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Structure of 2,4,6-Trinitro-9-oxo-2,4,6,8-tetraaza-1-decanyl Acetate

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Abstract. $C_8H_{15}N_7O_9$, $M_r = 353{\cdot}25$, orthorhombic, *Pbca*, $a = 12{\cdot}410$ (2), $b = 9{\cdot}695$ (1), $c = 25{\cdot}079$ (3) Å, $V = 3017{\cdot}5$ (6) Å^3, $Z = 8$, $D_x = 1{\cdot}555$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 11{\cdot}9$ cm^{-1}, $F(000) = 1472$, $T = 295$ K, final $R = 0{\cdot}066$, $wR = 0{\cdot}067$ for 1427 independent reflections. The acetoxy group is disordered such that the two conformations for the group have occupancies of 57 and 43%. Bond distances and angles are normal and intermolecular hydrogen bonding occurs between the secondary amine and the nondisordered acetyl O atom.

Experimental. A clear colorless 0·05 × 0·10 × 0·25 mm data crystal, crystallized from methyl chloride was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet *R*3*m* diffractometer with incident-beam monochromator, 25 centered reflections within $30 \le 2\theta \le 50°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}54$ Å^{-1}, range of *hkl*: $-13 \le h \le 1$, $0 \le k \le 10$, $0 \le l \le 14$. Standards 400, 040, 006, monitored every 60 reflections with random variation of 4·6% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (2·0° min^{-1} minimum, 30·0° min^{-1} maximum), 2782 reflections measured, 2082 unique, $R_{int} = 0{\cdot}02$, 1427 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not for absorption effects. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00040$.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

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

	x	*y*	*z*	U_{eq}
N(1)	−328 (3)	632 (4)	2225 (2)	49 (1)
C(2)	−1053 (4)	875 (5)	1791 (2)	47 (2)
N(3)	−523 (4)	1061 (4)	1273 (2)	47 (1)
C(4)	−385 (4)	2385 (5)	1017 (2)	46 (2)
N(5)	−1266 (3)	2767 (4)	662 (2)	45 (1)
C(6)	−1350 (4)	2316 (5)	114 (2)	46 (2)
N(7)	−1120 (5)	3357 (5)	−282 (2)	59 (2)
C(10)	187 (4)	1666 (6)	2467 (2)	47 (2)
O(11)	68 (3)	2862 (3)	2320 (1)	57 (1)
C(12)	875 (5)	1266 (6)	2923 (2)	74 (2)
N(13)	−78 (4)	−76 (5)	1042 (2)	56 (2)
O(14)	356 (3)	67 (4)	608 (2)	71 (1)
O(15)	−158 (3)	−1170 (4)	1287 (2)	71 (1)
N(16)	−2061 (4)	3583 (5)	854 (2)	51 (2)
O(17)	−2715 (3)	4035 (4)	537 (2)	69 (1)
O(18)	−2062 (3)	3823 (4)	1337 (2)	70 (1)
N(19)	−56 (6)	3637 (5)	−382 (2)	71 (2)
O(20)	611 (4)	3123 (5)	−83 (2)	81 (2)
O(21)	143 (5)	4398 (5)	−755 (2)	111 (2)
C(8)	−1925 (6)	3880 (7)	−627 (2)	104 (2)
O(9)*	−1701 (7)	3726 (7)	−1178 (3)	88 (3)
C(22)*	−2251 (7)	2487 (8)	−1285 (3)	54 (2)
O(23)*	−2766 (7)	1915 (8)	−931 (3)	79 (3)
C(24)*	−2298 (9)	2052 (12)	−1860 (3)	87 (3)
O(9*a*)†	−2093 (11)	2721 (13)	−1012 (4)	106 (3)
C(22*a*)†	−2612 (10)	2595 (11)	−1516 (4)	110 (4)
O(23*a*)†	−2525 (19)	3793 (10)	−1671 (5)	263 (3)
C(24*a*)†	−2197 (13)	1382 (11)	−1829 (6)	77 (3)

* Site-occupancy factor = 0·570 (4).
† Site-occupancy factor = 0·430 (4).

Table 2. *Bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and* H-*bond parameters* (Å, °)

N(1)–C(2)	1·432 (6)	N(1)–C(10)	1·336 (7)
C(2)–N(3)	1·467 (6)	N(3)–C(4)	1·445 (6)
N(3)–N(13)	1·361 (6)	C(4)–N(5)	1·456 (6)
N(5)–C(6)	1·446 (6)	N(5)–N(16)	1·354 (6)
C(6)–N(7)	1·445 (6)	N(7)–N(19)	1·372 (9)
N(7)–C(8)	1·415 (9)	C(10)–O(11)	1·227 (6)
C(10)–C(12)	1·477 (8)	N(13)–O(14)	1·222 (6)
N(13)–O(15)	1·229 (6)	N(16)–O(17)	1·217 (6)
N(16)–O(18)	1·233 (6)	N(19)–O(20)	1·222 (8)
N(19)–O(21)	1·217 (7)	C(8)–O(9)	1·418 (8)
C(8)–O(9*a*)	1·495 (13)	O(9)–C(22)	1·407 (11)
C(22)–O(23)	1·227 (11)	C(22)–C(24)	1·504 (11)
O(9*a*)–C(22*a*)	1·426 (15)	C(22*a*)–O(23*a*)	1·229 (15)
C(22*a*)–C(24*a*)	1·504 (17)		

