Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[4-(3-Aminopropyl)piperazin-1-yl]propan-1-aminium chloride

Paz Vaqueiro

Department of Chemistry, Heriot–Watt University, Edinburgh EH14 4AS, Scotland

Correspondence e-mail: chepv@hw.ac.uk

Received 31 May 2006
Accepted 1 June 2006

The title compound, $C_{10}H_{25}N_4^+\cdot Cl^-$, contains monoprotonated amine cations and chloride anions. The cations form chains along the [101] direction *via* N—H···N bonds, while N—H···Cl hydrogen bonds link the anions and cations into a three-dimensional structure.

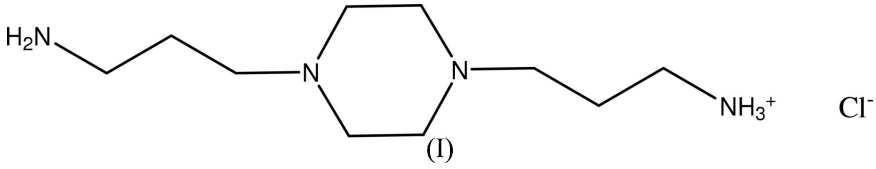
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.033
Data-to-parameter ratio = 10.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In solvothermal synthesis, organic amines are generally used as structure-directing agents, and it is known that sometimes salts of the amines appear as unwanted side products. However, recent work on the solvothermal synthesis of phosphates (Rao *et al.*, 2000) and sulfates (Behera *et al.*, 2004) suggests that these amine salts might play a role in the formation of open-framework phases. It has also been found that the use of amine salts as sources of structure-directing agents may result in the formation of new open-framework structures.



(I)

In the title compound, $C_{10}H_{25}N_4^+\cdot Cl^-$, (I), which was the unexpected product of a solvothermal reaction, the amine 1,4-bis(3-aminopropyl)piperazine (bapp) crystallizes as a monoprotonated cation, H^+bapp, accompanied by a charge-balancing chloride anion (Fig. 1). As well as electrostatic forces, the anions and cations in (I) interact by means of hydrogen bonds (Table 1). The H^+bapp cations are connected by strong N—H···N hydrogen bonds, forming infinite chains that run along the [101] direction. The chains are cross-linked by N—H···Cl bonds arising from the terminal $-NH_2$ and $-NH_3^+$ groups to form layers parallel to the *ac* plane (Fig. 2). Further N—H···Cl hydrogen bonds link the layers into a three-dimensional structure (Fig. 3).

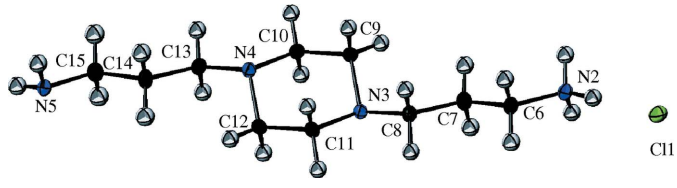


Figure 1
View of (I), showing 50% probability displacement ellipsoids (arbitrary spheres for the H atoms).

Experimental

A mixture of CuCl (2 mmol), Te (1 mmol) and 1,4-bis(3-aminopropyl)piperazine (4.2 ml) was loaded into a 23 ml Teflon-lined steel autoclave, heated for 13 days at 473 K and then cooled to room temperature over a period of 12 h. The product, consisting of hygroscopic colourless needles of (I) and a black powder, was filtered and washed with methanol and acetone.

