7,8,9,10-Tetrahydro-6,10-propano-6*H*-cyclohepta[*b*]quinoxaline

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Abstract. $C_{16}H_{18}N_2$, $M=238\cdot2$. Orthorhombic, $P2_12_12_1$, $a=7\cdot01$ (1), $b=7\cdot22$ (1), $c=25\cdot61$ (2) Å from precession photographs, refined by diffractometer. $Z=4$, $D_m=1\cdot23$ (flotation), $D_c=1\cdot22$ g cm^{-3}. The compound was recrystallized from light petroleum as thick needles. The crystal structure, refined to $R=0\cdot055$, shows that the molecule has the boat-chair conformation with some bond angles considerably increased from those in acyclic compounds.

Introduction. Systematic absences $h00$, h odd, $0k0$, k odd and $00l$, l odd gave the space group $P2_12_12_1$. Data were collected for layers $h0l$ to $h5l$ and $0kl$ to $5kl$ with a Hilger–Watts linear diffractometer (Mo $K\alpha$ radiation). After layer scaling and merging, the data consisted of 1124 reflexions with $I>3\sigma$. Lorentz and polarization corrections were applied (but none for absorption) and the data were put on an absolute scale by the Wilson-plot method. The structure was solved by direct methods with *MULTAN* and refined by the full-matrix least-squares technique. Hydrogen atoms were located by difference synthesis and anisotropic thermal parameters were introduced for all atoms. Those for the hydrogen atoms were not refined but derived by multiplying β_{ij} for the attached carbon atom by $1\cdot3$. The weighting scheme used was $w=1/(1+F^2/3600)$ and

Table 1. *Atomic positions and anisotropic temperature factors* $(\times 10^4)$

The expression used for the anisotropic temperature factor was $\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right]$.

