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The Crystal Structures of Azabicyclo Compounds. I. The Crystal and Molecular Structure of 1,2,4,4,5,8-Hexamethyl-8-*N*-acetamidobicyclo[3,3,1]-3-azanon-2-ene Hydrobromide (BRANA)

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The title compound (BRANA) crystallizes in the monoclinic system. The space group $P2_1/n$ has been chosen, $a=32{\cdot}385\ (7)$, $b=6{\cdot}526\ (1)$, $c=7{\cdot}778\ (2)$ Å, $\beta=88{\cdot}43\ (2)°$, $Z=4$. The structure was solved by the heavy-atom method using three-dimensional X-ray data. The refinement was carried out using the full-matrix least-squares method leading to a final R value of 0·049. The positions of the hydrogen atoms were located from a difference Fourier synthesis. Bond lengths and angles are discussed in detail.

Introduction

From the previously reported reaction of 2,3-dimethyl-1,3-butadiene with acetonitrile in the presence of sulphuric acid, Lora-Tamayo, García-Muñoz & Madroñero (1958), a solid basic compound was obtained to which was assigned a structure of substituted tetrahydropyridine. In the present work (consisting of two papers) a single-crystal X-ray diffraction study of both the hydrobromide ($C_{16}H_{28}N_2O.HBr$) (BRANA) and the hydrogenated derivative of this compound (ANA) is reported. This study led to a revised structural assignment of these compounds as bicyclo azanonane derivatives. In the first paper of this series, the structure of the BRANA compound is described. The work on ANA is presented in the next paper.

Experimental

A crystalline sample of BRANA was kindly provided by Professor García-Muñoz. The sample was recrystallized from absolute ethanol. Colourless crystals were obtained. A single crystal of $\sim 0{\cdot}3\times 0{\cdot}4\times 0{\cdot}2$ mm was selected for the X-ray investigation. The crystal belongs to the monoclinic system. The lattice constants were obtained by a least-squares analysis of 25 reflexions measured on a four-circle diffractometer with Mo $K\alpha$ radiation. The crystal data are listed in Table 1.

The intensities of 3301 unique reflexions were measured using graphite monochromatized Mo $K\alpha$ radiation

Table 1. *Crystal data for BRANA*

Standard deviations, given in parentheses, refer to the least significant digits.

Molecular formula	$C_{16}H_{28}N_2O.BrH$
Molecular weight	344·9
Melting point	232–233 °C
Space group	$P2_1/n$
$a=$ 32·385 (7) Å	$d_x=1{\cdot}348$ g cm^{-3}
$b=$ 6·526 (1)	$Z=4$
$c=$ 7·778 (2)	$F(000)=728$
$\beta=88{\cdot}430\ (2)°$	$V=1643{\cdot}23$ Å^3

Table 2. *The positional* ($\times 10^4$) *and thermal* ($\times 10^3$) *parameters for non-hydrogen atoms*