C(2)–N(1)–C(10)	121·6 (4)	N(1)–C(2)–N(3)	114·3 (4)
C(2)–N(3)–C(4)	123·7 (4)	C(2)–N(3)–N(13)	117·3 (4)
C(4)–N(3)–N(13)	118·8 (4)	N(3)–C(4)–N(5)	114·1 (4)
C(4)–N(5)–C(6)	123·9 (4)	C(4)–N(5)–N(16)	118·6 (4)
C(6)–N(5)–N(16)	117·5 (4)	N(5)–C(6)–N(7)	115·4 (4)
C(6)–N(7)–N(19)	117·0 (5)	C(6)–N(7)–C(8)	122·1 (5)
N(19)–N(7)–C(8)	119·9 (5)	N(1)–C(10)–O(11)	121·0 (5)
N(1)–C(10)–C(12)	115·6 (5)	O(11)–C(10)–C(12)	123·4 (5)
N(3)–N(13)–O(14)	117·7 (4)	N(3)–N(13)–O(15)	117·0 (4)
O(14)–N(13)–O(15)	125·3 (5)	N(5)–N(16)–O(17)	117·7 (4)
N(5)–N(16)–O(18)	117·3 (4)	O(17)–N(16)–O(18)	125·0 (5)
N(7)–N(19)–O(20)	117·4 (5)	N(7)–N(19)–O(21)	117·0 (6)
O(20)–N(19)–O(21)	125·6 (7)	N(7)–C(8)–O(9)	114·8 (6)
N(7)–C(8)–O(9*a*)	102·9 (7)	C(8)–O(9)–C(22)	100·4 (6)
O(9)–C(22)–O(23)	120·0 (8)	O(9)–C(22)–C(24)	116·1 (7)
O(23)–C(22)–C(24)	123·2 (8)	C(8)–O(9*a*)–C(22*a*)	134·4 (10)
O(9*a*)–C(22*a*)–O(23*a*)	99·2 (11)	O(9*a*)–C(22*a*)–C(24*a*)	112·0 (11)
O(23*a*)–C(22*a*)–C(24*a*)	123·0 (11)		

N(1)–C(2)–N(3)–C(4)	−103·7 (5)	N(5)–C(6)–N(7)–C(8)	−112·6 (5)
C(2)–N(3)–C(4)–N(5)	−90·7 (5)	C(6)–N(7)–C(8)–O(9)	−121·9 (5)
N(3)–C(4)–N(5)–C(6)	−83·4 (5)	C(6)–N(7)–C(8)–O(9*a*)	−73·0 (6)
C(4)–N(5)–C(6)–N(7)	−106·7 (5)		

	H···O	N···O	∠N–H···O
N(1)–H(1)···O(11)′	2·17 (3)	2·838 (7)	175·5 (25)

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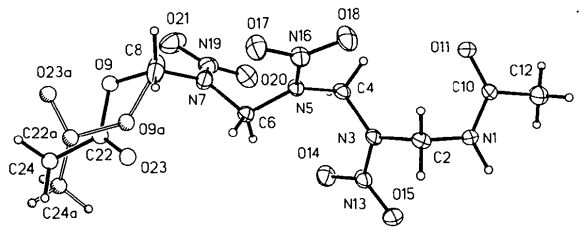

Fig. 1. Thermal ellipsoid plot of the title compound with ellipsoids drawn at the 20% probability level for nondisordered atoms. The disordered atoms are drawn as fixed-radius spheres. The open bonds represent the lower-occupancy conformation for the acetoxy group. The C(8) methylene H atoms for the lower-occupancy conformation are omitted for clarity.

Secondary-extinction parameter $p = 0{\cdot}0010\ (2)$ in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002\ (p)F_o^2/\sin(2\theta)]^{0.25}$. There were 256 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model in *SHELXTL*, H riding on C, C—H = 0·96 Å. $U(\mathrm{H}) = 1{\cdot}2U_{eq}(\mathrm{C})$. Amine hydrogens refined isotropically. Site occupation for the two conformations of the acetoxy group was variable and constrained to sum to unity, restraint applied to next-nearest-neighbor C···O distance (2·405 ±0·008 Å) from each disordered terminal methyl C to respective acetyl O, C(8) treated as a pivot atom with methylene hydrogen pairs idealized for the two conformations and with the previously stated constraint on site occupation. $(\Delta/\sigma)_{max} = 0{\cdot}12$, $R = 0{\cdot}066$, $wR = 0{\cdot}067$, $S = 1{\cdot}825$. Final difference Fourier excursions 0·38 and −0·34 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Table 1, atom coordinates, and Table 2, bond distances, bond angles and selected torsion angles, follows that shown in Fig. 1.

Related literature. For the structure of a similar compound, 2,4,6-trinitro-2,4,6-triaza-1,7-heptanediyl diacetate, see Cobbledick & Small (1973*a–c*). For a similarly substituted pentaazanonane, see George & Gilardi (1989).

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* Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51806 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4,7,8-Trimethoxyfuro[2,3-*b*]quinoline

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Abstract. Skimmianine, $C_{14}H_{13}NO_4$, $M_r = 259{\cdot}3$, monoclinic, $P2_1/c$, $a = 7{\cdot}390\ (1)$, $b = 10{\cdot}458\ (1)$, $c = 15{\cdot}584\ (4)$ Å, $\beta = 94{\cdot}92\ (1)°$, $V = 1199\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}435$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\bar{\alpha}) = 1{\cdot}5418$ Å, $\mu = 8{\cdot}4$ cm^{-1}, $F(000) = 544$, $T = 297$ K, $R = 0{\cdot}043$ for 2408 observations with $I > 3\sigma(I)$ (of 2656 unique data). The orientation of the C4-methoxy group towards the C3 hydrogen corresponds to that predicted in solution by NMR studies. No unusual bond distances or angles are seen in the structure. Intermolecular distances correspond to van der Waals contacts.

Experimental. The title compound was isolated from the chloroform extract of the dried fruits of *Fagara rhoifolia*. The concentrated chloroform extract was subjected to column chromatography on silica gel using

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