C6*A*	0.2031 (2)	−0.3841 (8)	0.3638 (2)	0.044 (1)
C1*B*	0.0683 (2)	−0.0155 (8)	0.3947 (2)	0.044 (1)
C2*B*	0.0310 (2)	0.1722 (9)	0.3672 (3)	0.073 (3)
C3*B*	−0.0337 (3)	0.169 (1)	0.3709 (3)	0.098 (4)
C4*B*	−0.0589 (2)	−0.018 (1)	0.4019 (3)	0.086 (3)
C5*B*	−0.0224 (2)	−0.203 (1)	0.4297 (3)	0.068 (3)
C6*B*	0.0410 (2)	−0.2036 (9)	0.4255 (2)	0.053 (3)

Table 2. *Geometric parameters* (Å, °)

N—C7	1.493 (5)	C6*B*—C5*B*	1.376 (6)
C7—C1*A*	1.522 (6)	C5*B*—C4*B*	1.359 (8)
C7—C1*B*	1.512 (6)	C4*B*—C3*B*	1.359 (9)
C1*A*—C2*A*	1.384 (5)	C3*B*—C2*B*	1.402 (8)
C1*A*—C6*A*	1.388 (6)	Cl···N	3.162 (2)
C2*A*—C3*A*	1.395 (7)	Cl···H1	2.2 (1)
C6*A*—C5*A*	1.385 (6)	Cl···N^i	3.192 (4)
C5*A*—C4*A*	1.362 (5)	Cl···N^{ii}	3.239 (4)
C4*A*—C3*A*	1.369 (7)	Cl···$H2^i$	2.3 (1)
C1*B*—C6*B*	1.383 (7)	Cl···$H3^{ii}$	2.3 (1)
C1*B*—C2*B*	1.377 (6)		
N—C7—C1*A*	111.9 (3)	C1*A*—C6*A*—C5*A*	120.7 (4)
N—C7—C1*B*	110.5 (3)	C6*A*—C5*A*—C4*A*	120.3 (4)
C1*A*—C7—C1*B*	113.7 (3)	C5*A*—C4*A*—C3*A*	120.0 (4)
C7—C1*A*—C2*A*	117.8 (4)	C2*A*—C3*A*—C4*A*	120.3 (4)
C6*B*—C5*B*—C4*B*	119.6 (5)	C7—C1*B*—C6*B*	121.3 (4)
C5*B*—C4*B*—C3*B*	121.1 (5)	C7—C1*B*—C2*B*	119.5 (4)
C4*B*—C3*B*—C2*B*	119.8 (5)	C6*B*—C1*B*—C2*B*	119.1 (4)
C1*B*—C2*B*—C3*B*	119.5 (5)	C1*B*—C6*B*—C5*B*	120.8 (5)
C7—C1*A*—C6*A*	123.7 (3)	Cl···H1—N	161.4 (4)
C2*A*—C1*A*—C6*A*	118.4 (4)	Cl···$H2^i$—N^i	161.1 (2)
C1*A*—C2*A*—C3*A*	120.2 (4)	Cl···$H3^{ii}$—N^{ii}	157.7 (3)

Symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (ii) $\frac{1}{2}-x, -\frac{1}{2}-y, 1-z$.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71303 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1017]

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Conformation of 4,4′-Dichloro-*N*,*N*-diethylbenzhydrylamine

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Abstract

The molecular conformation of the title compound, 4-chloro-α-(4-chlorophenyl)-*N*,*N*-diethylbenzylamine hydrochloride ($C_{17}H_{20}Cl_2N^+.Cl^-$), is helical C_s with ring twist angles of −77.0 (3) and 62.9 (4)°. The C—H bridge group is involved in a short C—H···Cl contact [3.599 (5) Å].

Comment

Benzhydrylamine (I) as well as its derived compounds, obtained by aminic and/or arylic substitution, displays inhibitive activity of the smooth musculature spasms. Some correlation between activity and hydrophobic effects has been observed (Bruno Blanch, 1990). A relationship between charge density localized at the bridge, as obtained by molecular-orbital calculations, and biological activity has been established for monoaminic substituted derivatives (Stiu, Cachau, Castro & Bruno Blanch, 1990).

The present study has been performed as part of a systematic study of derivatives of (I) to investigate the parameters governing the structure–activity relationship, especially in the case of diaminic substitution. Towards this goal the crystal structure of 4,4′-dichloro-*N*,*N*-diethylbenzhydrylamine hydrochloride (II) (the salt used

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for activity determinations owing to its great solubility) has been solved from X-ray single-crystal data.

Intramolecular bond distances and angles, listed in Table 2, do not deviate notably from expected values. The C1*A*—C7—C1*B* bridge angle, 111.9 (6)°, is smaller than that found in (I) [113.7 (3)° (Goeta, Rivero & Punte, 1993)], but similar to that found in diphenylmethane [112.5 (6)° (Barnes, Paton, Damewood & Mislow, 1981)]. The aromatic rings are planar within experimental error. The angles between the aromatic rings and the bridge plane, φ_1 and φ_2, are −77.0 (3) and 62.9 (4)°, respectively. According to this result the solid-state molecular conformation is deformed helical C_s (Barnes, Paton, Damewood & Mislow, 1981). The angle between mean ring planes is 108.8 (1)°.

