Kálmán, Czugler & Argay (1981). The orientation of both tosyl substituents, with respect to the heterocyclic ring, is the same for molecules 1 and 2. A pronounced tendency to form pyramidal bonds is observed for N4 and N7 in both molecules; the sums of the bond angles about N fall in the range 348·1–353·5°.

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Structural Studies of Some Benzodiazepines. II. Structure of 3-(Isopropylamino)-4,5-dihydro-1*H*-2,4-benzodiazepine Hydroiodide (2)

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Abstract. $C_{12}H_{19}N_3^+.I^-$, $M_r = 332{\cdot}207$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}235\,(3)$, $b = 14{\cdot}671\,(3)$, $c = 7{\cdot}723\,(2)$ Å, $V = 1386{\cdot}28\,(57)$ Å^3, $Z = 4$, $D_x = 1{\cdot}5917\,(7)$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 181{\cdot}8$ cm^{-1}, $F(000) = 664$, diffractometer data at room temperature, $R = 5{\cdot}56\%$ for 2672 reflections with $I > 3\sigma(I)$. The benzodiazepine ring is in an envelope-type conformation, all N atoms are protonated and form hydrogen bonds with an I^- ion.

Introduction. This work is the second part in a series of crystal structure investigations of some benzodiazepine derivatives. The structure of 3-(benzylamino)-4,5-dihydro-1*H*-2,4-benzodiazepine hydrochloride (1) was reported earlier (Brzozowski, Stępień, Brzezińska, Glinka & Bavoux, 1989). Pharmacological experiments (E. Brzezińska & R. Glinka, unpublished results) have shown that (1) and (2) exhibit a depressive action on the central nervous system, reflected by the depression of the spontaneous locomotor activity. Both compounds also depressed the apomorphine-induced excitation and stereotypy in rats and induced hypothermia in mice. Thus, these compounds possess neuroleptic activity, which, however, is not very strong. (1) and (2) are weakly bound to the D_1 and D_2 receptors. Compound (1) also shows analgesic activity.

Experimental. Colourless crystals, equidimensional in habit (~0·6 mm), were obtained from ethanol at room temperature; the specimen used for the X-ray work, of size 0·2 × 0·2 × 0·2 mm, was cut from a larger crystal. Preliminary cell parameters were obtained from Weissenberg photographs. Diffraction data measured at room temperature on a CAD-4 diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. Lattice parameters determined by least squares using setting angles of 25 reflections with $\theta_{\max} = 50{\cdot}0°$. 3142 reflections (1/4 Ewald sphere)

Table 1. *Final positional and thermal parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum\sum U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}(Å^2)
I1	3048 (1)	1497 (0)	3460 (1)	266 (2)
N2	87 (8)	2453 (6)	−560 (13)	290 (29)
N1	891 (8)	2988 (5)	1946 (11)	226 (22)
C2	181 (7)	3098 (6)	680 (13)	173 (26)
N3	−483 (7)	3813 (6)	520 (12)	285 (26)
C4	−598 (7)	4590 (6)	1712 (17)	277 (31)
C5	468 (7)	5063 (6)	2057 (12)	181 (25)
C6	1293 (8)	4568 (6)	2888 (12)	179 (24)
C7	1072 (8)	3610 (6)	3405 (14)	249 (26)
C8	641 (9)	5969 (6)	1585 (16)	270 (30)
C9	1630 (9)	6387 (8)	1989 (13)	336 (31)
C10	2439 (10)	5906 (8)	2777 (16)	334 (34)
C11	2278 (8)	5001 (1)	3246 (15)	280 (54)
C12	628 (8)	1573 (6)	−652 (14)	203 (27)
C13	−158 (16)	899 (10)	−1477 (25)	415 (49)
C14	1698 (10)	1628 (9)	−1575 (21)	458 (40)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