Standard deviations, given in parentheses, refer to the least significant digits. Thermal factors are those in the expression $\exp[-2\pi^2\sum U_{ij}a_i^*a_j^*h_ih_j]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	1060 (2)	606 (10)	4918 (8)	21 (3)	22 (3)	25 (3)	−2 (3)	−3 (3)	−2 (3)
C(2)	1132 (2)	2280 (11)	3601 (8)	26 (3)	31 (4)	24 (3)	−2 (3)	−1 (2)	−0 (3)
N(3)	1481 (1)	3230 (9)	3488 (6)	16 (3)	28 (3)	31 (3)	0 (2)	1 (2)	3 (3)
C(4)	1869 (2)	2757 (11)	4397 (8)	16 (3)	28 (4)	34 (3)	1 (3)	−1 (3)	−3 (3)
C(5)	1787 (2)	1211 (10)	5882 (8)	20 (3)	27 (3)	33 (3)	−0 (3)	−3 (3)	4 (3)
C(6)	1612 (2)	2206 (12)	7535 (8)	31 (4)	39 (4)	27 (3)	−6 (3)	−6 (3)	−5 (3)
C(7)	1180 (7)	3116 (13)	7345 (9)	32 (4)	38 (4)	28 (3)	−7 (4)	0 (3)	−4 (4)
C(8)	864 (2)	1622 (10)	6607 (8)	22 (3)	31 (4)	25 (3)	1 (3)	3 (3)	−0 (3)
C(9)	1479 (2)	−384 (10)	5302 (8)	21 (3)	25 (4)	33 (4)	−2 (3)	−3 (3)	−0 (3)
N(10)	785 (2)	−4 (9)	7900 (7)	25 (3)	41 (4)	27 (3)	−2 (3)	2 (2)	9 (3)
C(11)	435 (2)	−1098 (12)	8186 (8)	18 (3)	46 (4)	34 (4)	−7 (3)	3 (3)	5 (3)
O(12)	131 (1)	−967 (10)	7316 (7)	25 (3)	79 (4)	56 (3)	−12 (3)	−11 (2)	24 (3)
C(13)	439 (2)	−2525 (12)	9707 (9)	34 (4)	52 (6)	43 (4)	−6 (4)	3 (3)	12 (4)
C(14)	467 (2)	2846 (13)	6296 (9)	31 (4)	44 (4)	41 (4)	5 (4)	4 (3)	5 (4)
C(15)	774 (2)	−1060 (12)	4195 (9)	33 (4)	40 (4)	37 (4)	−9 (3)	0 (3)	−3 (4)
C(16)	815 (2)	2857 (14)	2323 (9)	32 (4)	59 (5)	37 (4)	−8 (4)	−12 (3)	18 (4)
C(17)	2148 (2)	1888 (13)	2931 (9)	23 (4)	51 (4)	48 (4)	5 (4)	9 (3)	−4 (4)
C(18)	2037 (2)	4817 (12)	4962 (9)	29 (4)	40 (4)	40 (4)	−2 (3)	−9 (3)	5 (3)
C(19)	2190 (2)	106 (12)	6351 (10)	29 (4)	33 (4)	59 (5)	−3 (3)	−13 (3)	7 (4)
Br(20)	1596 (2)	7067 (1)	566 (1)	41 (4)	46 (4)	36 (4)	−6 (4)	−1 (3)	4 (4)

(λ=0·7107 Å), on an automatic four-circle PW 1100 Philips diffractometer. Each reflexion was scanned in 11·5 s at a speed of 0·08 s^{-1}, the background was measured at both sides of the peak for 2 s. Every hour the intensity and location of three reference reflexions were checked. There was no significant variation during the measurement. Out of the 3301 reflexions 458 were unobserved, a reflexion being considered as unobserved when $I_o \leq 2\sigma(I_o)$ where $I_o = N - B_1 - B_2$, $\sigma(I_o) = \sqrt{(N + B_1 + B_2)}$, N=peak count and B_1, B_2 are the normalized background counts. No absorption correction was applied. The data were corrected for Lorentz and polarization effects.

Structure determination and refinement

Structure factor calculations based on the coordinates of the Br atom, obtained from a three-dimensional Patterson function, and isotropic temperature factor, gave an R index of 0·289. A subsequent Fourier map revealed twelve atoms. The remaining atoms of the structure appeared in an electron-density map calculated from the phases of the previous twelve atoms; with all non-hydrogen atoms the conventional reliability index R was 0·167. Atomic scattering factors and anomalous dispersion correction for the Br atom used throughout the refinement were taken from *International Tables for X-ray Crystallography* (1974). The refinement of the structure was carried out by the full-matrix least-squares program *CRYLSQ* of the X-RAY System. Three isotropic cycles for all non-hydrogen atoms and the observed reflexions, reduced the reliability index to 0·086. Another three cycles of full-matrix least-squares refinement with anisotropic temperature factors reduced R to 0·068. All hydrogen atoms were clearly revealed in a difference Fourier synthesis with reasonable bond lengths and bond angles. Two more cycles of least-squares refinement with isotropic temperature factors for the hydrogen atoms and varying anisotropic temperature factors for the rest were carried out. The thermal parameters of the hydrogen atoms have the same assignment as those of the atoms to which they are bonded. The final discrepancy for all observed reflexions was 0·049. Each sequence of the refinement was ended when all shifts of the parameters were less than one fifth of the corresponding standard

Table 3. *Positional* ($\times 10^3$) *and thermal parameters for hydrogen atoms*

Standard deviations, given in parentheses, refer to the least significant digits.

