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Structure at 198 K of a Chiral Tricyclic Aminochloride, $C_{10}H_{17}N.0.5HCl$

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Abstract. [3aR-(3aα,4aβ,7aβ,7bα)]-Decahydro-1H-dicyclopenta[b,d]pyrrole 0.5-hydrochloride, $C_{10}H_{17}N\cdot0.5HCl$, $M_r = 169.5$, trigonal, $R32$, $a = 17.320$ (2), $c = 16.963$ (2) Å, $V = 4406.8$ (8) Å^3, $Z = 18$, $D_x = 1.149$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.96$ cm^{-1}, $F(000) = 1674$, $T = 198$ K, $R = 0.0462$ for 1589 reflections [$I \geq 3\sigma(I)$]. Crystal chirality was assigned to correspond to the known chirality of the parent amine and confirmed by refining the η value [chirality/polarity, 1.2 (2)]. The three five-membered rings (A, B and C) exhibit envelope conformations which appear to be preferred over possible twist conformations. Several closely related compounds exhibit similar envelope conformations. Atoms C(3), C(10) and C(7) (IUPAC numbering: C2, C4a and C7, respectively) occupy the flap position in rings A, B and C, respectively. All bond lengths and angles appear to be normal except for a slight elongation of bonds around the trisubstituted ring-junction atoms C(1), C(5), C(6) and C(10). The lengthening of these bonds, relative to those around the disubstituted ring atoms, has been seen in related compounds. Two kinds of intermolecular interactions are observed in the crystal structure. H(1b) (with 0.5 occupancy at the N atom) links cations related by a twofold axis in the ab planes to form dimers [N(1)—H(1b)···N(1) (0.333 + y, −0.333 + x, 0.667 − z): N—H = 0.93 (7), H···N = 1.80 (8), N···N = 2.727 (3) Å, N—H···N = 175 (3)°]. The Cl$^-$ anion interacts with two H(1a) atoms related by a twofold axis (passing through the Cl$^-$ anion) in the ab planes [N(1)—H(1a)···Cl ($x − y$, −y, 1 − z): N—H =

0.89 (3),   H···Cl = 2.28 (4),   N···Cl = 3.154 (3) Å, N—H···Cl = 167 (2)°]. The combination of these two kinds of intermolecular interactions produces infinite spirals of ions along the c axis.

Experimental. The mandelic acid salt of this tricyclic amine was synthesized according to a previous procedure (Whitesell, Minto & Chen, 1988). The amine was freed from its mandelic acid salt in 95% yield using 4N sodium hydroxide and dichloromethane. Colorless crystals of the half-hydrochloride salt (I) were obtained after drying and concentrating the organic layer. The data crystal had dimensions 0.31

(I)
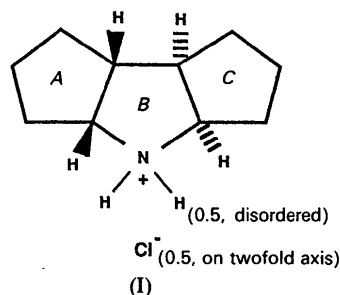
$\times$ 0.35 $\times$ 0.38 mm. A Nicolet $R3m/V$ diffractometer equipped with a graphite monochromator and a Nicolet LT-2 low-temperature delivery system (198 K) was used to collect the data. The lattice parameters were obtained from the least-squares refinement of 50 reflections with 22.25 < 2θ < 24.81°. The data were collected using the ω-scan technique with a 2θ range 3.0–55.0° and a 1.0° ω scan at 3–6° min^{-1} (h = −22→22, k = −22→22, l = 0→22).