|	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N(1)	−138 (5)	7337 (4)	1673 (1)	151 (8)	172 (6)	10 (0)	0 (7)	−8 (1)	−1 (1)
N(2)	−3734 (5)	6810 (4)	1199 (1)	114 (8)	172 (6)	12 (0)	−5 (7)	−2 (1)	−7 (1)
C(1)	1332 (7)	5033 (6)	1125 (1)	159 (11)	169 (8)	13 (0)	25 (8)	−6 (2)	2 (2)
C(2)	1188 (9)	2937 (6)	1239 (1)	324 (16)	180 (9)	14 (1)	55 (10)	−1 (2)	12 (2)
C(3)	−92 (9)	1827 (6)	869 (2)	312 (16)	136 (7)	17 (1)	34 (10)	9 (3)	5 (2)
C(4)	−2137 (8)	2400 (6)	850 (2)	235 (13)	149 (8)	20 (1)	−53 (9)	14 (3)	−7 (2)
C(5)	−2545 (7)	4393 (5)	646 (1)	144 (11)	159 (7)	15 (1)	−17 (8)	0 (2)	−13 (2)
C(6)	−1421 (7)	4866 (5)	136 (1)	169 (10)	167 (8)	11 (0)	13 (8)	−8 (2)	−9 (2)
C(7)	277 (7)	6139 (6)	193 (1)	218 (12)	152 (7)	11 (0)	−10 (9)	0 (2)	4 (2)
C(8)	1894 (6)	5461 (6)	549 (2)	115 (10)	192 (8)	15 (1)	1 (8)	−2 (2)	4 (2)
C(9)	−392 (6)	6100 (5)	1298 (1)	143 (10)	143 (7)	10 (0)	10 (7)	−4 (2)	4 (2)
C(10)	−2229 (6)	5829 (5)	1067 (1)	140 (9)	134 (7)	11 (1)	−10 (7)	2 (2)	0 (2)
C(11)	−1702 (6)	8337 (5)	1830 (1)	145 (10)	145 (7)	10 (1)	−8 (8)	1 (2)	0 (2)
C(12)	−1456 (8)	9669 (6)	2234 (1)	227 (13)	217 (10)	12 (1)	4 (10)	−7 (2)	−9 (2)
C(13)	−3013 (9)	10697 (7)	2388 (1)	299 (16)	212 (10)	15 (1)	18 (11)	3 (3)	−14 (2)
C(14)	−4800 (8)	10489 (6)	2142 (1)	232 (12)	207 (10)	14 (1)	38 (10)	13 (2)	−4 (2)
C(15)	−5035 (8)	9200 (6)	1751 (1)	221 (13)	174 (8)	13 (1)	22 (10)	6 (2)	−2 (2)
C(16)	−3475 (6)	8106 (5)	1598 (1)	150 (10)	137 (7)	11 (1)	−6 (7)	4 (2)	2 (2)
H(1)	2463 (104)	5569 (76)	1318 (19)	245	236	18	51	8	6
H(2)	590 (137)	2743 (84)	1581 (21)	514	255	18	78	1	17
H(3)	2697 (135)	2431 (90)	1259 (22)	514	255	18	78	1	17
H(4)	−189 (133)	338 (80)	968 (22)	439	186	27	67	10	10
H(5)	544 (129)	1788 (86)	496 (23)	439	186	27	67	10	10
H(6)	−2735 (121)	2338 (86)	1170 (24)	298	198	32	−62	19	−6
H(7)	−3091 (11)	1560 (84)	576 (24)	298	198	32	−62	19	−6
H(8)	−4021 (104)	4525 (78)	589 (19)	230	219	20	−30	1	−16
H(9)	−916 (100)	3645 (76)	−8 (19)	215	254	17	4	−8	−15
H(10)	−2489 (101)	5475 (83)	−99 (18)	215	254	17	4	−8	−15
H(11)	−220 (108)	7385 (79)	327 (19)	298	210	18	−29	0	9
H(12)	996 (107)	6365 (75)	−161 (21)	298	210	18	−29	0	9
H(13)	2994 (104)	6389 (84)	583 (19)	198	296	18	2	−7	13
H(14)	2374 (108)	4312 (91)	402 (19)	198	296	18	2	−7	13
H(15)	−241 (136)	9749 (86)	2414 (20)	339	317	17	9	−9	−16
H(16)	−2806 (137)	11789 (90)	2622 (21)	427	337	20	30	6	−16
H(17)	−5993 (117)	11292 (81)	2272 (20)	333	282	20	26	12	−2
H(18)	−6423 (121)	8963 (80)	1594 (20)	371	244	17	35	16	−4
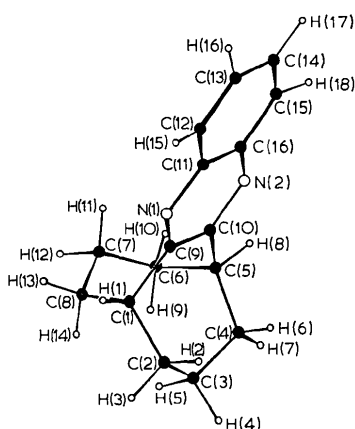
Fig. 1. The configuration of the molecule.

after ten cycles of refinement the final R was 0·055.* Scattering factors were taken from *International Tables for X-ray Crystallography* (1968), and the programs used were our adaptation of the CRYSTAL 69 system of Powell & Griffiths (1969). Atomic parameters are given in Table 1, the configuration of the molecule in Fig. 1, bond lengths in Table 2, bond angles in Table 3, and torsional angles in Table 4. Lengths and angles involving hydrogen atoms have not been tabulated.