N2—C2	1·351 (13)	N2—C12	1·453 (13)
N1—C2	1·318 (13)	N1—C7	1·467 (13)
C2—N3	1·332 (12)	N3—C4	1·472 (14)
C4—C5	1·501 (12)	C5—C6	1·399 (13)
C5—C8	1·394 (13)	C6—C7	1·486 (13)
C6—C11	1·390 (14)	C8—C9	1·392 (15)
C9—C10	1·359 (16)	C10—C11	1·390 (16)
C12—C13	1·519 (20)	C12—C14	1·493 (17)
C2—N2—C12	128·2 (9)	C2—N1—C7	126·4 (8)
N2—C2—N1	119·8 (8)	N1—C2—N3	124·6 (9)
N2—C2—N3	115·7 (9)	C2—N3—C4	127·6 (9)
N3—C4—C5	112·7 (8)	C4—C5—C8	121·7 (8)
C4—C5—C6	117·9 (8)	C6—C5—C8	120·3 (8)
C5—C6—C11	118·7 (8)	C5—C6—C7	118·9 (8)
C7—C6—C11	122·5 (9)	N1—C7—C6	114·2 (8)
C5—C8—C9	119·7 (9)	C8—C9—C10	120·3 (1·0)
C9—C10—C11	120·6 (1·1)	C6—C11—C10	120·5 (1·0)
N2—C12—C14	112·0 (9)	N2—C12—C13	108·1 (1·0)
C13—C12—C14	113·0 (1·1)		
C2—N2—C12—C13	145·4 (1·2)	C2—N2—C12—C14	−89·5 (1·3)
C12—N2—C2—N1	5·0 (1·6)	C12—N2—C2—N3	−174·4 (1·0)
C7—N1—C2—N2	−178·4 (9)	N2—C2—N3—C4	176·8 (9)
C2—N1—C7—C6	−53·4 (1·3)	C2—N3—C4—C5	55·2 (1·3)
C7—N1—C2—N3	1·0 (1·6)	N1—C2—N3—C4	−2·6 (1·6)
C4—C5—C8—C9	177·7 (9)	C7—C6—C11—C10	−178·3 (1·0)

measured with an ω–2θ scan; ranges of *hkl*: −14→14, 0→17, 0→9 respectively, $2\theta_{max} = 154°$. Standard reflections: $\bar{2}60$, $\bar{7}8\bar{4}$, $\bar{2}5\bar{6}$; count variations less than 2·5%. 2672 independent reflections with $I > 3\sigma(I)$ accepted as observed. The structure was solved by the Patterson method (localization of I^- ions) and analysis of Fourier maps (*SHELX76*; Sheldrick, 1976) on which all light atoms were identified. Atomic scattering factors (except for I) from *SHELX76*, for the I^- ion from *International Tables for X-ray Crystallography* (1974, Vol. IV). Refinement was carried out by full-matrix least squares using *F* magnitudes. 217 parameters, unit weights. H atoms located on a ΔF map and included in the refinement with isotropic temperature factors. Final $R = 5·56\%$, $S = 2·97$, $(\Delta/\sigma)_{max} = 0·698$. Largest final minimum and maximum $\Delta\rho$ peaks on difference Fourier map, all near the I^- ion, are −4·83 and 1·918 e Å^{-3}, and are caused by the influence of the high anomalous scattering of I and by the cut off of the Fourier series. The absolute conformation of (2) was also determined by the comparison of the refinement of both configurations. The final *R* factor for the configuration opposite to that described in this paper was 8·19%. Torsion angles and best planes were calculated by the *PARST* program from the *CRYSRULER* package (Rizzoli, Sangermano, Calestani, Andreetti, 1976). Final atomic parameters are given in Table 1 and bond lengths, bond angles and selected torsion angles are in Table 2.*

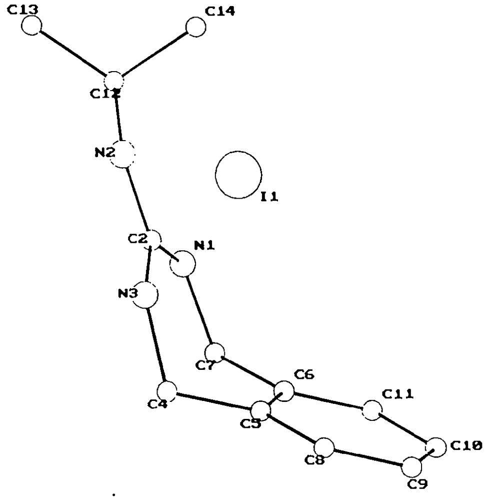

Fig. 1. A view of a molecule of the title compound (2) with the atomic numbering scheme.

