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

N1–C2	1·496 (3)	N1′–C2′	1·490 (3)
N1–C5	1·473 (3)	N1′–C5′	1·469 (3)
N1–C8	1·478 (3)	N1′–C8′	1·472 (3)
C2–C3	1·540 (3)	C2′–C3′	1·532 (3)
C2–C9	1·496 (3)	C2′–C9′	1·494 (3)
C2–C11	1·511 (3)	C2′–C11′	1·527 (3)
C3–C4	1·520 (4)	C3′–C4′	1·522 (4)
C4–C5	1·491 (3)	C4′–C5′	1·501 (3)
C5–O6	1·438 (3)	C5′–O6′	1·427 (3)
O6–C7	1·421 (3)	O6′–C7′	1·415 (3)
C7–C8	1·537 (3)	C7′–C8′	1·544 (3)
C8–C13	1·501 (3)	C8′–C13′	1·503 (3)
C9–N10	1·145 (4)	C9′–N10′	1·138 (4)
C11–C12	1·521 (4)	C11′–C12′	1·527 (3)
C13–C14	1·387 (3)	C13′–C14′	1·392 (3)
C13–C18	1·405 (3)	C13′–C18′	1·403 (3)
C14–C15	1·387 (4)	C14′–C15′	1·382 (4)
C15–C16	1·378 (4)	C15′–C16′	1·368 (4)
C16–C17	1·370 (4)	C16′–C17′	1·371 (4)
C17–C18	1·384 (4)	C17′–C18′	1·393 (4)
C2–N1–C5	108·2 (2)	C2′–N1′–C5′	107·8 (2)
C2–N1–C8	119·4 (2)	C2′–N1′–C8′	120·7 (2)
C5–N1–C8	107·4 (2)	C5′–N1′–C8′	107·1 (2)
N1–C2–C3	104·2 (2)	N1′–C2′–C3′	104·9 (2)
N1–C2–C9	104·7 (2)	N1′–C2′–C9′	105·6 (2)
N1–C2–C11	115·9 (2)	N1′–C2′–C11′	115·6 (2)
C3–C2–C9	108·1 (2)	C3′–C2′–C9′	108·6 (2)
C3–C2–C11	114·7 (2)	C3′–C2′–C11′	113·5 (2)
C9–C2–C11	108·5 (2)	C9′–C2′–C11′	108·2 (2)
C2–C3–C4	102·8 (2)	C2′–C3′–C4′	102·8 (2)
C3–C4–C5	103·5 (2)	C3′–C4′–C5′	102·8 (2)
N1–C5–C4	107·3 (2)	N1′–C5′–C4′	107·4 (2)
N1–C5–O6	105·1 (2)	N1′–C5′–O6′	105·7 (2)
C4–C5–O6	111·5 (2)	C4′–C5′–O6′	111·7 (2)
C5–O6–C7	104·3 (2)	C5′–O6′–C7′	104·0 (2)
O6–C7–C8	104·5 (2)	O6′–C7′–C8′	105·0 (2)
N1–C8–C7	102·7 (2)	N1′–C8′–C7′	102·4 (2)
N1–C8–C13	113·5 (2)	N1′–C8′–C13′	112·9 (2)
C7–C8–C13	113·7 (2)	C7′–C8′–C13′	114·2 (2)
C2–C9–N10	179·5 (3)	C2′–C9′–N10′	179·1 (3)
C2–C11–C12	113·3 (2)	C2′–C11′–C12′	113·2 (2)
C8–C13–C14	122·2 (2)	C8′–C13′–C14′	121·8 (2)
C8–C13–C18	120·0 (2)	C8′–C13′–C18′	120·5 (2)
C14–C13–C18	117·8 (2)	C14′–C13′–C18′	117·6 (2)
C13–C14–C15	121·3 (2)	C13′ C14′ C15′	121·2 (2)
C14–C15–C16	120·0 (3)	C14′–C15′–C16′	120·5 (3)
C15–C16–C17	119·7 (3)	C15′–C16′–C17′	119·6 (3)
C16–C17–C18	120·8 (3)	C16′–C17′–C18′	120·8 (3)
C13–C18–C17	120·4 (3)	C13′–C18′–C17′	120·2 (2)

envelope conformation with O(6) out of the plane of the four other atoms by 0·55 Å. In the crystal only normal van der Waals contacts are observed.