	x	y	z	B
H(3)	153 (2)	454 (13)	263 (10)	2·520 (1)
H(6,1)	163 (3)	116 (14)	850 (11)	3·316 (1)
H(6,2)	176 (2)	331 (13)	806 (1)	3·316 (1)
H(7,1)	124 (3)	446 (14)	668 (11)	3·251 (1)
H(7,2)	110 (2)	358 (13)	852 (11)	3·251 (1)
H(9,1)	146 (2)	846 (12)	616 (1)	2·723 (1)
H(9,2)	159 (2)	886 (12)	420 (1)	2·723 (1)
H(10)	100 (2)	970 (13)	843 (1)	3·054 (1)
H(13,1)	72 (2)	−319 (14)	996 (10)	4·139 (1)
H(13,2)	25 (2)	−174 (14)	68 (10)	4·139 (1)
H(13,3)	24 (2)	−379 (14)	957 (11)	4·139 (1)
H(14,1)	48 (3)	416 (14)	579 (11)	3·885 (1)
H(14,2)	24 (2)	207 (14)	558 (10)	3·885 (1)
H(14,3)	29 (2)	327 (16)	751 (10)	3·885 (1)
H(15,1)	74 (2)	774 (13)	502 (10)	3·714 (1)
H(15,2)	89 (2)	838 (13)	329 (11)	3·714 (1)
H(15,3)	45 (3)	−41 (13)	396 (10)	3·714 (1)
H(16,1)	49 (3)	276 (14)	286 (11)	4·315 (1)
H(16,2)	87 (3)	426 (14)	194 (11)	4·315 (1)
H(16,3)	83 (3)	187 (14)	135 (11)	4·315 (1)
H(17,1)	212 (2)	298 (14)	194 (10)	4·014 (1)
H(17,2)	202 (3)	45 (14)	252 (11)	4·014 (1)
H(17,3)	240 (3)	670 (14)	185 (10)	4·014 (1)
H(18,1)	224 (3)	459 (13)	544 (11)	3·603 (1)
H(18,2)	202 (2)	590 (13)	407 (10)	3·603 (1)
H(18,3)	183 (2)	558 (13)	592 (10)	3·603 (1)
H(19,1)	209 (2)	904 (14)	733 (11)	4·148 (1)
H(19,2)	236 (3)	115 (14)	660 (11)	4·148 (1)
H(19,3)	226 (3)	911 (14)	534 (11)	4·148 (1)

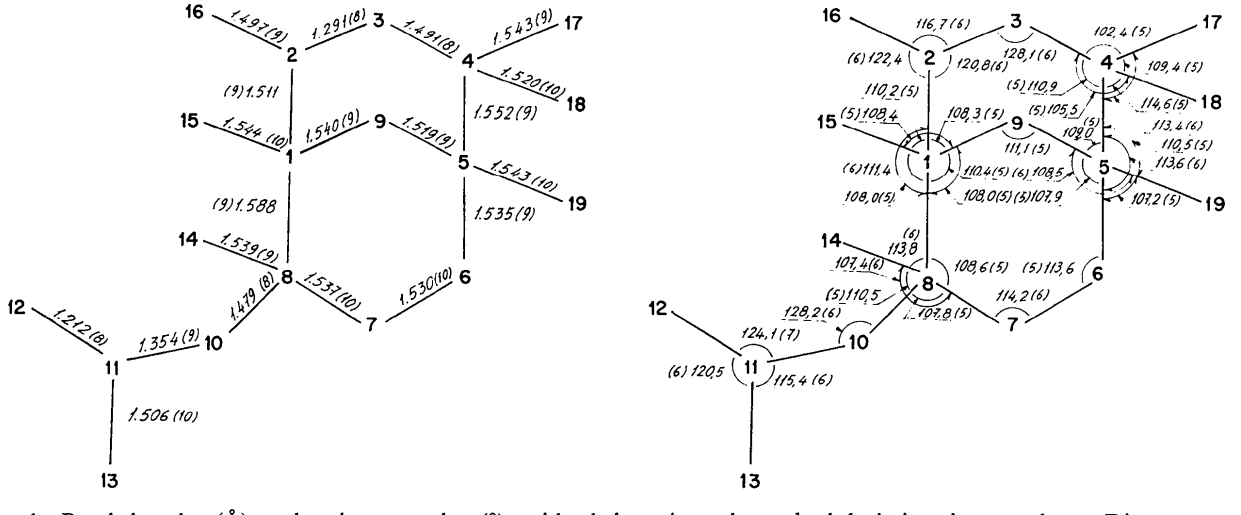

Fig. 1. Bond lengths (Å) and valency angles (°), with their estimated standard deviations in parentheses. Distances not shown on the diagram are N(3)–C(7)=3·130 (9), N(3)–C(9)=2·748 (9) and C(7)–C(9)=2·931 (9) Å.