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^3$)

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

	x	y	z	U_{eq}
Cl	4761 (1)	0	5000	93 (1)
N(1)	4591 (1)	361 (1)	3203 (1)	38 (1)
C(1)	5503 (2)	894 (2)	2882 (2)	43 (1)
C(2)	5659 (2)	1683 (2)	2377 (2)	65 (2)
C(3)	6217 (3)	1707 (3)	1692 (2)	78 (2)
C(4)	5860 (3)	751 (3)	1512 (2)	99 (2)
C(5)	5647 (2)	269 (2)	2322 (2)	58 (1)
C(6)	4786 (2)	– 614 (2)	2344 (2)	56 (1)
C(7)	4748 (2)	– 1296 (3)	2950 (3)	74 (2)
C(8)	3774 (2)	– 1852 (2)	3139 (3)	86 (2)
C(9)	3387 (2)	– 1237 (2)	3100 (2)	60 (1)
C(10)	4078 (2)	– 408 (2)	2661 (2)	45 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

N(1)—C(1)	1.478 (3)	N(1)—C(10)	1.491 (3)
C(1)—C(2)	1.519 (5)	C(1)—C(5)	1.552 (5)
C(2)—C(3)	1.498 (6)	C(3)—C(4)	1.480 (6)
C(4)—C(5)	1.553 (5)	C(5)—C(6)	1.511 (4)
C(6)—C(7)	1.543 (6)	C(6)—C(10)	1.535 (6)
C(7)—C(8)	1.500 (5)	C(8)—C(9)	1.517 (7)
C(9)—C(10)	1.527 (4)		

C(1)—N(1)—C(10)	107.7 (2)	N(1)—C(1)—C(2)	115.1 (3)
N(1)—C(1)—C(5)	106.5 (2)	C(2)—C(1)—C(5)	105.2 (2)
C(1)—C(2)—C(3)	106.6 (4)	C(2)—C(3)—C(4)	103.0 (3)
C(3)—C(4)—C(5)	105.9 (3)	C(1)—C(5)—C(4)	104.2 (3)
C(1)—C(5)—C(6)	105.6 (3)	C(4)—C(5)—C(6)	114.5 (3)
C(5)—C(6)—C(7)	115.7 (3)	C(5)—C(6)—C(10)	105.8 (3)
C(7)—C(6)—C(10)	102.2 (3)	C(6)—C(7)—C(8)	103.7 (4)
C(7)—C(8)—C(9)	106.8 (3)	C(8)—C(9)—C(10)	105.6 (3)
N(1)—C(10)—C(6)	103.6 (2)	N(1)—C(10)—C(9)	112.1 (2)
C(6)—C(10)—C(9)	107.0 (3)		

Of the 6936 reflections measured, 2267 were unique with $R_{int} = 0.0137$ from averaging symmetry-equivalent reflections. Four reflections ($\overline{4}32$, $\overline{1}32$, 323, $21\overline{2}$) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 1.1%). The data were also corrected for Lp effects. Reflections having $I \le 3\sigma(I)$ were considered unobserved (678 reflections). Absorption corrections were applied based on measured crystal faces using *SHELXTL-Plus* (Sheldrick, 1987; minimum and maximum transmission 0.937 and 0.960 respectively. Data reduction and decay correction were performed using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1987). The structure was solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976). In all, 158 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. The H-atom positions were obtained from a ΔF map. The H atoms were refined with isotropic thermal parameters except for H(4a), H(4b), H(8a) and H(8b) which were calculated in idealized positions and their isotropic thermal parameters fixed; H(3b) and H(7a) were refined freely with fixed isotropic thermal parameters. The

