

O3—P1—C6	110.9 (2)	O3—C12—C13	106.9 (4)
O4—P1—C6	107.1 (2)	O4—C14—C15	106.8 (3)
O5—P2—C1	114.6 (2)	P2—C16—C17	125.5 (3)
O5—P2—C16	111.2 (2)	P2—C16—C21	114.5 (3)
O5—P2—C22	112.0 (2)	C17—C16—C21	119.8 (4)
C1—P2—C16	106.3 (2)	C16—C17—C18	119.7 (4)
C1—P2—C22	108.4 (2)	C17—C18—C19	120.1 (4)
C16—P2—C22	103.6 (2)	C18—C19—C20	120.6 (4)
O6—P3—C1	114.0 (2)	C19—C20—C21	119.5 (4)
O6—P3—C28	112.0 (2)	C16—C21—C20	120.2 (4)
O6—P3—C34	110.9 (2)	P2—C22—C23	121.7 (3)
C1—P3—C28	107.7 (2)	P2—C22—C27	118.9 (3)
C1—P3—C34	104.4 (2)	C23—C22—C27	119.4 (3)
C28—P3—C34	107.4 (2)	C22—C23—C24	120.3 (4)
P1—O3—C12	123.8 (3)	C23—C24—C25	118.7 (5)
P1—O4—C14	120.5 (3)	C24—C25—C26	121.4 (4)
P2—C1—P3	112.4 (2)	C25—C26—C27	120.2 (4)
P2—C1—C2	111.2 (2)	C22—C27—C26	120.0 (4)
P3—C1—C2	109.3 (2)	P3—C28—C29	123.1 (3)
C1—C2—C3	120.1 (3)	P3—C28—C33	117.6 (3)
C1—C2—C7	121.3 (3)	C29—C28—C33	119.4 (4)
C3—C2—C7	118.3 (3)	C28—C29—C30	119.6 (4)
C2—C3—C4	124.0 (3)	C29—C30—C31	120.0 (5)
C3—C4—C5	116.7 (3)	C30—C31—C32	119.9 (5)
C3—C4—C8	122.9 (3)	C31—C32—C33	121.1 (4)
C5—C4—C8	120.4 (3)	C28—C33—C32	120.0 (4)
O1—C5—C4	118.0 (3)	P3—C34—C35	116.6 (3)
O1—C5—C6	121.9 (3)	P3—C34—C39	123.9 (3)
C4—C5—C6	120.1 (3)	C35—C34—C39	119.6 (4)
P1—C6—C5	120.8 (3)	C34—C35—C36	121.1 (4)
P1—C6—C7	118.6 (3)	C35—C36—C37	118.5 (4)
C5—C6—C7	120.6 (3)	C36—C37—C38	120.6 (4)
C2—C7—C6	120.2 (3)	C37—C38—C39	120.8 (4)
C4—C8—C9	109.3 (3)	C34—C39—C38	119.4 (4)
C4—C8—C10	110.3 (3)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *LSFM* in *MolEN*. Software used to prepare material for publication: *PLATON* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 2307–2309

Isopropylamidinium Chloride

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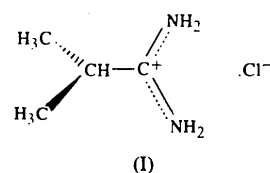
(Received 8 August 1994; accepted 27 February 1996)

Abstract

The amidine fragment in each of the two independent molecules of $C_4H_{11}N_2^+ \cdot Cl^-$ is essentially planar; the bond angles around the central C atom sum to $360.0(4)^\circ$ in each case. This, and the almost equal C—N bond lengths [1.299(6) to 1.320(5) Å], indicate that an amidinium salt has been formed.

Comment

A number of papers concerning the structures of amidines have appeared (Tykarska, Jaskolski & Kos-turkiewicz, 1986; Thailambal, Pattabhi & Guru Row, 1986; Barker, Gould & Kilner, 1987; Barker & Kilner, 1993; Alcock, Barker & Kilner, 1988; Alcock, Barker, Blacker, Errington & Wallbridge, 1994; Dehnicke, 1990), reflecting the widespread interest in such compounds because of their pharmaceutical and biological importance, bonding characteristics and ligand properties. The structural investigation of the title compound, (I), was undertaken to investigate the effect of alkyl substitution on the central amidine fragment.



The asymmetric unit consists of two molecules with N—H...Cl hydrogen bonding present (Fig. 1), as it is throughout the structure (Fig. 2). The skeletal C—N bond lengths are clearly not differentiated in terms of single- or double-bond character, with mean C—N bond lengths of 1.317(5) and 1.300(6) Å for each molecule, respectively. They are similar to those found for acetamidinium chloride [1.305(2) and 1.310(3) Å; Cannon, White & Willis, 1976] and bis(acetamidinium)-carbonate monohydrate [1.308(5) and 1.315 Å; Norrestam, 1984]. They differ somewhat to those found for acetamidine [1.298(1) and 1.344(1) Å; Norrestam, Mertz & Crossland, 1983], *N,N*-diphenylbenzamidine [1.302(7) and 1.360(8) Å; Alcock *et al.*, 1988] and