Table 4. *Least-squares planes, torsion angles and dihedral angles* (°)

	Key to planes		Equations of the planes
1	C(1), C(2), C(4), C(5)	1	$0{\cdot}2876X-0{\cdot}7183Y-0{\cdot}6335Z+1{\cdot}7273=0$
2	C(1), C(5), C(6), C(8)	2	$-0{\cdot}0052X+0{\cdot}8916Y-0{\cdot}4572Z+1{\cdot}3969=0$
3	C(1), C(2), N(3), C(4), C(5)	3	$0{\cdot}2596X-0{\cdot}6980Y-0{\cdot}6549Z+1{\cdot}7859=0$
4	C(1), C(5), C(6), C(7), C(8), C(9)	4	$-0{\cdot}0909X-0{\cdot}7234Y+0{\cdot}6844Z-2{\cdot}2710=0$
5	C(1), C(2), N(3), C(4), C(5), C(9)	5	$0{\cdot}3004X-0{\cdot}5799Y-0{\cdot}7573Z+1{\cdot}8615=0$
6	C(8), N(10), C(11), O(12), C(13)	6	$0{\cdot}3559X-0{\cdot}7127Y-0{\cdot}6045Z+2{\cdot}7853=0$
7	C(8), N(10), C(14)	7	$0{\cdot}2560X+0{\cdot}6352Y+0{\cdot}7287Z-5{\cdot}1678=0$
8	C(4), C(17), C(18)	8	$0{\cdot}5804X-0{\cdot}4594Y+0{\cdot}6724Z-5{\cdot}0400=0$

Distances (Å) of atoms from the planes. Italicized values belong to atoms forming the planes.

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
C(1)	*0·0382*	*0·0001*	*0·0299*	*−0·2617*	*−0·2009*	1·4505	−1·2251	−0·5984
C(2)	*−0·0385*	1·4363	*−0·0022*	−1·7712	*0·0029*	1·3644	−1·2247	−1·6690
N(3)	−0·1035	2·0231	*−0·0505*	−2·3825	*0·0490*	1·3775	−0·6058	−1·3575
C(4)	*0·0375*	1·4212	*0·0716*	−1·7915	*0·0761*	1·6243	0·0398	*0·0000*
C(5)	*−0·0371*	*−0·0001*	*−0·0488*	*−0·2496*	*−0·2841*	1·5613	0·1802	1·1035
C(6)	−1·4710	*0·0001*	−1·4980	*0·2083*	−1·7939	0·1322	1·3937	1·3610
C(7)	−2·2068	0·6040	−2·2214	*−0·1953*	−2·4472	−0·7004	1·3036	0·1745
C(8)	−1·4422	*−0·0001*	−1·4664	*0·2119*	−1·7953	*−0·0289*	*0·0000*	−0·3671
C(9)	0·7052	−0·7186	0·6801	*0·2865*	*0·3571*	2·2157	−1·0678	0·6919
N(10)	−1·3822	−1·4003	−1·4499	1·6883	−1·9739	*0·0378*	*0·0000*	0·6640
C(11)	−1·3353	−2·1314	−1·4246	2·4595	−2·0678	*0·0109*	−0·5798	0·4866
O(12)	−1·2565	−1·7443	−1·3315	2·0264	−1·9063	*0·0022*	−1·2752	−0·5891
C(13)	−1·4015	−3·4973	−1·5349	3·9380	−2·4085	*−0·0230*	−0·2974	1·7374
C(14)	−2·2345	0·8283	−2·2398	−0·4140	−2·4281	−0·9120	*0·0000*	−1·6471
C(15)	0·9042	−0·7100	0·8840	0·2258	0·5722	2·2299	−2·5662	−1·0234
C(16)	0·0171	2·2278	0·0803	−2·6282	0·2202	1·3215	−1·9791	−3·1219
C(17)	1·4174	1·4276	1·4662	−2·2411	1·5299	3·0275	−0·9274	*0·0000*
C(18)	−1·0473	2·4182	−0·9941	−2·5137	−0·8699	0·5977	1·3562	*0·0000*
C(19)	0·6282	−0·8147	0·5968	0·4017	0·2532	2·3229	0·3244	2·4429

