

squares planes of rings *A* to *G*, and the dihedral angles between them, also attest to the localization of strain at the center of the molecule. The largest deviation from the least-squares plane through the atoms of ring *A* is 0.013 Å. The largest deviation from ring *B* is 0.050 Å and increases to 0.124 Å for ring *C* and 0.142 Å for ring *D*. The dihedral angles between the planes of consecutive rings *A*, *B*, *C*, *D* are 6.8, 14.1, and 17.4° respectively.

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Dibenzepin Hydrochloride: 10-[2-(Dimethylamino)ethyl]-5,10-dihydro-5-methyl-11*H*-dibenzo[*b,e*]1,4]diazepin-11-one Hydrochloride

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Abstract. C₁₈H₂₂N₃O⁺.Cl⁻, *M_r* = 331.85, triclinic, $\bar{P}1$, *a* = 11.516 (1), *b* = 10.668 (1), *c* = 7.441 (1) Å, α = 106.34 (1), β = 91.62 (1), γ = 96.06 (1)°, *U* = 870.6 (3) Å³, *Z* = 2, *D_x* = 1.27 Mg m⁻³, μ (Cu *K*α) = 2.013 mm⁻¹. Final *R* = 0.047 for 2335 independent reflexions. The molecules are held together through salt bridges of type N⁺—H...Cl⁻.

Introduction. From this study of the crystal structure of dibenzepin hydrochloride and studies of other anti-

depressant drugs in progress, we have accumulated information about their conformations and charge distributions from which conclusions may be drawn about structure–activity relations.

A crystal sample of dibenzepin hydrochloride was kindly provided by Sandoz Laboratories (Madrid). A crystal ~0.3 × 0.3 × 0.4 mm was selected for X-ray investigation. The intensities of all 2930 unique reflexions with 2 < θ < 65° were measured at 295 K with monochromatic Cu *K*α radiation on a Philips PW1100 diffractometer. The ω -2 θ scan technique was used. There was no appreciable change in periodically

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monitored standard reflexions. No correction for absorption was applied and 2335 data were accepted on the criterion $I > 2\sigma(I)$. Structure amplitudes were obtained after Lorentz and polarization corrections.

10 of the 21 non-H atoms were located on an *E* map with the phases of 499 *E*'s ≥ 1.349 calculated by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), the remaining non-H atoms being located from a difference synthesis. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). The H atoms were set at their calculated positions. The structure was refined anisotropically by least squares. To prevent bias on $w\Delta^2 F$ vs F_o or $\sin \theta/\lambda$, weights were assigned as $w = w_1 w_2$ where $w_1 = 1/\sigma_1^2$ and $w_2 = 1/\sigma_2^2$ with $\sigma_1 = a + b|F_o|$ and $\sigma_2 = c + d \sin \theta/\lambda$ with the coefficients given in Table 1.

Final refinement with an isotropic fixed contribution for H atoms gave $R = 0.047$ and $R_w = 0.055$.

Table 1. Coefficients for weighting scheme (Martínez-Ripoll & Cano, 1975)

	<i>a</i>	<i>b</i>
$F_o \leq 5.7$	0.37	0.0
$F_o > 5.7$	0.19	0.03
	<i>c</i>	<i>d</i>
$\sin \theta/\lambda \leq 0.40 \text{ \AA}^{-1}$	4.15	-8.48
$0.40 < \sin \theta/\lambda \leq 0.54$	1.00	-6.20
$\sin \theta/\lambda > 0.54$	-3.09	7.02

Table 2. Atomic coordinates ($\times 10^5$) and equivalent isotropic thermal parameters ($\times 10^4$) with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

