C1—C9—C10	112.1 (2)	C17—C25—C26	112.6 (3)
O1—C10—C9	121.9 (4)	O2—C26—C25	121.5 (3)
O1—C10—C11	119.1 (2)	O2—C26—C27	120.3 (3)
C9—C10—C11	119.0 (3)	C25—C26—C27	118.2 (3)
C10—C11—C12	122.1 (2)	C26—C27—C28	124.0 (3)
C10—C11—C16	117.5 (3)	C26—C27—C32	116.9 (4)
C12—C11—C16	120.4 (3)	C28—C27—C32	119.1 (3)
C11—C12—C13	119.3 (2)	C27—C28—C29	120.4 (4)
C12—C13—C14	120.2 (3)	C28—C29—C30	120.2 (3)
C13—C14—C15	120.5 (4)	C29—C30—C31	119.8 (4)
C14—C15—C16	120.1 (3)	C30—C31—C32	120.6 (4)
C11—C16—C15	119.5 (3)	C27—C32—C31	119.9 (3)

The structure was solved by direct methods and difference Fourier syntheses. Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for H atoms.

All calculations were performed on a PDP 11/44 computer with the *SDP* (Frenz, 1978) programs.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1131). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,6-Dichlorophenylguanidinium Chloride

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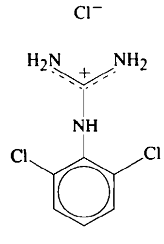
Abstract

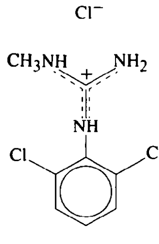
The C—N bonds in $C_7H_8Cl_2N_3^+.Cl^-$ were found to differ in length [1.304 (4), 1.308 (4) and 1.493 (4) Å]. There is not, therefore, the symmetrical delocalization around the central N_3C expected for a guanidinium system, and the molecule is closer in nature to a substituted amidinium salt.

Comment

Structural studies of guanidines and their salts continue to attract attention (Carpy, Leger, Wermuth & Leclerc, 1981; Hafelinger & Kuske, 1991) because of their biological and pharmaceutical, *e.g.* neuroleptic and antihypersensitive, activity (Rouot, Leclerc, Wermuth, Miesch & Schwarz, 1978; Bream, Lauener, Picard, Scholtysik & White, 1975). In the title compound, (I), the asymmetric unit contains one molecule, resolved as the *E* isomer (Fig. 1).



(I)



(II)

Two of the three C—N bond lengths of the N_3C fragment are similar [N(14)—C(12) 1.304 (4) and N(13)—C(12) 1.308 (4) Å] and are only slightly longer than the mean of 277 C_{sp^2}=N double bonds [1.295 Å] located in the Cambridge Structural Database (Allen *et al.*, 1987), indicating delocalization around the N—C—N fragment. The third C—N bond length [C(12)—N(11) 1.493 (4) Å] and the C(aryl)—N distance [C(1)—N(11) 1.519 (4) Å] are clearly single bond in character, and thus the delocalization is not across the whole of the

N_3C fragment, nor does it extend into the aromatic ring. The C(1)—N(11)—C(12) bond angle of 115.6 (3)°, which is midway between that expected for an sp^2 and for an sp^3 N atom, and the C(12)—N(11)—C(1)—C(2) torsion angle of 82.3 (4)°, support this point of view. The structure has, therefore, more in common with a substituted amidinium salt than the closely related 2,6-dichlorophenyl-2-methyl-1-guanidinium chloride hydrate, (II) (Carpy, Leger, Wermuth & Leclerc, 1981), in which the C—N bond lengths of 1.312 (6), 1.349 (6) and 1.388 (6) Å indicate delocalization around the N_3C fragment, as expected for a guanidinium salt. The only chemical difference between the two compounds is the methyl substituent in (II). In the title compound, two of the N—C—N angles are similar: N(13)—C(12)—N(14) 120.8 (3) and N(13)—C(12)—N(11) 122.1 (3)°. The third, N(11)—C(12)—N(14), is smaller, at 117.1 (3)°, which is similar to that found for 2,6-dichlorophenyl-2-methyl-1-guanidinium chloride hydrate (Carpy, Leger, Wermuth & Leclerc, 1981).

There are weak intermolecular hydrogen bonds present extending through the cell, as indicated by the close Cl···N contacts [Cl(1)···N(13) 3.210 (3), Cl(1)···N(13)$(1-x, y-\frac{1}{2}, -\frac{1}{2}-z)$ 3.269 (2), Cl(1)···N(14)$(x-\frac{1}{2}, \frac{1}{2}-y, -z-1)$ 3.260 (3) and Cl(1)···N(14)$(1-x, y-\frac{1}{2}, -\frac{1}{2}-z)$ 3.209 (3) Å].