benzamidinium hydrochloride monohydrate [1.293 (7) and 1.328 (7) Å; Thailambal *et al.*, 1986]. The almost equal C—N bond lengths in the title compound and the planarity of the CCN₂ fragment are indicative of an amidinium salt configuration. Further, the mean C—N distance of 1.309 (6) Å is, as expected, longer than that found in [CH₃CICNH₂]⁺Cl⁻ (1.265 Å) which contains only one C—NH₂ linkage. Closer comparison of the C—N bond lengths in benzamidinium hydrochloride (Thailambal *et al.*, 1986) and isopropylamidinium chloride indicate the aryl substituent to be more restrictive toward delocalization than the alkyl group. The N—C—N angle [mean 120.4 (4)°] is similar to that of acetamidinium chloride [120.5 (2)°; Cannon *et al.*, 1976], but deviates slightly from those found for bis-acetamidinium carbonate monohydrate [121.6 (3)°; Norrestam, 1984] and benzamidinium hydrochloride hydrate [121.6 (4)°; Thailambal *et al.*, 1987], and deviates considerably from that found for acetamide [125.5 (1)°; Norrestam *et al.*, 1983]. This indicates the amidinium

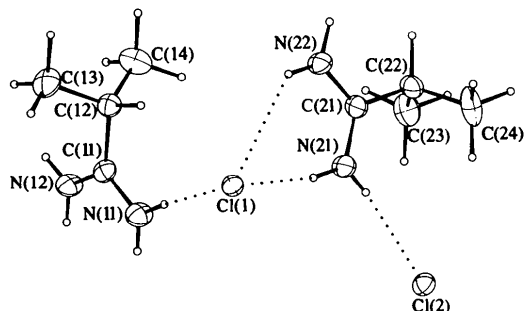


Fig. 1. Molecular structure and non-H atomic labels of isopropylamidinium chloride. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radius.

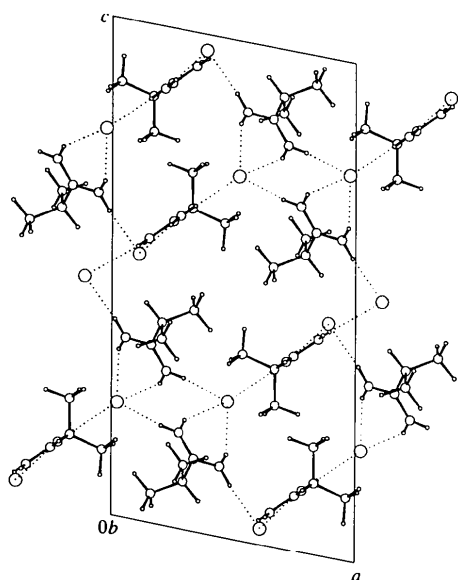


Fig. 2. Projection down the *c* axis.

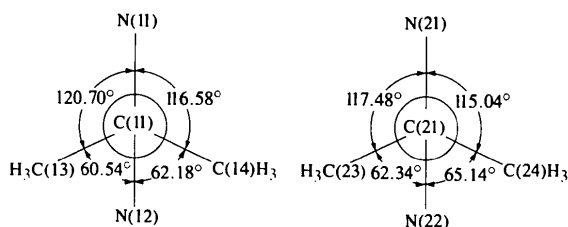


Fig. 3. View of each molecule along C(x1)—C(x2).

to chloride interaction to be stronger in acetamidinium chloride and isopropylamidinium chloride than in benzamidinium hydrochloride. Again this may be due to the influence of the aryl substituent. Further, the amidinium oxygen interaction is not as strong as the amidinium chloride interaction. The delocalization around the N—C—N fragment is also responsible for a short C—CNN bond [mean 1.491 (6) Å]. There are a number of close Cl⁻ ion to amino-H atom contacts as evidenced by short N...Cl distances (3.15–3.26 Å), indicating strong interactions. Consideration of the relationship of the methyl groups to the planar N—C—N fragment shows that they adopt an *anti* position to it (Fig. 3).

Experimental

The title compound was obtained from the Associated Ocel Company Ltd.

Crystal data

C₄H₁₁N₂⁺.Cl⁻
M_r = 122.60
 Monoclinic
*P*2₁/*n*
a = 10.6713 (7) Å
b = 6.6936 (6) Å
c = 21.0352 (7) Å
 β = 100.776 (4)°
V = 1476.04 Å³
Z = 8
D_x = 1.103 Mg m⁻³
D_m not measured

Data collection

Rigaku AFC-7R diffractometer
 $\theta/2\theta$ scans
 Absorption correction:
 empirical, ψ scan
 (Molecular Structure Corporation, 1985, 1992)
T_{min} = 0.92, *T_{max}* = 1.00
 2435 measured reflections
 1521 independent reflections

Refinement

Refinement on *F*
R = 0.0368
wR = 0.0443
S = 4.976

Cu K α radiation

λ = 1.5418 Å
 Cell parameters from 24 reflections
 θ = 37.1–37.8°
 μ = 3.85 mm⁻¹
T = 153 K
 Prism
 0.30 × 0.24 × 0.20 mm
 Colourless