Table 2. *Bond lengths* (Å) *(e.s.d.* = 0·005 Å)

N(1)–C(9)	1·323	C(5)—C(10)	1·513
N(1)–C(11)	1·373	C(6)—C(7)	1·511
N(2)–C(10)	1·315	C(7)—C(8)	1·535
N(2)–C(16)	1·397	C(9)—C(10)	1·431
C(1)–C(2)	1·545	C(11)–C(12)	1·424
C(1)–C(9)	1·500	C(11)–C(16)	1·388
C(2)–C(3)	1·531	C(12)–C(13)	1·377
C(3)–C(4)	1·493	C(13)–C(14)	1·409
C(4)–C(5)	1·558	C(14)–C(15)	1·378
C(5)–C(6)	1·563	C(15)–C(16)	1·406
C(1)–C(8)	1·558		

Table 3. *Bond angles* (°) *(e.s.d.* = 0·4°)

N(1)–C(9)—C(1)	116·9	C(4)—C(5)—C(6)	113·0
N(1)–C(9)—C(10)	120·9	C(4)—C(5)—C(10)	111·5
N(1)–C(11)–C(12)	118·1	C(5)—C(6)—C(7)	116·7
N(1)–C(11)–C(16)	121·8	C(5)—C(10)–C(9)	121·4
N(2)–C(10)–C(5)	115·8	C(6)—C(5)—C(10)	111·8
N(2)–C(10)–C(9)	122·8	C(6)—C(7)—C(8)	116·4
N(2)–C(16)–C(11)	120·7	C(8)—C(1)—C(9)	112·4
N(2)–C(16)–C(15)	118·7	C(9)—N(1)—C(11)	117·4
C(1)–C(2)—C(3)	115·7	C(10)–N(2)—C(16)	116·4
C(1)–C(8)—C(7)	116·1	C(11)–C(12)–C(13)	118·4
C(1)–C(9)—C(10)	122·2	C(11)–C(16)–C(15)	120·6
C(2)–C(1)—C(8)	112·9	C(12)–C(11)–C(16)	120·1
C(2)–C(1)—C(9)	113·3	C(12)–C(13)–C(14)	121·4
C(2)–C(3)—C(4)	116·0	C(13)–C(14)–C(15)	120·1
C(3)–C(4)—C(5)	116·3	C(14)–C(15)–C(16)	119·3

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30703 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Torsional angles in the cyclooctane ring* (°)
(e.s.d. = 0·5°)

C(1)–C(2)	– 52·7	C(5)–C(6)	– 104·7
C(2)–C(3)	– 60·0	C(6)–C(7)	60·5
C(3)–C(4)	63·3	C(7)–C(8)	– 58·2
C(4)–C(5)	46·3	C(8)–C(1)	104·4

Discussion. The conformation of the bicyclo[3,3,2]decane nucleus is of considerable interest. The evidence is not decisive between boat-chair (Russell & Keske, 1970) and twin-chair (Doyle & Parker, 1969; Doyle, Hafter & Parker, 1971) configurations, and empirical calculations (Engler, Chang & Schleyer, 1972) suggest that these may be of similar energy. In this case substituents of the nucleus might be important in determining the geometry and in particular it has been suggested by Doyle & Parker (1969) that the introduction of a rigid sp^2 system at C(9)–C(10) produces the boat-chair geometry. As bicyclo[3,3,2]dec-9-ene itself is very volatile, the title compound was synthesized to provide an analogue.

The molecule is in the expected boat-chair conformation, and has almost exact C_s symmetry. The only contact to neighbouring molecules less than 3·50 Å is between C(13) and N(1) at $\bar{x},\frac{1}{2}+y,\frac{1}{2}-z$. The bond lengths are all within the expected limits and the angles in the aromatic moiety are normal. The bridgehead atoms lie in the plane of this which therefore constrains the molecule to have mirror symmetry. As twisting is impossible, the molecule must relieve the strain in the bridged system by distortion of bond and torsional angles. Thus the average value for the methylene group bond angle is 116·2 (2)° and the angles at the bridgeheads are in the range 112·2 to 112·9°. The torsion angles also vary by up to 15° from those predicted from a strain-free model. The most obvious cause of the molecular distortion is the non-bonded repulsions between H(5) and both of H(9) and H(14). This is shown clearly in the dihedral angles C(2)–C(3)–C(4)/C(1)–C(2)–C(4)–C(5) and C(6)–C(7)–C(8)/C(1)–C(5)–C(6)–C(8) of 53·0 and 51·1° compared with about 66° estimated from models.

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