In spite of the symmetry displayed by the substitutions at the aryl and amino moieties in (II), its conformation differs from the distorted helical C_2 adopted by (I) (Rivero, Punte, Rigotti, Goeta, Quinteros Rivero, Vega & Bruno Blanch, 1991) and by diphenylmethane (Barnes, Paton, Damewood & Mislow, 1981) in the solid state. The conformation of (II) resembles that observed in some monoaminic substituted derivatives of (I) (Goeta, Castelletto, Punte, Rigotti, Rivero, Bruno Blanch & Castellano, 1991) which display, as (II), greater biological activity than the parent compound (Bruno Blanch, 1990). Fig. 2 is a stereoscopic view of the molecular packing. This shows that the $Cl1^-$ and CH_4Cl-CH_4Cl-CH-NEt_2H^+ ions are joined through hydrogen bonds N1—H1···Cl1 [N1···Cl1 3.050 (5) Å]. Among the intermolecular distances is a short contact C7—H7···Cl1 of 3.599 (5) Å that can be interpreted as a hydrogen bond (Taylor & Kennard, 1982).

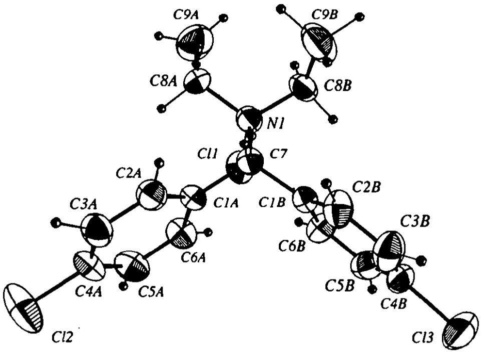

Fig. 1. *ORTEP* illustration of the molecular structure showing non-H atom labelling. The atoms are represented by their 50% probability ellipsoids for thermal motion. H atoms have been arbitrarily reduced.

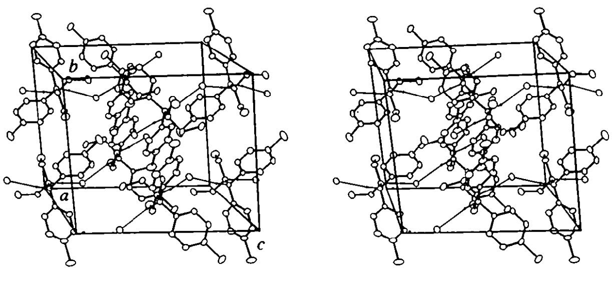

Fig. 2. Stereoscopic view of the molecular packing (*c* across, *a* down).

Experimental

Crystal data

$C_{17}H_{20}Cl_2N^+.Cl^-$
M_r = 344.7
Monoclinic
$P2_1/n$
a = 11.865(3) Å
b = 11.916(1) Å
c = 13.550(2) Å
β = 112.97(2)°
V = 1764(1) Å^3
Z = 4

D_x = 1.298 Mg m^{-3}
Mo *Kα* radiation
λ = 0.7093 Å
Cell parameters from 23 reflections
θ = 6–14°
μ = 0.514 mm^{-1}
T = 295 K
Irregular shape
0.37 × 0.15 × 0.12 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.693, T_{max} = 1.066
2720 measured reflections
2720 independent reflections

2566 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.009
θ_{max} = 25°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 16$
1 standard reflection frequency: 100 min intensity variation: 3%

Refinement

Refinement on *F*
Final *R* = 0.0475
wR = 0.0496
S = 2.41
1404 reflections
198 parameters
Only H-atom *U*'s refined
$w = k/[\sigma^2(F) + 0.000226F^2]$

$(\Delta/\sigma)_{max}$ = 0.027
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

The H atoms were refined riding on their bound atoms. Overall temperature factors were refined for H atoms of the phenyl, CH_3 and CH_2 groups. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Software used to prepare material for publication: *ORTEP* (Johnson, 1965); *MolEN*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Cl1	0.0822 (1)	0.3383 (1)	0.6582 (1)	0.0664 (7)
Cl2	0.0270 (2)	−0.2061 (1)	0.9578 (2)	0.099 (1)
Cl3	0.1694 (2)	0.5685 (2)	1.2009 (1)	0.102 (1)
C1*A*	0.2160 (5)	0.1118 (4)	0.9275 (4)	0.044 (2)
C2*A*	0.1040 (5)	0.0822 (5)	0.8498 (5)	0.060 (2)