function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = (0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2})$. The intensity I is given by $(I_{peak} - I_{background}) \times$ (scan rate); the factor 0.02 serves to downweight intense reflections and to account for instrument instability and k is the correction for Lp effects and decay. $\sigma(I)$ was estimated from counting statistics as $\sigma(I) = [(I_{peak} + I_{background})^{1/2} \times$ (scan rate)]. An extinction correction $\chi = 0.00053$ (9) {where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$} was also applied (Sheldrick, 1987). The final $R = 0.0462$ for 1589 reflections, with $wR = 0.0524$ ($R_{all} = 0.0801$, $wR_{all} = 0.0727$) and goodness of fit = 1.71. The minimum and maximum peaks in the final ΔF map were -0.37 and 0.30 e Å^{-3}, respectively, and the maximum $|\Delta/\sigma|$ was 0.001. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient were taken from
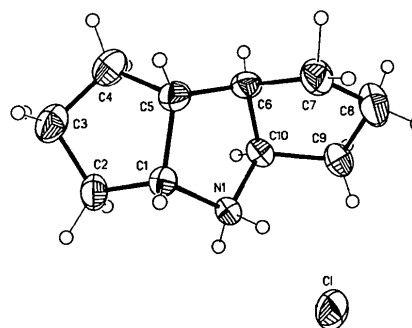

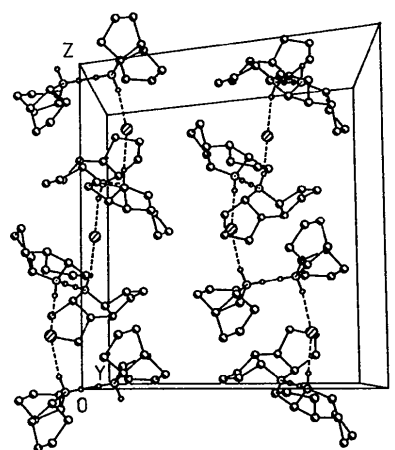Fig. 1. The title compound with 50% probability ellipsoids showing the atom-numbering scheme.

Fig. 2. Packing diagram showing the intermolecular interactions.

International Tables for X-ray Crystallography (1974, Vol. IV, p. 55). Graphics were generated using *SHELXTL-Plus*. The positional and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are given in Table 2.* A drawing of the molecule with the atom-labeling scheme is shown in Fig. 1, and the packing diagram is presented in Fig. 2.

Related literature. The chiral tricyclic amine was prepared (Whitesell *et al.*, 1988) as a chiral auxiliary for asymmetric inductions. Materials incorporating this twofold symmetric (C_2) amine are possible candidates for nonlinear optical applications (Chemla & Zyss, 1987). The packing of this salt (as well as the full hydrochloride salt currently being prepared) is of interest as an example of species with molecular C_2 symmetry. Conformations of similar tricyclic amine compounds are discussed by Minton, Whitesell, Mountzouris, Abboud & Davis (1990), Chen,

Whitesell, Price, Abboud & Davis (1990), and Abboud, Minton, Whitesell & Davis (1990).

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* Lists of anisotropic displacement parameters, H-atom positional parameters, bond lengths and angles involving H atoms, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55814 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0476]

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Structure of Phenotellurazine

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Abstract. Dibenzo[*b,e*]tellurazine, $C_{12}H_9NTe$, $M_r = 294.8$, orthorhombic, $P2_12_12_1$, $a = 6.023$ (2), $b = 8.007$ (3), $c = 20.877$ (9) Å, $V = 1006.8$ (7) Å^3, $Z = 4$, $D_x = 1.945$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.915$ mm^{-1}, $F(000) = 560$, $T = 296$ K, $R = 0.042$ for 1504 reflections and 130 parameters. The structure is similar to that of phenoxatellurine and is a hetero-cyclic analogue of anthracene in which the two CH groups of the central ring have been replaced by Te and NH. The central ring is folded along the N–Te axis, $\varphi = 142$ (3)°, and the outer phenyl rings make an angle of $\chi = 151$ (1)° with each other. The average bond distances are Te—C = 2.098 (8), N—C = 1.401 (9), C—C = 1.389 (3) Å with bond angles C—Te—C = 90.9 (3), C—N—C = 125.5 (6) and (average) C—C—C = 119.9 (2)°. All of these values appear to be normal. There are no abnormally short contact distances, and in particular, none that might be attributed to hydrogen bonding.

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