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Structure of 7-Acetyl-2,5,9-trinitro-2,5,7,9-tetraazabicyclo[4.3.0]nonan-8-one

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Abstract. $C_7H_9N_7O_8$, $M_r = 319.2$, monoclinic, $P2_1/n$, $a = 10.615$ (1), $b = 9.115$ (2), $c = 12.948$ (2) Å, $\beta = 108.57$ (1)°, $V = 1187.6$ (3) Å^3, $Z = 4$, $D_x = 1.785$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.38$ mm^{-1}, $F(000) = 656$, $T = 295$ K, final $R = 0.039$, $wR = 0.044$ for 1327 independent observed reflections. The five-membered ring has a normal envelope conformation while the six-membered ring has adopted a twisted boat conformation. Of the three nitroamine groups one is planar and the other two are pyramidal with N—N to C—N—C angles of 0.5, 43.4 and 44.8° respectively.

Experimental. A clear colorless prism 0.15 × 0.20 × 0.26 mm data crystal was provided by Dr Clifford L. Coon of Livermore National Laboratory, California. An automated Siemens *R3m/V* diffractometer with incident beam monochromator was used for data collection. 25 centered reflections within $20.0 \le 2\theta \le 77.0°$ were used for determining lattice parameters. $[\sin(\theta)/\lambda]_{max} = 0.54$ Å^{-1}; range of *hkl*: $-11 \le h \le 10$ $-9 \le k \le 2$, $0 \le l \le 13$. Standards 200, 020 and 002, monitored every 97 reflections, showed random variation of 2.5% over data collection: $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]°$, ω-scan rate a function of count rate (7.0° min^{-1} minimum, 15.0° min^{-1} maximum), 1808 reflections measured, 1545 unique, $R_{int} = 1.2\%$, 1327 observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects.

The structure solution, by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL*80 (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ was minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.000225$. Secondary-extinction parameter $p = 0.004$ (1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 236 parameters were refined: atomic coordinates for all atoms, anisotropic thermal parameters for all non-H atoms and isotropic thermal parameters for H atoms. $(\Delta/\sigma)_{max} = 0.12$, ratio of observations to parameters = 5.6:1, $R = 0.039$, $wR = 0.044$, $S = 1.39$ ($R = 0.046$ for all data). Final difference Fourier excursions were 0.18 and −0.20 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).† Atom numbering for Tables 1 (atom coordinates) and 2 (bond distances and angles) follows that shown in Fig. 1.

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54969 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0573]

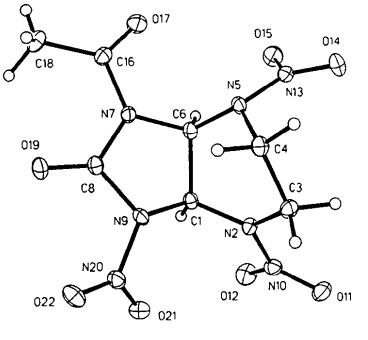

Fig. 1. A thermal ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	4273 (3)	−2976 (3)	6364 (3)	30 (1)
N(2)	4247 (2)	−1434 (3)	6185 (2)	31 (1)
C(3)	4953 (3)	−385 (4)	7018 (3)	36 (1)
C(4)	5389 (3)	−1164 (4)	8108 (3)	32 (1)
N(5)	4341 (2)	−2146 (3)	8205 (2)	31 (1)
C(6)	4015 (3)	−3358 (3)	7442 (2)	30 (1)
N(7)	4881 (2)	−4608 (3)	7853 (2)	30 (1)
C(8)	5839 (3)	−4798 (3)	7344 (2)	32 (1)
N(9)	5611 (2)	−3612 (3)	6577 (2)	32 (1)
N(10)	3514 (3)	−889 (3)	5186 (2)	36 (1)
O(11)	3622 (2)	424 (3)	5060 (2)	47 (1)
O(12)	2806 (2)	−1733 (3)	4514 (2)	55 (1)
N(13)	3283 (3)	−1482 (3)	8446 (2)	36 (1)
O(14)	3492 (2)	−259 (3)	8857 (2)	50 (1)
O(15)	2260 (2)	−2179 (3)	8286 (2)	50 (1)
C(16)	4566 (3)	−5599 (3)	8581 (3)	36 (1)
O(17)	3609 (2)	−5300 (3)	8854 (2)	50 (1)
C(18)	5427 (4)	−6898 (4)	8938 (4)	47 (2)
O(19)	6670 (2)	−5717 (2)	7488 (2)	43 (1)
N(20)	6089 (3)	−3775 (3)	5669 (2)	40 (1)
O(21)	5318 (2)	−3427 (3)	4793 (2)	49 (1)
O(22)	7212 (3)	−4213 (3)	5877 (2)	60 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—N(2)	1.423 (4)	C(1)—C(6)	1.545 (5)
C(1)—N(9)	1.476 (4)	N(2)—C(3)	1.458 (4)
N(2)—N(10)	1.373 (3)	C(3)—C(4)	1.514 (5)
C(4)—N(5)	1.464 (4)	N(5)—C(6)	1.449 (4)
N(5)—N(13)	1.395 (4)	C(6)—N(7)	1.454 (4)
N(7)—C(8)	1.388 (5)	N(7)—C(16)	1.419 (4)
C(8)—N(9)	1.435 (4)	C(8)—O(19)	1.187 (4)
N(9)—N(20)	1.429 (5)	N(10)—O(11)	1.218 (4)
N(10)—O(12)	1.223 (3)	N(13)—O(14)	1.225 (4)
N(13)—O(15)	1.218 (4)	C(16)—O(17)	1.208 (5)
C(16)—C(18)	1.476 (5)	N(20)—O(21)	1.211 (3)
N(20)—O(22)	1.203 (4)		