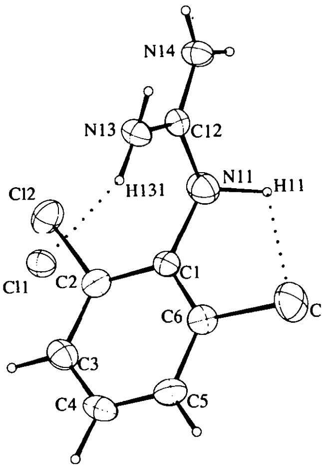


Fig. 1. Molecular structure and non-H atomic labels of (I). Displacement ellipsoids are shown at 50% probability levels. H atoms are drawn as small circles of arbitrary radii.

Experimental

Crystal data

$C_7H_8Cl_2N_3^+.Cl^-$
$M_r = 240.52$
Orthorhombic
$P2_12_12_1$
$a = 10.3302 (3)$ Å
$b = 10.6380 (2)$ Å
$c = 9.7044 (2)$ Å

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 16 reflections
$\theta = 28.74–34.80°$
$\mu = 7.644$ mm^{-1}
$T = 293$ K

$V = 1066.44$ Å^3
$Z = 4$
$D_x = 1.50$ Mg m^{-3}

Irregular block
0.40 × 0.25 × 0.22 mm
Colourless
Crystal source: Associated Octel Company Ltd

Data collection

Rigaku AFC-7*R* diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.403$, $T_{\max} = 1.000$
1800 measured reflections
1744 independent reflections
1687 observed reflections $[F > 3\sigma(F)]$

$R_{\rm int} = 0.0411$
$\theta_{\max} = 75°$
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 12$
3 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on *F*
$R = 0.0405$
$wR = 0.0429$
$S = 1.0991$
1687 reflections
145 parameters
H atoms riding on attached C or N
Weighting scheme: Chebyshev
$(\Delta/\sigma)_{\max} = 0.056$
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Extinction correction: Larson (1970)
Extinction coefficient: 205.4 (69)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
Absolute configuration: Flack (1983) enantiopole parameter refined to 1.02 (3) for the opposite hand of the solution, and 0.02 (3) for the correct hand

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

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

	x	y	z	$U_{\rm eq}$
Cl(1)	0.40364 (8)	0.07692 (7)	−0.23293 (8)	0.0389 (4)
Cl(2)	0.41486 (9)	0.15515 (9)	−0.7043 (1)	0.0520 (5)
Cl(6)	0.8276 (1)	0.0082 (1)	−0.4028 (1)	0.0665 (7)
N(11)	0.6806 (3)	0.1988 (3)	−0.5850 (4)	0.0534 (2)
N(13)	0.5681 (3)	0.2910 (2)	−0.3868 (3)	0.0386 (1)
N(14)	0.6745 (3)	0.4188 (3)	−0.5361 (3)	0.0406 (1)
C(1)	0.6171 (3)	0.0734 (3)	−0.5533 (3)	0.0334 (1)
C(2)	0.4960 (3)	0.0432 (3)	−0.6065 (3)	0.0354 (1)
C(3)	0.4373 (4)	−0.0726 (3)	−0.5878 (4)	0.0419 (2)
C(4)	0.5005 (4)	−0.1605 (3)	−0.5081 (4)	0.0438 (2)
C(5)	0.6198 (4)	−0.1360 (3)	−0.4531 (4)	0.0458 (2)
C(6)	0.6762 (4)	−0.0191 (3)	−0.4770 (4)	0.0419 (2)
C(12)	0.6377 (3)	0.3068 (3)	−0.4980 (3)	0.0327 (1)

Table 2. *Selected geometric parameters* (Å, °)

Cl(2)—C(2)	1.738 (3)	C(1)—C(2)	1.391 (5)
Cl(6)—C(6)	1.746 (4)	C(1)—C(6)	1.374 (5)
N(11)—C(1)	1.519 (4)	C(2)—C(3)	1.384 (5)
N(11)—C(12)	1.493 (4)	C(3)—C(4)	1.378 (5)
N(13)—C(12)	1.308 (4)	C(4)—C(5)	1.369 (5)
N(14)—C(12)	1.304 (4)	C(5)—C(6)	1.392 (5)
C(1)—N(11)—C(12)	115.6 (3)	C(3)—C(4)—C(5)	121.1 (3)
N(11)—C(1)—C(2)	121.1 (3)	C(4)—C(5)—C(6)	118.8 (3)

N(11)—C(1)—C(6)	123.1 (3)	Cl(6)—C(6)—C(1)	120.1 (3)
C(2)—C(1)—C(6)	115.7 (3)	Cl(6)—C(6)—C(5)	117.0 (3)
Cl(2)—C(2)—C(1)	118.6 (2)	C(1)—C(6)—C(5)	122.9 (3)
Cl(2)—C(2)—C(3)	118.0 (3)	N(11)—C(12)—N(13)	122.1 (3)
C(1)—C(2)—C(3)	123.4 (3)	N(11)—C(12)—N(14)	117.1 (3)
C(2)—C(3)—C(4)	118.0 (3)	N(13)—C(12)—N(14)	120.8 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *MSC/AFC Diffractometer Control Software*. Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *CAMERON* (Pearce & Watkin, 1993). Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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