Table 3. *Comparison of selected torsion angles* (°) *of the two molecules*

C5–N1–C2–C3	14·9 (2)	14·1 (2)
N1–C2–C3–C4	−32·1 (2)	−32·1 (2)
C2–C3–C4–C5	37·4 (2)	37·6 (2)
C3–C4–C5–N1	−28·9 (2)	−29·9 (2)
C4–C5–N1–C2	8·6 (2)	9·9 (2)
N1–C5–O6–C7	−37·6 (2)	−38·2 (2)
C4–C5–O6–C7	−153·5 (3)	−154·7 (3)
C5–O6–C7–C8	40·0 (2)	39·0 (2)
O6–C7–C8–N1	−26·8 (2)	−24·9 (2)
C7–C8–N1–C5	4·0 (2)	1·7 (2)
C8–N1–C5–O6	19·9 (2)	21·9 (2)
N1–C8–C13–C14	22·7 (2)	24·6 (2)
N1–C8–C13–C18	−159·2 (3)	−155·8 (3)
C7–C8–C13–C14	139·5 (3)	141·1 (3)
C7–C8–C13–C18	−42·4 (2)	−39·4 (2)
C8–N1–C2–C9	138·4 (2)	136·1 (2)
C8–N1–C2–C11	18·9 (2)	16·5 (2)
C5–N1–C2–C11	142·0 (2)	139·9 (2)
N1–C2–C11–C12	169·7 (3)	172·9 (3)
C3–C2–C9–N10	9·8 (3)	5·3 (3)

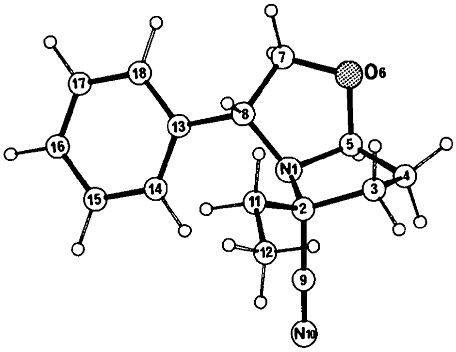

Fig. 1. Molecular structure and atomic numbering.

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1,6-Diazabicyclo[4.4.0]decane Hydrate

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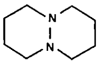
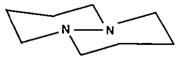
Abstract. $C_8H_{16}N_2.H_2O$, $M_r = 158{\cdot}25$, monoclinic, $C2/c$, $a = 12{\cdot}139\ (8)$, $b = 7{\cdot}106\ (4)$, $c = 11{\cdot}295\ (5)$ Å, $\beta = 105{\cdot}79\ (4)°$, $V = 937{\cdot}5\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}11$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}70$ cm^{-1}, $F(000) = 352$, $R = 0{\cdot}041$ for 592 unique observed reflections. The title compound adopts a *trans*-decalin conformation in the solid state. The molecules are linked together by water molecules in an extended hydrogen-bonded network in the direction of the z axis; the N(1)···O(1) distance is 3·019 (3) Å and the N(1)···H(1)–O(1) angle is 170 (2)°. The N(1)–N(1′) bond length, corrected for rigid-body motion, is 1·486 (3) Å.

0108-2701/88/101777-03$03.00

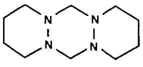
Introduction. The conformational analysis of cyclic tetraalkylhydrazine derivatives has received much attention over recent years (Nelsen, 1981; Shvo 1975, and references cited therein). Unconstrained tetraalkylhydrazines adopt conformations with the torsion angle between the lone pairs of *ca* 90° to minimize the destabilizing four-electron lone-pair/lone-pair interaction. In cyclic compounds, this conformation may not be attainable without introducing other unfavourable interactions and the observed structure is a compromise. It has been suggested that the title compound (1) exists in solution and the gas phase in the *trans*-decalin-like conformation (2). Assignment of this geometry, which has the nitrogen lone pairs *anti*, is supported by ^{13}C NMR (Nelsen & Clennan, 1978) and photoelectron spectroscopy (Nelsen & Buschek, 1974). The title compound is an oil, but it was found to produce a crystalline hydrate, from which single crystals were readily obtained by sublimation.