Dihedral angles

Planes	Angle	Planes	Angle	Planes	Angle	Planes	Angle
1–2	69·2	1–5	10·7	1–7	32·4	1–8	85·9
1–3	1·7	2–5	79·9	2–7	76·4	2–8	44·2
2–3	70·9	3–5	9·0	3–7	32·2	3–8	87·2
1–4	86·6	4–5	82·8	4–7	89·1	4–8	42·3
2–4	17·4	1–6	4·3	5–7	32·5	5–8	86·1
3–4	88·3	2–6	68·7	6–7	36·7	6–8	82·7
		3–6	4·8			7–8	69·7
		4–6	86·0				
		5–6	12·0				

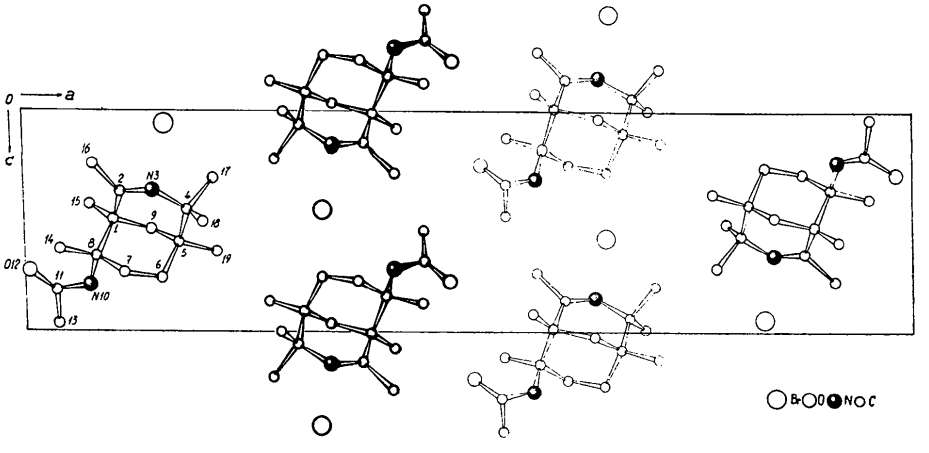

Fig. 2. Projection of the crystal structure along the *b* axis and the atom numbering.

deviations. Unit weight was assigned for each reflexion. The final positional and thermal parameters for the non-hydrogen atoms and for the hydrogen atoms are given in Tables 2 and 3 respectively.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31511 (13 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

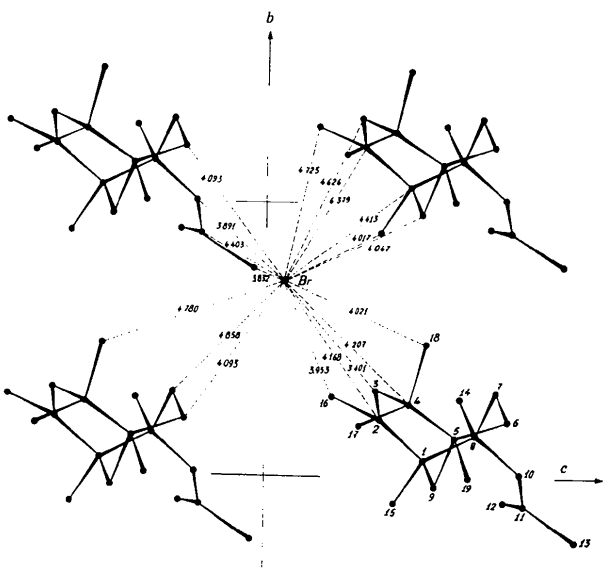

Fig. 3. Some distances involving the Br^- ion.

Discussion

The geometry of the bicyclo[3,3,1]nonane system presents interesting conformational problems and shows the possibility of severe non-bonded hydrogen–hydrogen interactions irrespective of the molecule conformation, *e.g.* twin-chair, twin-boat, boat–chair or twin-twist-boat (Brown, Eglinton, Martin, Parker & Sim, 1964). Accordingly, the C(3)···C(7) methylene interactions in the twin-chair conformation can be eliminated by the introduction into the bicyclononane skeleton of a double bond at C(3) or at C(3) and C(7). This is the case in the BRANA compound. In Fig. 1 the bond lengths and bond angles are shown.