1366 observed reflections

[*F* > 2.5 σ (*F*)]

R_{int} = 0.0196

θ_{\max} = 50.78°

h = -10 → 10

k = -1 → 6

l = -1 → 20

3 standard reflections

monitored every 100

reflections

intensity decay: -0.015%

$\Delta\rho_{\max}$ = 0.488 e Å⁻³

$\Delta\rho_{\min}$ = -0.325 e Å⁻³

Extinction correction:

Larson (1970)

1366 reflections
216 parameters
H atoms riding on attached
C or N
 $w = 1/[\sigma^2(F) + 0.0004F^2]$
 $(\Delta/\sigma)_{\max} = 0.0181$

Extinction coefficient:
32.7 (22)
Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Cl(1)	−0.11100 (9)	0.7296 (2)	0.46747 (5)	0.0349 (6)
Cl(2)	−0.52314 (9)	0.5846 (2)	0.27248 (5)	0.0374 (6)
N(11)	0.0557 (4)	0.8273 (6)	0.3595 (2)	0.0455 (2)
N(12)	0.2237 (4)	0.7725 (6)	0.3083 (2)	0.0429 (2)
N(21)	−0.2828 (4)	0.4288 (6)	0.3745 (2)	0.0427 (2)
N(22)	−0.1522 (3)	0.2014 (6)	0.4318 (2)	0.0408 (2)
C(11)	0.1618 (4)	0.7311 (7)	0.3551 (2)	0.0366 (3)
C(12)	0.2060 (4)	0.5734 (7)	0.4039 (2)	0.0395 (3)
C(13)	0.3382 (4)	0.6236 (9)	0.4410 (2)	0.0557 (3)
C(14)	0.2051 (5)	0.3697 (8)	0.3713 (2)	0.0576 (3)
C(21)	−0.2525 (4)	0.2440 (7)	0.3884 (2)	0.0377 (3)
C(22)	−0.3310 (4)	0.0758 (7)	0.3553 (2)	0.0415 (3)
C(23)	−0.3302 (5)	0.0793 (8)	0.2834 (2)	0.0551 (3)
C(24)	−0.4628 (5)	0.0770 (9)	0.3722 (3)	0.0587 (4)

Table 2. Geometric parameters (\AA , $^\circ$)

N(11)—C(11)	1.320 (5)	Cl(1)··N(11)	3.20 (2)
N(12)—C(11)	1.315 (5)	Cl(1)··N(21)	3.15 (2)
N(21)—C(21)	1.299 (6)	Cl(1)··N(22)	3.62 (2)
N(22)—C(21)	1.301 (5)	Cl(1)··N(22 ⁱ)	3.26 (2)
C(11)—C(12)	1.486 (6)	Cl(1)··N(22 ⁱⁱ)	3.22 (2)
C(12)—C(13)	1.517 (6)	Cl(2)··N(11 ⁱⁱⁱ)	3.23 (2)
C(12)—C(14)	1.526 (6)	Cl(2)··N(12 ^{iv})	3.19 (2)
C(21)—C(22)	1.495 (6)	Cl(2)··N(12 ⁱⁱⁱ)	3.24 (2)
C(22)—C(23)	1.514 (6)	Cl(2)··N(21)	3.20 (2)
C(22)—C(24)	1.513 (6)		
N(11)—C(11)—N(12)	120.4 (4)	N(21)—C(21)—N(22)	120.3 (4)
N(11)—C(11)—C(12)	117.7 (4)	N(21)—C(21)—C(22)	121.2 (4)
N(12)—C(11)—C(12)	121.9 (4)	N(22)—C(21)—C(22)	118.5 (4)
C(11)—C(12)—C(13)	110.0 (4)	C(21)—C(22)—C(23)	110.3 (4)
C(11)—C(12)—C(14)	110.4 (4)	C(21)—C(22)—C(24)	110.6 (4)
C(13)—C(12)—C(14)	110.9 (4)	C(23)—C(22)—C(24)	114.4 (4)

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

Data collection: Rigaku AFC-7R software. Cell refinement: Rigaku AFC-7R software. Data reduction: Rigaku AFC-7R software. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). 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*.

HRP wishes to thank the CCDC and the University Chemical Laboratories, Cambridge, for the use of X-ray diffraction and computing facilities.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1996). **C52**, 2309–2311

Diisopropylammonium Bromide

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(Received 20 December 1995; accepted 22 March 1996)

Abstract

In the title compound, $\text{C}_6\text{H}_{16}\text{N}^+\text{Br}^-$, each diisopropylammonium cation interacts with the bromide anion via two hydrogen bonds leading to an infinite one-dimensional chain of molecules in the crystal. The cation has an approximate twofold axis of symmetry.

Comment

Diisopropylammonium bromide (I) was obtained in the course of studying the reaction of $\text{BrRe}(\text{CO})_5$ with lithium dialkylamides with the intention of generating lithium carbamoylmetallates as precursors for amino-