(1)



(2)



(3)

Experimental. Colourless block (0·7 × 0·6 × 0·3 mm) grown by slow sublimation at 273 K. Nicolet *P*3*m* diffractometer, 18 reflections centred ($10 < \theta < 15°$), graphite-monochromated Mo $K\alpha$ for data collection $2 < \theta < 25°$, room temperature, ω–2θ scans in 96 steps, ω-scan width $(1{\cdot}0 + 0{\cdot}35\tan\theta)°$, 971 data measured over 22 h with no detectable decay or movement; systematic absences ($hkl\ h + k = 2n + 1$, $h0l\ l = 2n + 1$). 830 unique data, $R_{\rm merge} = 0{\cdot}051$, 592 reflections with $F > 3\sigma(F)$ retained, used for structure solution, and refinement ($-14 < h < 14$, $0 < k < 9$, $0 < l < 14$), no absorption correction. Direct methods and difference Fourier methods, blocked-cascade least-squares refinement on F, $w = [\sigma^2(F) + 0{\cdot}0006(F^2)]^{-1}$, anisotropic temperature factors for all non-H atoms; methylene protons were refined in idealized positions (C—H 0·96 Å) all other atoms were refined without constraint. $R = 0{\cdot}0409$, $wR = 0{\cdot}0558$, $S = 1{\cdot}514$, data:variable ratio 10·6:1, max. peak and min. trough in final ΔF synthesis 0·12 and $-0{\cdot}14$ e Å^{-3} respectively, max. shift/e.s.d. ratio in final cycle 0·001; complex neutral scattering factors for C, H, N, O from *International Tables for X-ray Crystallography* (1974). Computer programs *SHELXTL* (Sheldrick, 1985).

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

	x	y	z	$U/U_{\rm eq}$
N(1)	327 (1)	836 (2)	5286 (1)	44 (1)*
C(1)	128 (2)	2330 (3)	4353 (2)	58 (1)*
C(2)	−1114 (2)	2890 (3)	3939 (2)	72 (1)*
C(3)	−1838 (2)	1201 (3)	3455 (2)	68 (1)*
C(4)	−1548 (1)	−350 (3)	4395 (2)	56 (1)*
O(1)	0	2905 (3)	7500	75 (1)*
H(1)	24 (22)	2250 (37)	6898 (22)	96 (8)

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

Table 2. *Selected bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and intermolecular contacts* (Å)

Uncorrected		Corrected	
N(1)—C(1)	1·469 (2)	N(1)—C(1)	1·479
N(1)—N(1′)	1·476 (3)	N(1)—N(1′)	1·486
N(1)—C(4′)	1·468 (2)	N(1)—C(4′)	1·478
C(1)—C(2)	1·506 (3)	C(1)—C(2)	1·511
C(2)—C(3)	1·500 (3)	C(2)—C(3)	1·510
C(3)—C(4)	1·504 (3)	C(3)—C(4)	1·514
O(1)—H(1)	0·83 (3)		
C(1)—N(1)—N(1′)	107·7 (1)	C(1)—N(1)—C(4′)	107·7 (1)
N(1′)—N(1)—C(4′)	107·7 (2)	N(1)—C(1)—C(2)	111·7 (2)
C(1)—C(2)—C(3)	109·8 (2)	C(2)—C(3)—C(4)	109·1 (2)
C(3)—C(4)—N(1′)	111·9 (2)	H(1)—O(1)—H(1″)	111·9 (35)
N(1′)—N(1)—C(1)—C(2)	62·5 (2)	N(1)—C(1)—C(2)—C(3)	−57·0 (2)
C(1)—C(2)—C(3)—C(4)	52·1 (2)	C(2)—C(3)—C(4)—N(1′)	−57·2 (2)
C(3)—C(4)—N(1′)—N(1)	63·1 (2)	C(4)—N(1′)—N(1)—C(1)	−64·1 (2)
C(3)—C(4)—N(1′)—C(1′)	179·0 (2)	C(4′)—N(1)—C(1)—C(2)	178·5 (2)
O(1)···N(1)	3·019 (3)	H(1)···N(1)	2·196 (26)

H(1″) generated from H(1) by the operation $-x,y,1{\cdot}5-z$.
Atoms with a ′ superscript are generated from the coordinate list by the operation $-x,-y,1-z$.