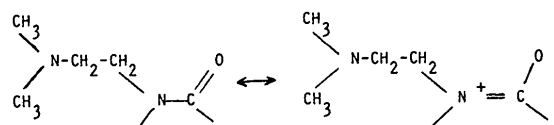
	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq} (\text{\AA}^2)$
Cl(1)	-30953 (7)	-43901 (6)	-12696 (9)	720 (3)
C(1)	-80197 (20)	13497 (21)	22085 (29)	467 (7)
C(2)	-91264 (23)	16963 (25)	26019 (35)	586 (9)
C(3)	-100131 (23)	7480 (30)	26701 (36)	624 (10)
C(4)	-98198 (19)	-5603 (26)	22759 (34)	534 (8)
N(5)	-85004 (14)	-22787 (17)	13171 (24)	409 (6)
C(6)	-87558 (18)	-37146 (20)	-19592 (33)	464 (7)
C(7)	-83715 (21)	-40571 (22)	-37548 (33)	521 (8)
C(8)	-73528 (21)	-34350 (24)	-41781 (31)	531 (8)
C(9)	-66871 (19)	-24868 (22)	-27642 (29)	453 (7)
N(10)	-62596 (13)	-12968 (15)	5231 (21)	350 (5)
C(11)	-65596 (16)	-2188 (19)	17961 (25)	351 (6)
C(12)	-78061 (16)	415 (19)	18334 (24)	360 (6)
C(13)	-87209 (17)	-9384 (20)	18051 (25)	372 (6)
C(14)	-81060 (16)	-27299 (18)	-5188 (27)	363 (6)
C(15)	-70397 (16)	-21648 (18)	-9348 (26)	349 (6)
O(16)	-58257 (13)	5348 (15)	29309 (21)	470 (5)
C(17)	-50727 (16)	-16497 (20)	7334 (27)	391 (6)
C(18)	-50623 (16)	-23787 (20)	22140 (28)	395 (6)
N(19)	-39245 (15)	-28980 (18)	23879 (24)	445 (6)
C(20)	-40059 (27)	-37303 (31)	36928 (44)	698 (11)
C(21)	-29320 (20)	-18368 (31)	29863 (35)	610 (9)
C(22)	-93328 (22)	-31988 (26)	19011 (38)	584 (9)

Table 2 shows the final atomic parameters of the non-H atoms.*

Discussion. The crystal structure is shown in projection along **b** in Fig. 1. The molecule is involved in a salt bridge as a result of proton transfer from HCl to N(19) forming the hydrogen-bonded ion pair $N^+H \cdots Cl^-$ (Table 3).

The geometrical features of the molecule are shown in Fig. 2. The phenyl rings are normal. The central ring has a boat conformation: C(12), C(13), C(14) and C(15) lie in a plane, C(11), N(10) and N(5) being 0.822 (4), 0.840 (3) and 0.615 (4) Å out of it respectively. O(16) and C(22) are also on the same side of the plane, 1.493 (3) and 1.026 (4) Å out of it.

The distance C(11)-O(16) = 1.228 (2) Å vs the normal value 1.19 Å and C(11)-N(10) = 1.352 (2) vs N-C(*sp*²) ~ 1.42 Å indicate that the compound may be represented by some polar canonical formula which implies a lower charge on N(10) and a partial anionic character of O(16).



* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35942 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

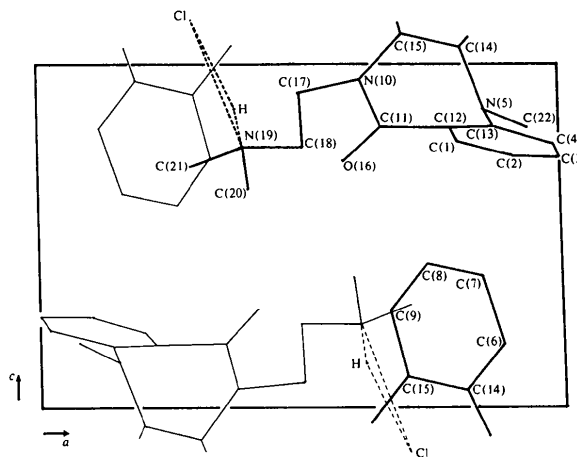


Fig. 1. The crystal structure of dibenzepin hydrochloride as seen along **b**.

Table 3. The geometry of the hydrogen bond A-H...B with e.s.d.'s in parentheses

A-H...B	A-H	H...B	A...B	AHB
N(19)-H(N19)...Cl	0.92 (5) Å	2.06 (5) Å	2.980 (2) Å	171 (4)°