Table 2 (*cont.*)

N(2)—C(1)—C(6)	111.7 (3)	N(2)—C(1)—N(9)	112.7 (2)
C(6)—C(1)—N(9)	100.4 (2)	C(1)—N(2)—C(3)	123.3 (2)
C(1)—N(2)—N(10)	119.1 (2)	C(3)—N(2)—N(10)	117.6 (2)
N(2)—C(3)—C(4)	108.3 (3)	C(3)—C(4)—N(5)	110.6 (2)
C(4)—N(5)—C(6)	115.5 (3)	C(4)—N(5)—N(13)	116.2 (2)
C(6)—N(5)—N(13)	116.7 (2)	C(1)—C(6)—N(5)	111.8 (2)
C(1)—C(6)—N(7)	103.6 (3)	N(5)—C(6)—N(7)	111.2 (2)
C(6)—N(7)—C(8)	113.4 (3)	C(6)—N(7)—C(16)	119.1 (3)
C(8)—N(7)—C(16)	126.8 (2)	N(7)—C(8)—N(9)	104.3 (2)
N(7)—C(8)—O(19)	129.3 (3)	N(9)—C(8)—O(19)	126.4 (3)
C(1)—N(9)—C(8)	111.4 (3)	C(1)—N(9)—N(20)	117.4 (2)
C(8)—N(9)—N(20)	117.7 (2)	N(2)—N(10)—O(11)	115.6 (2)
N(2)—N(10)—O(12)	118.6 (3)	O(11)—N(10)—O(12)	125.8 (3)
N(5)—N(13)—O(14)	116.4 (3)	N(5)—N(13)—O(15)	118.2 (3)
O(14)—N(13)—O(15)	125.2 (3)	N(7)—C(16)—O(17)	116.6 (3)
N(7)—C(16)—C(18)	117.7 (3)	O(17)—C(16)—C(18)	125.7 (3)
N(9)—N(20)—O(21)	115.6 (3)	N(9)—N(20)—O(22)	115.9 (3)
O(21)—N(20)—O(22)	128.5 (4)		

Related literature. The structures of two related compounds, both having the acetyl group on N(7) replaced with a fourth nitro group and one having the carbonyl O atom replaced with an acetoxy moiety, have been reported (Flippen-Anderson, George & Gilardi, 1990).

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Structures of (I) 6-Nitro-2,4-dipropionyl-8-oxa-2,4,6-triazabicyclo[3.3.0]octane and (II) 2,4-Diacetyl-6-nitro-8-oxa-2,4,6-triazabicyclo[3.3.0]octane

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Abstract. (I) $C_{10}H_{16}N_4O_5$, $M_r = 272.3$, monoclinic, $P2_1/n$, $a = 6.151$ (2), $b = 13.484$ (7), $c = 15.165$ (9) Å, $\beta = 90.94$ (5)°, $V = 1257.6$ (9) Å^3, $Z = 4$, $D_x = 1.438$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.11$ mm^{-1}, $F(000) = 576$, $T = 295$ K, final $R = 0.046$, $wR = 0.038$ for 1274 independent observed reflections. (II) $C_8H_{12}N_4O_5$, $M_r = 244.2$, monoclinic, $P2_1/c$, $a = 7.801$ (1), $b = 19.885$ (3), $c = 7.077$ (1) Å, $\beta = 91.35$ (2)°, $V = 1097.5$ (3) Å^3, $Z = 4$, $D_x = 1.478$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 1.02$ mm^{-1}, $F(000) = 512$, $T = 295$ K, final $R = 0.039$, $wR = 0.051$ for 1718 independent observed reflections. In (I) both rings have an envelope conformation while in (II) the acetyl substituted ring is planar. The nitroamine group is pyramidal in both molecules with an N—N to C—N—C angle of 43.1 in (I) and 42.6° in (II). In (I) the carbonyl O atoms of the propionyl groups are on opposite sides of the C—N—C—N—C chain whereas in (II) the acetyl carbonyls are on the same side of the chain.

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