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3,7-Di-*tert*-butyl-1,5-dinitro-3,7-diazabicyclo[3.3.1]-nonane

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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 = 0.064
wR factor = 0.195
Data-to-parameter ratio = 13.4

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

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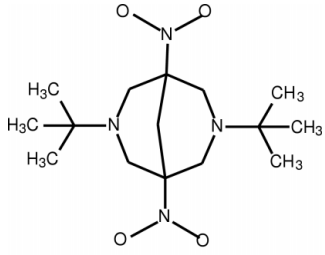
The conformation of the title compound, $C_{15}H_{28}N_4O_4$, was determined. The tricyclic nine-membered ring has a boat-shaped azacyclohexane ring on one side of the bridge and a chair-shaped ring on the other side.

Comment

The development of synthetic routes to new cyclic energetic materials with performance properties superior to HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazacyclooctane), but with less sensitivity toward certain stimuli, is a continuing effort. Although they would be expected to be chemically equivalent, the bridged nine-membered ring of the title compound, (I), has a boat-shaped azacyclohexane on one side and and a chair-shaped azacyclohexane ring on the other side. This must be due to steric hindrance between the two *tert*-butyl groups. The all-nitro version (1,3,5,7-tetranitro-3,7,-diazabicyclo[3.3.1]-nonane; Gilardi, 2001) of this molecule does have two chair-shaped rings. A least-squares fit of the two independent molecules, using all atoms except the nitro-O atoms, gives a weighted r.m.s. deviation of 0.08 Å.



(I)

Experimental

The title compound was the minor reaction product formed by the Mannnich condensation of nitromethane with formaldehyde and *tert*-butylamine. The major product formed was the corresponding six-membered analog 1,2-di-*tert*-butyl-5-(*tert*-butylaminomethyl)-5-nitro-1,3-diazacyclohexane.

Crystal data

$C_{15}H_{28}N_4O_4$
M_r = 328.41
Monoclinic, $P2_1/n$
a = 11.9782 (2) Å
b = 9.9382 (1) Å
c = 30.5174 (4) Å
β = 100.812 (1)°
V = 3568.35 (8) Å^3
Z = 8
D_x = 1.223 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 5644 reflections
θ = 3.0–67.0°
μ = 0.73 mm^{-1}
T = 293 (2) K
Plate, colorless
0.36 × 0.30 × 0.06 mm

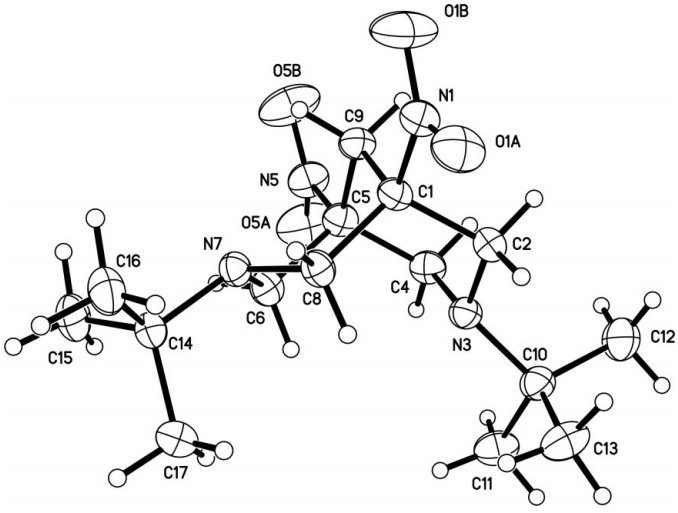


Figure 1
View of 3,7-di-*tert*-butyl-1,5-dinitro-3,7-diazabicyclo[3.3.1]nonane with 20% probability ellipsoids. Only one of the two molecules in the asymmetric unit is shown.

Data collection

Bruker SMART 6000 diffractometer	5860 independent reflections
ω scans	4673 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)	$R_{int} = 0.038$
$T_{min} = 0.622$, $T_{max} = 0.929$	$\theta_{max} = 67.0^\circ$
17521 measured reflections	$h = -12 \rightarrow 13$
	$k = -10 \rightarrow 11$
	$l = -36 \rightarrow 33$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1273P)^2 + 0.8387P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.064$	$(\Delta/\sigma)_{max} = 0.028$
$wR(F^2) = 0.195$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
5860 reflections	Extinction correction: *SHELXL*97
436 parameters	Extinction coefficient: 0.0080 (5)
H-atom parameters constrained	

Of the four nitro groups in the asymmetric unit only one is disordered (66:34).

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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