

## 1,3,5,7-tetranitro-3,7-diazabicyclo[3.3.1]nonane

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## Key indicators

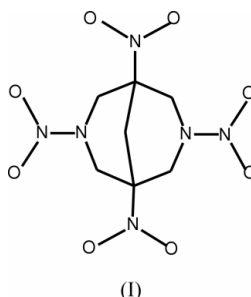
Single-crystal X-ray study  
 $T = 293$  K  
 Mean  $\sigma(C-C) = 0.002$  Å  
 Disorder in main residue  
 $R$  factor = 0.056  
 $wR$  factor = 0.147  
 Data-to-parameter ratio = 12.9

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

The conformation and density were determined for the title compound,  $C_7H_{10}N_6O_8$ . As expected, the bicyclic nine-membered ring has two chair-shaped azacyclohexane rings.

## Comment

A key thrust of much current research in energetic materials is the development of higher performance materials to defeat increasingly hardened targets. Important goals are new crystalline explosive fillers with energy density exceeding HMX (1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane), as well as new nitramines capable of replacing ammonium perchlorate as the mainstay of many present missile propellants. The title compound, (I), is a variant similar to HMX in that it contains four nitro groups, but only two are nitramines (four in HMX) and the other two are C-nitro groupings that are considered less sensitive than nitramines. However, the density is 10% less than HMX (Cady *et al.*, 1963), which lowers its efficacy as an energetic material. Although there could be a vertical twofold axis passing through C9, and this is roughly so for the ring system (two chair-shaped azacyclohexane rings), there is no crystallographic symmetry, and corresponding nitro groups, especially those on N3 and N7, differ from each other in their orientation (Fig. 2).



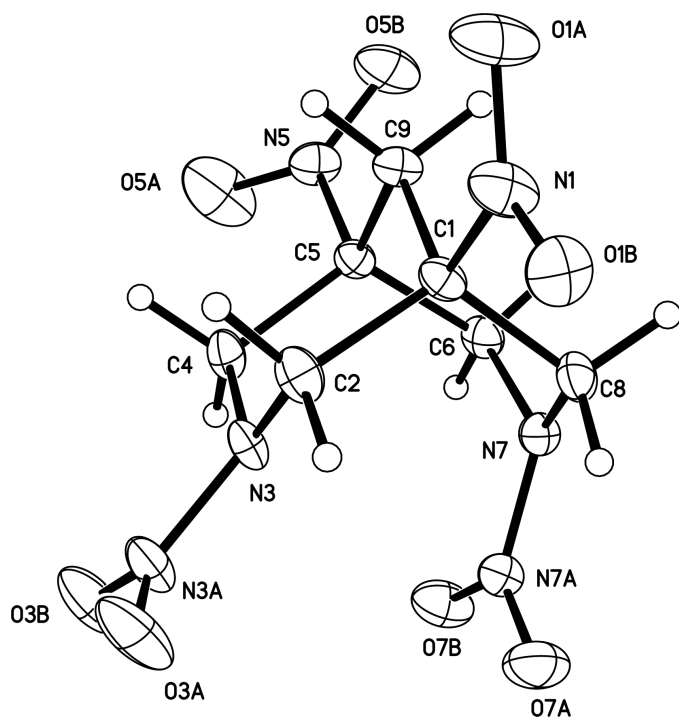
## Experimental

The nitrolysis of 3,7-di-*tert*-butyl-1,5-dinitro-3,7-diazabicyclo[3.3.1]-nonane (Flippen-Anderson *et al.*, 2001) was carried out in 100% nitric acid to afford the title compound in 52% yield.

## Crystal data

$C_7H_{10}N_6O_8$   
 $M_r = 306.21$   
 Monoclinic,  $P2_1/c$   
 $a = 13.5750$  (8) Å  
 $b = 7.9570$  (4) Å  
 $c = 11.0684$  (6) Å  
 $\beta = 93.436$  (1)°  
 $V = 1193.42$  (11) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.704$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 5710 reflections  
 $\theta = 4.6$ – $56.6$ °  
 $\mu = 0.16$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, colorless  
 $0.66 \times 0.66 \times 0.23$  mm



**Figure 1**

View of 1,3,5,7-tetranitro-3,7-diazabicyclo[3.3.1]nonane with 30% probability ellipsoids.

#### Data collection

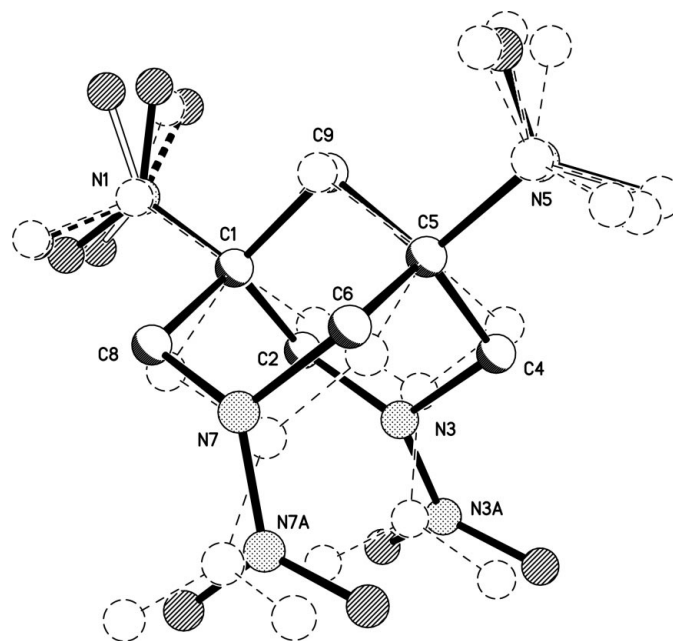
Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2001)  
 $T_{\min} = 0.89$ ,  $T_{\max} = 0.96$   
 8750 measured reflections

2971 independent reflections  
 2466 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$   
 $\theta_{\text{max}} = 29.1^\circ$   
 $h = -18 \rightarrow 17$   
 $k = -4 \rightarrow 10$   
 $l = -14 \rightarrow 13$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.147$   
 $S = 1.10$   
 2971 reflections  
 230 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.5997P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.003$   
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.23 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: SHELXL  
 Extinction coefficient: 0.035 (4)



**Figure 2**

Superposition of the molecule and its image obtained by twofold rotation about C9, showing the slight differences in orientation of the nitro groups and the disordered nitro group on N1.

One of the nitro groups, involving N1, is disordered over three sites with occupancies of 0.54 (3), 0.24 (2) and 0.22 (2); the other three nitro groups are well ordered.

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

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#### References

- Bruker (2001). SMART and SAINT. Version 5.624 and version 6.04 for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Cady, H. H., Larson, A. C. & Cromer, D. T. (1963). *Acta Cryst.* **16** 617–623.  
 Flippen-Anderson, J. L., Axenrod, T., Sun, J. & Gilardi, R. D. (2001). *Acta Cryst.* **E57**, o1128–o1129.  
 Sheldrick, G. M. (1997). SHELXTL, Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (2001). SADABS. Version 2.03. Bruker AXS Inc., Madison, Wisconsin, USA.