Discussion. Atomic coordinates for all non-constrained atoms are presented in Table 1,* selected bond lengths, bond angles, and torsion angles, and important intermolecular contacts in Table 2. A perspective view of the structure is presented in Fig. 1. The hydrazine molecule exists in the solid state in the *trans*-decalin conformation and has a crystallographic centre of inversion, the molecules being linked together by water molecules (which lie on crystallographic twofold axes) in an extended hydrogen-bonded chain along the z direction (see Fig. 2). The O(1)···N(1) distance is 3·019 (3) Å and the O(1)—H(1)···N(1) angle is 170·2 (20)°. This involvement of a neutral tetraalkylhydrazine in a hydrogen bond to water seems unusual, but has been previously observed in the structure of the tetraazacyclohexane (3) (Katritzky, Baker, Camalli, Spagna & Vaciago, 1980).

Librational analysis of rigid-body motion of the hydrazine molecule was carried out using the method of Schomaker & Trueblood (1968); this gave a generalized R index for the agreement of observed and calculated orthogonalized U_{ij} of 0·0247. Bond lengths corrected for this motion are presented in Table 2. The N(1)—N(1′) bond length corrected for rigid-body motion is 1·486 (3) Å and the average C—N—C angle is

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* 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 51111 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

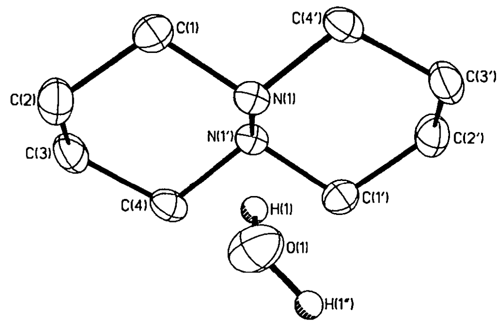

Fig. 1. Molecular structure of (1).H_2O with methylene-group hydrogens omitted for clarity. Non-hydrogen atoms are drawn to enclose 50% probability density.

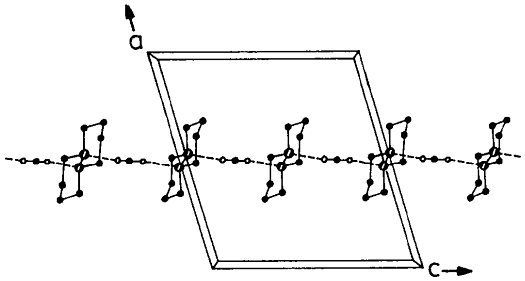

Fig. 2. Part of the crystal structure of (1).H_2O viewed down the *b* axis and showing the hydrogen-bonded chain ···(1)··· H_2O···(1)··· along the *c* axis.

107·7 (1)°, which compares favourably with other tetraalkylhydrazine structures having the N lone pairs *anti* which have N—N distances in the range 1·47–1·51 Å (Nelsen, Hollinsed & Calabrese, 1977; Nelsen, Hollinsed & Kessel, 1978; Katritzky *et al.*, 1980). In hydrazine structures having the N lone pairs in an approximately *gauche* conformation, the N—N distances are shorter, in the range 1·44–1·46 Å; they also tend to have larger C—N—C bond angles (Nelsen, Hollinsed & Calabrese, 1977; Spagna & Vaciago, 1978; Katritzky *et al.*, 1980). This difference in the N—N bond lengths between *anti* or *syn* and *gauche* hydrazines has been discussed in detail elsewhere (Nelsen, Hollinsed & Calabrese, 1977; Nelsen, 1981, 1986).

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Structure of Histaminium Dinitrate*

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(*Received* 13 *November* 1986; *accepted* 18 *May* 1988)

Abstract. $C_5H_{11}N_3^{2+}.2NO_3^-$, $M_r = 237{\cdot}17$, monoclinic, $P2_1/c$, $a = 7{\cdot}083$ (2), $b = 11{\cdot}775$ (3), $c = 12{\cdot}930$ (2) Å, $\beta = 105{\cdot}13$ (2)°, $V = 1041{\cdot}0$ (4) $Å^3$, $Z = 4$, $D_m = 1{\cdot}52$ (1), $D_x = 1{\cdot}513$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}094$ mm^{-1}, $F(000) = 496$, room temperature, $R = 0{\cdot}058$ for 829 observed reflections.

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* 4-(2-Ammonioethyl)imidazolium dinitrate.

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