Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,5-Diacetyl-7,7-dinitro-1,5-diazacyclooctan-3-one ethylene acetal

Richard D. Gilardi,[a] Theodore Axenrod,[b] Xiao-Pei Guan[b] and Judith L. Flippen-Anderson[a]*

[a]Laboratory for the Structure of Matter, Code 6030, Naval Research Laboratory, Washington, DC 20375, USA, and [b]Department of Chemistry, The City College of the City University of New York, New York, NY 10031, USA

Correspondence e-mail: flippen@harker.nrl.navy.mil

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.113
Data-to-parameter ratio = 12.0

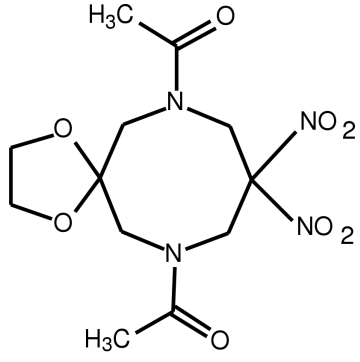
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 October 2001
Accepted 8 November 2001
Online 17 November 2001

The solid state structure of the title compound, $C_{12}H_{18}N_4O_8$, has been determined. There are two molecules in the asymmetric unit that differ in the conformations of their eight-membered heterocyclic rings and in the orientations of the nitro groups.

Comment

Several synthetic routes are being developed to prepare the diversified energetic heterocycle 3,3-bis(difluoramino)octahydro-1,5,7,7-tetranitro-1,5-diazacyclooctane (TNFX; Axenrod *et al.*, 2001). The title compound, (I), is a key intermediate in this process that permits the selective introduction of the asymmetrically functionalized nitro and difluoramino substituents while simultaneously circumventing the common transannular reactions that occur in the diazacyclooctane ring system. It crystallizes with two molecules per asymmetric unit which exhibit different conformations. One molecule (Fig. 1) has an approximate twofold axis passing through C3 and C7. The eight-membered ring has four atoms (N1, N5, C2 and C6) in a plane (±0.02 Å), with C3 and C4 approximately 1 Å above the plane, and C7 and C8 approximately 1 Å below the plane. The angle between the planes through the two nitro groups is 78.30 (6)°. The second molecule (Fig. 2) does not exhibit a non-crystallographic twofold axis. Its heterocyclic ring has five atoms (N11, N15, C13, C14 and C18) in a plane (±0.04 Å) with C16 and C17 approximately 1 Å above the plane and C12 0.8 Å below it. In this molecule, the angle between the planes through the geminal nitro groups is 99.87 (7)°.



(I)

Experimental

Crystals of the title compound were synthesized and prepared by T. Axenrod (Axenrod *et al.*, 2001).

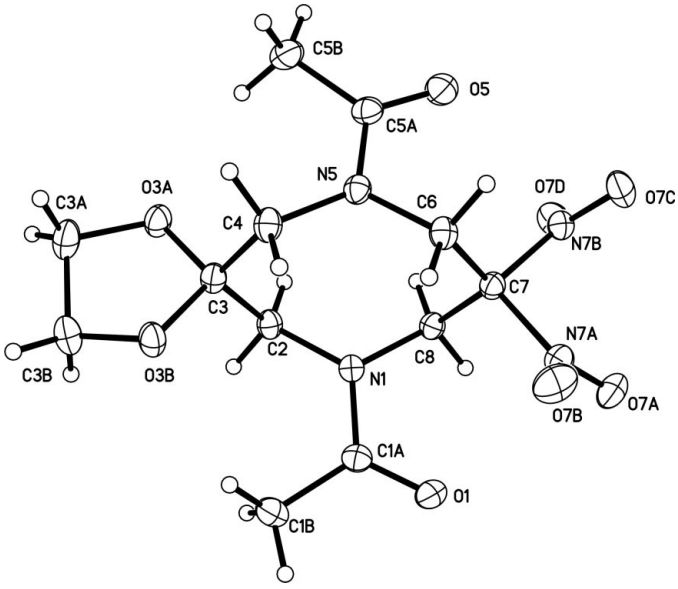


Figure 1
View of one of the two independent molecules of (I) with 20% probability ellipsoids.

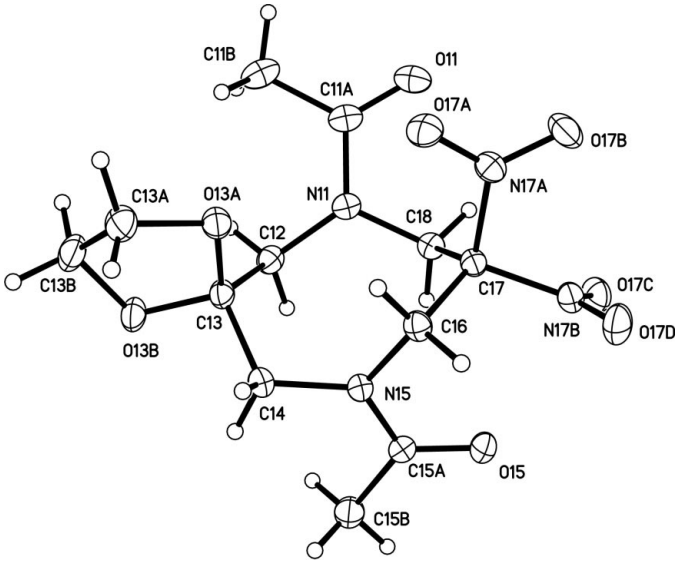


Figure 2
View of the second independent molecule of (I) with 20% probability ellipsoids.

Crystal data

$C_{12}H_{18}N_4O_8$
$M_r = 346.30$
Monoclinic, $P2_1/c$
$a = 17.9560\ (2)$ Å
$b = 9.7885\ (1)$ Å
$c = 18.2898\ (1)$ Å
$\beta = 108.507\ (1)^\circ$
$V = 3048.41\ (5)$ Å^3
$Z = 8$
$D_x = 1.509$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 7327 reflections
$\theta = 4.7$–57.1°
$\mu = 1.11$ mm^{-1}
$T = 293\ (2)$ K
Irregular, colorless
$0.24 \times 0.21 \times 0.10$ mm

Data collection

Bruker SMART 6000 diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.670$, $T_{max} = 0.864$
21447 measured reflections
5243 independent reflections
4537 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 67.2^\circ$
$h = -20 \rightarrow 21$
$k = -11 \rightarrow 10$
$l = -21 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.113$
$S = 1.05$
5243 reflections
438 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0766P)^2 + 0.2099P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.031$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0109 (5)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001).

The NRL authors wish to acknowledge financial support from the Office of Naval Research, Mechanics Division.

References

Axenrod, T., Guan, X.-P., Sun, J., Qi, L., Chapman, R. & Gilardi, R. D. (2001). *Tetrahedron Lett.* **42**, 2621–2623.
Bruker (2001). *SMART* (Version 5.624) and *SAINT* (Version 6.04) for Windows-NT, and *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10) for UNIX. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.