In Table 4 the deviations of the atoms from some least-squares planes of the molecule and the dihedral and torsional angles are listed. The bond length N(3)–C(2) is 1·291 Å indicating the presence of a double bond between these atoms. Consequently the H atoms that could have some interaction with the *endo* H atom of the C(7) atom are not present. Hence, the conformation of the other cyclohexane ring of the bicyclo system resembles a slightly flattened chair. The distances of the atoms C(7) and C(9) to the plane through the atoms C(1), C(5), C(6), C(8) which are 0·604 and −0·719 Å, respectively, confirm this conformation. These are in fact close to the value of 0·728 Å calculated for these displacements in the ideal chair conformation (Brown, Martin & Sim, 1965). The N(3)···C(7) non-bonded separation is 3·13 Å.

The 110·8° angle between the plane through atoms C(1), C(5), C(6) and C(8) and the plane through C(1),

Table 5. *Bond lengths* (Å) *and valence angles* (°) *concerning the hydrogen atoms*

N(3)—H(3)	1·089	C(2)—N(3)—H(3)	121·2	C(1)—C(15)–H(153)	110·4
C(6)—H(6,1)	1·020	C(4)—N(3)—H(3)	110·7	H(151)–C(15)–H(152)	102·7
C(6)—H(6,2)	0·957	C(5)—C(6)—H(61)	107·7	H(151)–C(15)–H(153)	108·4
C(7)—H(7,1)	1·034	C(5)—C(6)—H(62)	120·3	H(152)–C(15)–H(153)	114·4
C(7)—H(7,2)	0·991	C(7)—C(6)—H(61)	114·0	C(2)—C(16)–H(161)	113·2
C(9)—H(9,1)	1·003	C(7)—C(6)—H(62)	101·8	C(2)—C(16)–H(162)	108·6
C(9)—H(9,2)	1·045	H(61)—C(6)—H(62)	98·8	C(2)—C(16)–H(163)	108·9
N(10)–H(10)	0·847	C(6)—C(7)—H(71)	101·9	H(161)–C(16)–H(162)	108·7
C(13)–H(13,1)	1·009	C(6)—C(7)—H(72)	104·8	H(161)–C(16)–H(163)	105·4
C(13)–H(13,2)	1·083	C(8)—C(7)—H(71)	118·5	H(162)–C(16)–H(163)	112·0
C(13)–H(13,3)	1·056	C(8)—C(7)—H(72)	111·9	C(4)—C(17)–H(171)	103·0
C(14)–H(14,1)	0·944	H(71)—C(7)—H(72)	104·0	C(4)—C(17)—H(172)	108·9
C(14)–H(14,2)	1·071	C(8)—N(10)–H(10)	111·4	C(4)—C(17)–H(173)	117·9
C(14)–H(14,3)	1·125	C(11)—N(10)–H(10)	120·1	H(171)–C(17)–H(172)	108·8
C(15)–H(15,1)	1·017	C(11)—C(13)–H(131)	117·7	H(171)–C(17)–H(173)	112·5
C(15)–H(15,2)	0·872	C(11)—C(13)–H(132)	104·0	H(172)–C(17)–H(173)	105·6
C(15)–H(15,3)	1·158	C(11)—C(13)–H(133)	112·5	C(4)—C(18)–H(181)	106·5
C(16)–H(16,1)	1·131	H(131)–C(13)–H(132)	119·9	C(4)—C(18)–H(182)	113·8
C(16)–H(16,2)	0·976	H(131)–C(13)–H(133)	104·4	C(4)—C(18)–H(183)	112·0
C(16)–H(16,3)	0·994	H(132)–C(13)–H(133)	96·4	H(181)–C(18)–H(182)	121·7
C(17)–H(17,1)	1·059	C(8)—C(14)–H(141)	120·1	H(181)–C(18)–H(183)	105·7
C(17)–H(17,2)	1·073	C(8)—C(14)–H(142)	115·7	H(182)–C(18)–H(183)	96·4
C(17)–H(17,3)	0·824	C(8)—C(14)–H(143)	113·4	C(5)—C(19)–H(191)	103·3
C(18)–H(18,1)	0·786	H(141)–C(14)–H(142)	104·3	C(5)—C(19)–H(192)	103·0
C(18)–H(18,2)	0·991	H(141)–C(14)–H(143)	98·2	C(5)—C(19)–H(193)	106·0
C(18)–H(18,3)	1·106	H(142)–C(14)–H(143)	102·4	H(191)–C(19)–H(192)	120·6
C(19)–H(19,1)	1·073	C(1)—C(15)–H(151)	111·0	H(191)–C(19)–H(193)	100·9
C(19)–H(19,2)	0·902	C(1)—C(15)–H(152)	109·1	H(192)–C(19)–H(193)	121·1
C(19)–H(19,3)	1·044				