C3*A*	0.0464 (6)	−0.0168 (5)	0.8582 (5)	0.068 (2)
C4*A*	0.1004 (6)	−0.0843 (5)	0.9446 (6)	0.059 (2)
C5*A*	0.2143 (6)	−0.0589 (5)	1.0225 (5)	0.062 (2)
C6*A*	0.2707 (5)	0.0410 (5)	1.0144 (4)	0.054 (2)
C1*B*	0.2462 (5)	0.3118 (4)	0.9931 (4)	0.043 (2)
C2*B*	0.1719 (5)	0.4015 (4)	0.9479 (4)	0.051 (2)
C3*B*	0.1475 (5)	0.4808 (5)	1.0118 (5)	0.059 (2)
C4*B*	0.1981 (5)	0.4680 (5)	1.1212 (4)	0.055 (2)
C5*B*	0.2714 (5)	0.3785 (5)	1.1665 (4)	0.058 (2)
C6*B*	0.2965 (5)	0.2996 (5)	1.1036 (4)	0.054 (2)
C7	0.2665 (5)	0.2257 (4)	0.9178 (4)	0.044 (2)
N1	0.4003 (4)	0.2221 (4)	0.9338 (4)	0.049 (2)
C8*A*	0.4376 (5)	0.3385 (5)	0.9126 (5)	0.058 (2)
C8*B*	0.4275 (5)	0.1323 (5)	0.8685 (4)	0.055 (2)
C9*A*	0.5740 (6)	0.3434 (6)	0.9306 (6)	0.092 (3)
C9*B*	0.3670 (7)	0.1499 (6)	0.7471 (5)	0.095 (3)

Table 2. *Geometric parameters* (Å, °)

C1*A*—C2*A*	1.381 (7)	C6*B*—C1*B*	1.387 (7)
C2*A*—C3*A*	1.390 (8)	Cl2—C4*A*	1.737 (7)
C3*A*—C4*A*	1.358 (8)	Cl3—C4*B*	1.733 (7)
C4*A*—C5*A*	1.385 (7)	C1*A*—C7	1.510 (8)
C5*A*—C6*A*	1.391 (8)	C1*B*—C7	1.531 (9)
C6*A*—C1*A*	1.387 (7)	N1—C7	1.517 (9)
C1*B*—C2*B*	1.369 (7)	N1—C8*A*	1.517 (9)
C2*B*—C3*B*	1.387 (9)	N1—C8*B*	1.502 (9)
C3*B*—C4*B*	1.374 (7)	C8*A*—C9*A*	1.541 (9)
C4*B*—C5*B*	1.362 (8)	C8*B*—C9*B*	1.531 (8)
C5*B*—C6*B*	1.377 (9)		
C1*A*—C2*A*—C3*A*	120.8 (4)	Cl2—C4*A*—C5*A*	118.9 (5)
C2*A*—C3*A*—C4*A*	119.5 (5)	Cl3—C4*B*—C5*B*	120.4 (5)
C3*A*—C4*A*—C5*A*	121.3 (6)	C2*A*—C1*A*—C7	117.4 (4)
C4*A*—C5*A*—C6*A*	118.9 (5)	C6*A*—C1*A*—C7	123.4 (4)
C5*A*—C6*A*—C1*A*	120.5 (4)	C2*B*—C1*B*—C7	117.8 (5)
C6*A*—C1*A*—C2*A*	119.0 (5)	C6*B*—C1*B*—C7	122.4 (5)
C1*B*—C2*B*—C3*B*	120.5 (5)	C1*A*—C7—N1	113.0 (5)
C2*B*—C3*B*—C4*B*	119.3 (5)	C1*B*—C7—N1	110.6 (5)
C3*B*—C4*B*—C5*B*	120.5 (6)	C1*A*—C7—C1*B*	111.9 (6)
C4*B*—C5*B*—C6*B*	120.6 (6)	C7—N1—C8*A*	108.3 (5)
C5*B*—C6*B*—C1*B*	119.4 (5)	C7—N1—C8*B*	112.9 (5)
C6*B*—C1*B*—C2*B*	119.7 (6)	C8*A*—N1—C8*B*	113.2 (6)
Cl2—C4*A*—C3*A*	119.8 (4)	N1—C8*A*—C9*A*	111.8 (5)
Cl3—C4*B*—C3*B*	119.1 (4)	N1—C8*B*—C9*B*	114.7 (5)

Crystal source: the compound was synthesized according to non-literature described techniques, by reaction of 4,4′-dichlorobenzhydryl bromide with diethylamine in nitromethane.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71164 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1018]

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Structure of Ethyl 2-Cyano-3-(4-methylphenyl)propenoate

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Abstract

The dihedral angle between the plane of the 4-methylbenzylidene group and the cyanopropenoic acid ethyl ester group is 3.33°. The molecule is nearly planar.

Comment

2-Cyano-3-(2-methoxyphenyl)propenoic acid methyl ester (Nakatani, Hayashi & Hidaka, 1992) crystallizes in a non-centrosymmetric space group and has a large second-harmonic generation (SHG) efficiency. We have synthesized a series of substituted α-cyano cinnamic acid esters. The title compound is one of them, which happens to crystallize in a centrosymmetric space group and therefore has no non-linear optical properties. This has been confirmed by SHG efficiency measurements on a powder sample using the method of Kurtz & Perry (1968).

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