overall, the most important distinction between molecules *A* and *B* is the different orientations of the nitro groups. Atom N3 is displaced from the plane of its three neighboring C atoms by 0.439 (3) and  $-0.432$  (2) Å in molecules *A* and *B*, respectively, reflecting the trigonal-pyramidal configuration of this fragment. The phenacyl group is in an axial position in the cyclohexene ring, whereas it has an equatorial orientation with respect to the piperidine ring (the relevant torsion angles are listed in Table 1).

## Experimental

Compound (I) was obtained according to the procedure of Leonova *et al.* (2001). Crystals of (I) were grown by slow evaporation of a toluene solution.

## Crystal data

$C_{17}H_{17}N_3O_6$   
 $M_r = 359.34$   
 Triclinic,  $P\bar{1}$   
 $a = 11.374$  (6) Å  
 $b = 11.669$  (5) Å  
 $c = 13.715$  (6) Å  
 $\alpha = 84.56$  (4)°  
 $\beta = 75.64$  (4)°  
 $\gamma = 75.07$  (4)°  
 $V = 1702.9$  (14) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.402$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 24 reflections  
 $\theta = 10\text{--}11^\circ$   
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Square prism, yellow  
 $0.50 \times 0.30 \times 0.30$  mm

## Data collection

Siemens P3 diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 6198 measured reflections  
 5875 independent reflections  
 4198 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.038$

$\theta_{max} = 25.0^\circ$   
 $h = 0 \rightarrow 13$   
 $k = -13 \rightarrow 13$   
 $l = -15 \rightarrow 16$   
 2 standard reflections  
 every 98 reflections  
 intensity decay: 3.4%

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.061$   
 $wR(F^2) = 0.169$   
 $S = 0.95$   
 5875 reflections  
 469 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1289P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.27$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (°).

N1A—C1A—C9A—C5A	173.81 (15)	N1B—C1B—C9B—C5B	-175.72 (17)
N2A—C5A—C9A—C1A	176.45 (15)	N2B—C5B—C9B—C1B	-176.40 (17)
O1A—N1A—C1A—C8A	25.3 (2)	O1B—N1B—C1B—C8B	-4.1 (3)
O4A—N2A—C5A—C6A	47.0 (3)	O4B—N2B—C5B—C6B	-58.9 (3)
C4A—C5A—C9A—C11A	172.19 (16)	C4B—C5B—C9B—C11B	-173.97 (16)
C6A—C5A—C9A—C11A	-66.5 (2)	C6B—C5B—C9B—C11B	66.0 (2)

Zefirov–Palyulin puckering parameters were calculated with *RICON* (Zotov *et al.*, 1997). All H atoms were located from difference Fourier syntheses. Methyl H atoms were refined as part of rigid groups, which were allowed to rotate but not tip or distort, and with  $U_{iso}(H) = 1.5U_{eq}(C)$ . Other H atoms were refined using a riding model, with  $U_{iso}(H) = 1.2U_{eq}(C)$ .

Data collection: P3 (Siemens, 1989); cell refinement: P3; data reduction: XDISK (Siemens, 1991); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXL97; software used to prepare material for publication: SHELXL97.

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