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Structure of 6-Hydroxy-2,5-dinitro-2,5-diazacyclohexyl Acetate

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Abstract. $C_6H_{10}N_4O_7$, $M_r = 250{\cdot}17$, monoclinic, $P2_1/c$, $a = 13{\cdot}588$ (2), $b = 7{\cdot}276$ (1), $c = 10{\cdot}830$ (1) Å, $\beta = 109{\cdot}75$ (1)°, $V = 1007{\cdot}66$ (19) Å^3, $Z = 4$, $D_x = 1{\cdot}65$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1{\cdot}28$ mm^{-1}, $F(000) = 520$, $T = 295$ K, final $R = 0{\cdot}029$, $wR = 0{\cdot}028$ for 1230 observed reflections. The ring has a normal chair conformation. Both ring N atoms are pyramidal; the angles between the exocyclic N–N bonds and the CNC planes are 32·9 and 36·6°. There is an intermolecular hydrogen bond (2·998 Å) between the hydroxy oxygen and one of the nitro-group oxygens.

Experimental. Colorless 0·1 × 0·15 × 0·4 mm crystal. Synthesized by C. Coon of Lawrence Livermore Laboratory, Livermore, CA, USA. Automated Nicolet *R*3*M* diffractometer with incident-beam graphite monochromator, 25 centered reflections within $15 < 2\theta < 81°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{max} = 115°$, range of hkl $-14 \le h \le 14$, $0 \le k \le 7$, $0 \le l \le 10$. Standards $\bar{1}3,0,0$, $\bar{2}40$, 008, monitored every 60 reflections with random variation 2·0% over data collection, θ–2θ mode, scan width $(2{\cdot}0 + \Delta_{\alpha 1 \alpha 2})$, scan rate a function of count rate (10° min^{-1} minimum, 30° min^{-1} maximum), 1686 reflections measured, 1551 unique, $R_{int} = 0{\cdot}008$, 1230 observed $[F_o > 3\sigma(|F_o|)]$.

Structure solved by direct methods. Full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1{\cdot}0$, isotropic secondary extinction value = $2{\cdot}3 \times 10^{-6}$. 185 parameters refined: atom coordinates and anisotropic temperature factors for all non-H atoms, coordinates for H atoms. Isotropic temperature factors for H atoms set at $1{\cdot}1 \times U_{eq}$ of covalently bonded atoms. $(\Delta/\sigma)_{max} = 0{\cdot}004$, $R = 0{\cdot}029$, $wR = 0{\cdot}028$, $S = 0{\cdot}6$. Final difference Fourier excursions, 0·13 and −0·18 e Å^{-3}. All calculations performed with *SDP* system of programs (Enraf–Nonius, 1985).

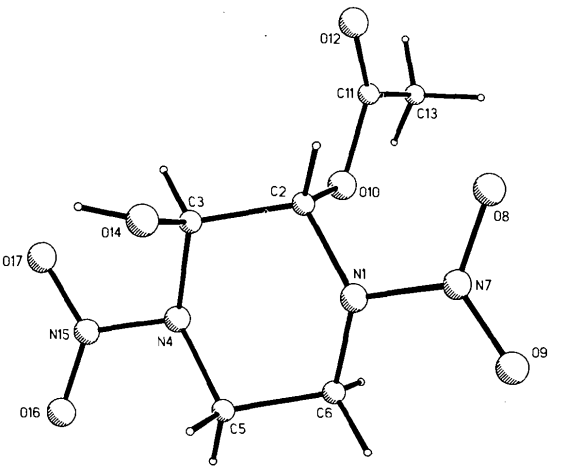

Fig. 1. A perspective drawing illustrating the results of the X-ray study on the title compound.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atom numbering for Tables 1 and 2, which report atom coordinates, bond distances and angles, follows that shown in Fig. 1.* The hydrogen-bond parameters are: H14···O8 =

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44086 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/87/102022-02$01.50

Table 1. *Positional parameters and equivalent isotropic thermal parameters with estimated standard deviations in parentheses*