C(2), C(4) and C(5) is near to the tetrahedral value. The bond valency angles of 113·6° and 114·2° at C(6) and C(7) are a little greater than the tetrahedral value.

All the other distances and bond angles are normal and in good agreement with those obtained by Dobler & Dunitz (1964) for 3-azabicyclo[3,3,1]nonane hydrobromide. The bond lengths and angles of the hydrogen atoms are shown in Table 5.

The arrangement of the molecules in the crystal as seen in projection along the *b* axis is shown in Fig. 2.

The nearest contacts between the molecule and the unattached Br^- ion are shown in Fig 3.

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The Crystal Structures of Azabicyclo Compounds. II. The Crystal and Molecular Structure of 1,2,4,4,5,8-Hexamethyl-8-*N*-acetamidobicyclo[3,3,1]-3-azanonane (ANA)

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The title compound (ANA) crystallizes in the monoclinic space group $C2/c$ with $Z=8$. The cell dimensions are $a=19{\cdot}409$ (2), $b=18{\cdot}368$ (1), $c=10{\cdot}258$ (3) Å, $\beta=117{\cdot}478$ (1)°. The phasing model was obtained using direct methods and the refinement was carried out by full-matrix least-squares methods. The final R value was 0·075. The positions of the hydrogen atoms were located from a difference Fourier synthesis.

Introduction

The compound ANA is the hydrogenated derivative of the compound BRANA (described in part I). Therefore the double bond between the N(3) and C(2) atoms is eliminated. The resolution of this structure was undertaken in order to ascertain the new conformation of the bicyclo compound.

Experimental

A crystalline sample of ANA was provided by Professor García-Muñoz. Crystals, obtained by slow evaporation from a solution in absolute ethanol, were colourless, belonging to the monoclinic system. Accurate values for the cell parameters were determined by least-squares calculations on an automatic four-circle diffractometer with Mo $K\alpha$ radiation. The crystal data are given in Table 1.

Table 1. *Crystal data*

Standard deviations, given in parentheses, refer to the least significant digits.

Molecular formula	$C_{16}H_{30}N_2O$
Molecular weight	266·427
Space group	$C2/c$
$a=$ 19·409 (2) Å	$d_x=1{\cdot}074$ g cm^{-3}
$b=$ 18·368 (1)	$Z=8$
$c=$ 10·258 (3)	$F(000)=1168{\cdot}00$
$\beta=117{\cdot}478$ (1)°	$V=3244{\cdot}48$ Å^3

The θ–2θ scan mode of the Philips PW 1100 diffrac-

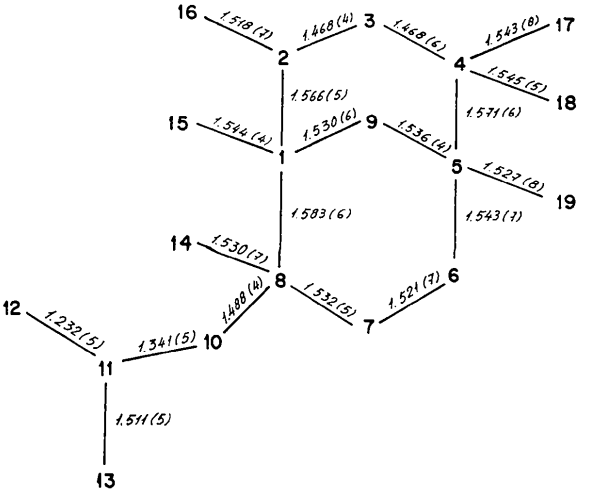

Fig. 1. Bond lengths (Å) with their estimated standard deviations in parentheses. Distances not shown on the diagram are N(3)–C(7) = 2·880, N(3)–C(9) = 2·542 and C(7)–C(9) = 2·920 Å.