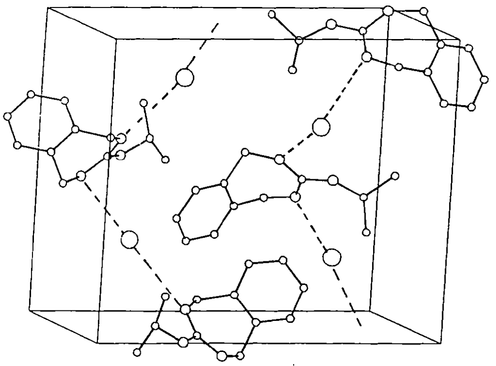

Fig. 2. A view of the molecular packing of (2) in the unit cell (hydrogen bonds as dotted lines).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and bond angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54344 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The benzodiazepine ring of (2) possesses an envelope-type conformation (see Fig. 1), the same as its benzylamino derivative (1). The angle between planes C4—C5—C6—C7—C8—C9—C10—C11 (plane 1) and C4—C7—N3—N1—C2 (plane 2) is 124·5 (3)° [124·7 (1)° in (1)]. The displacements of atoms from plane 1 [highest value for C8: −0·02 (1) Å] and from plane 2 [highest value for N3 0·012 (9) Å] are lower than in (1) [0·131 (7) for C7 and 0·116 (10) Å for N2]. The differences between torsion angles C2—N1—C7—C6 = −53·4 (1·3) and C2—N3—C4—C5 = 55·2 (1·3)° are also less in (2) than in (1) [−48·8 (1·0) and 65·8 (1·0)° respectively]. This indicates smaller distortions of the benzodiazepine ring in compound (2) than in compound (1). Both diazepine N atoms are protonated. The bonds N2—C2 = 1·351 (13), N1—C2 = 1·318 (13) and N3—C2 = 1·332 (12) Å are shorter than normal single C—N bonds [*e.g.* N2—C12 = 1·453 (13) Å], which is characteristic of the ionic form of the benzodiazepine group, occurring also in crystals of (1). The substitution of a Cl^- ion in (1) by an I^- ion in (2) did not change the motif of hydrogen bonds (see Fig. 2); the ionic form of (2) is stabilized by interactions N2···I1 with a distance of 3·689 (10), N(3)···I1 of 3·570 (9) and N1′*···I1 of 3·622 (9) Å (N1′* means the atom related to N1 by $\frac{1}{2}+x, \frac{1}{2}-y, -z$).

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Tetrabenzo[*b*,*g*,*k*,*p*][1,10,4,6,13,15]dioxatetraazacyclooctadeca[2,4,5,7,11,13,14,16]-octaene (III)*

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Abstract. $C_{28}H_{20}N_4O_2$, $M_r = 444·49$, monoclinic, $C2/c$, $a = 19·532$ (3), $b = 7·858$ (1), $c = 14·997$ (5) Å, $\beta = 98·91$ (2)°, $V = 2274$ (6) Å^3, $Z = 4$, $D_m = 1·294$, $D_x = 1·298$ (1) g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 5·91 cm^{-1}, $F(000) = 928$, room temperature, R = 0·0377 for 1897 reflections with $I \geq 3\sigma(I)$. The molecule contains an 18-membered heterocyclic ring which adopts a 'saddle'-like conformation with two non-linear diimino systems. A crystallographic twofold axis passes through the centre of the ring. Two of the four benzene rings are nearly parallel to each other.

Introduction. Many examples of the biological activity of carbodiimides are known, *e.g.* their bacteriostatic and anticancer properties (Roberts, Rounds & Shankman, 1961), and its application to immunological research (Goodfriend, Levine & Fasman, 1964). At present, however, cyclic carbo-

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* Dedicated to Professor Reginald Gruehn (Justus-Liebig-Universitat-Giessen, Germany) on the occasion of his 60th birthday.

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