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as $\frac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	B_{eq}(Å^2)
N1	0·1914 (1)	0·5908 (2)	0·1245 (2)	2·75 (4)
C2	0·2921 (2)	0·5032 (3)	0·1756 (2)	2·58 (4)
C3	0·2780 (2)	0·2971 (3)	0·1918 (2)	2·65 (5)
N4	0·2028 (1)	0·2696 (2)	0·2602 (2)	2·68 (4)
C5	0·1007 (2)	0·3583 (3)	0·2048 (3)	4·00 (6)
C6	0·1170 (2)	0·5613 (3)	0·1934 (2)	3·83 (5)
N7	0·1915 (1)	0·7648 (3)	0·0732 (2)	3·39 (4)
O8	0·2713 (1)	0·8161 (2)	0·0545 (2)	4·28 (4)
O9	0·1110 (1)	0·8544 (2)	0·0469 (2)	4·94 (5)
O10	0·3471 (1)	0·5819 (2)	0·3019 (1)	2·86 (3)
C11	0·4481 (2)	0·6338 (3)	0·3239 (2)	3·04 (5)
O12	0·4937 (1)	0·5970 (3)	0·2507 (2)	5·08 (4)
C13	0·4899 (2)	0·7396 (4)	0·4488 (2)	3·84 (6)
O14	0·2431 (1)	0·2238 (2)	0·0645 (1)	3·77 (4)
N15	0·2000 (1)	0·0920 (3)	0·3056 (2)	3·15 (4)
O16	0·1201 (1)	0·0413 (2)	0·3227 (2)	4·31 (4)
O17	0·2796 (1)	0·0004 (2)	0·3293 (2)	4·53 (4)

Table 2. *Bond distances* (Å) *and angles* (°)

N1–C2	1·440 (2)	C5–C6	1·505 (3)
N1–C6	1·462 (3)	N7–O8	1·226 (3)
N1–N7	1·383 (3)	N7–O9	1·221 (2)
C2–C3	1·529 (3)	O10–C11	1·364 (2)
C2–O10	1·437 (2)	C11–O12	1·191 (3)
C3–N4	1·464 (3)	C11–C13	1·492 (3)
C3–O14	1·403 (2)	N15–O16	1·218 (3)
N4–C5	1·463 (3)	N15–O17	1·221 (2)
N4–N15	1·387 (3)		
C2–N1–C6	118·1 (2)	N4–C5–C6	108·7 (2)
C2–N1–N7	115·5 (2)	N1–C6–C5	109·4 (2)
C6–N1–N7	115·7 (2)	N1–N7–O8	117·8 (2)
N1–C2–C3	109·8 (2)	N1–N7–O9	117·4 (2)
N1–C2–O10	108·1 (2)	O8–N7–O9	124·9 (2)
C3–C2–O10	109·1 (2)	C2–O10–C11	116·5 (2)
C2–C3–N4	109·1 (2)	O10–C11–O12	122·7 (2)
C2–C3–O14	105·9 (2)	O10–C11–C13	110·7 (2)
N4–C3–O14	112·5 (2)	O12–C11–C13	126·6 (2)
C3–N4–C5	117·4 (2)	N4–N15–O16	117·9 (2)
C3–N4–N15	114·5 (2)	N4–N15–O17	117·0 (2)
C5–N4–N15	114·8 (2)	O16–N15–O17	125·0 (2)

2·14 (2), O14···O8 = 2·998 (4) Å and ∠O–H···O = 169·4 (1·1)°.

Related literature. For the structures of some other substituted 1,4-diazacyclohexanes see Sekido, Okamoto & Hirokawa (1985) and Ko Tien-Ming & Moncrief (1975) and references therein. The first article referenced above relates to the structure of 1,4-dinitrosopiperazine, which has a chair-shaped ring with essentially planar amino nitrogen atoms, in contrast to the pyramidal nitrogens in the title compound.

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Structure of *N*-(*N*-Chlorobenzimidoyl)benzamidinium Decachlorodiselenate(IV) Acetonitrile Solvate

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Abstract. $[C_{14}H_{13}ClN_3]_2[Se_2Cl_{10}].2CH_3CN$, M_r = 1112·0, triclinic, $P\bar{1}$, a = 10·457 (1), b = 10·860 (1), c = 11·762 (2) Å, α = 64·49 (1), β = 74·61 (1), γ = 74·52 (1)°, V = 1144 (1) Å^3, Z = 1, D_x = 1·61 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 23·4 cm^{-1}, $F(000)$ = 552, T = 293 K, R = 0·060 for 1767 unique observed reflections. The structure of the centrosymmetric decachlorodiselenate(IV) ion can be characterized as two $SeCl_6$ octahedra sharing an edge with average Se–Cl distances of 2·729, 2·209, and 2·382 Å for the

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