Crystal data

$C_{10}H_{25}N_4^+\cdot Cl^-$
M_r = 236.79
Monoclinic, $P2_1/c$
a = 10.9035 (9) Å
b = 15.9679 (13) Å
c = 7.8750 (6) Å
β = 96.693 (4)°
V = 1361.74 (19) Å^3
Z = 4
D_x = 1.155 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 K
Needle, colourless
0.50 × 0.10 × 0.10 mm

Data collection

Bruker–Nonius APEX2 CCD area-detector diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.811, T_{max} = 0.974
18638 measured reflections
3967 independent reflections
2257 reflections with $I > 3.00\sigma(I)$
R_{int} = 0.023
θ_{max} = 30.1°

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.033
S = 1.11
2257 reflections
211 parameters
Only H-atom coordinates refined
$W = [1 - (\delta F/6\sigma F)^2]^2/[0.491T_0(x) + 0.340T_1(x) + 0.263T_2(x)]$ where T_i are Chebychev polynomials and $x = F/F_{max}$ (Watkin, 1994; Prince, 1982)
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H23···N5^{i}	0.93 (2)	1.83 (2)	2.7574 (18)	175 (2)
N2—H21···Cl1^{i}	0.91 (2)	2.27 (2)	3.1761 (12)	176 (2)
N2—H22···Cl1^{ii}	0.88 (2)	2.30 (2)	3.1853 (13)	178 (1)
N5—H52···Cl1^{iii}	0.89 (2)	2.58 (2)	3.4104 (13)	156 (2)
N5—H53···Cl1^{iv}	0.85 (2)	2.61 (2)	3.4344 (13)	164 (2)

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

H atoms were located in difference maps and their positions were freely refined; U_{iso}(H) = 1.2U_{eq}(carrier).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ATOMS* (Dowty, 2000); software used to prepare material for publication: *CRYSTALS*.

The author thanks the UK EPSRC for an Advanced Research Fellowship.

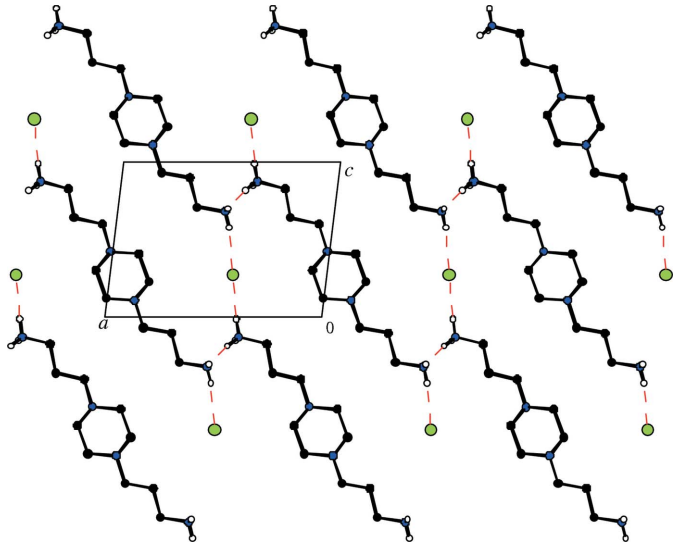


Figure 2
View of a layer parallel to the (010) plane, showing the network of hydrogen bonds (dashed lines). Hydrogen atoms not participating in hydrogen bonding have been omitted for clarity.

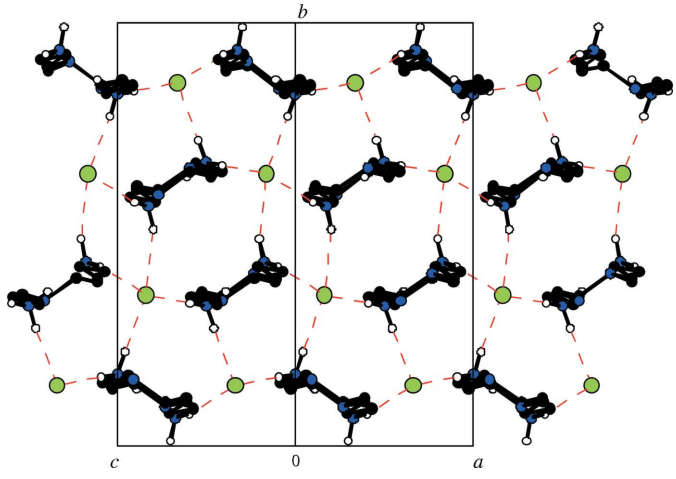


Figure 3
View of the packing in (I). Drawing conventions as in Fig. 2.

References

Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Behera, J. N., Gopalkrishnan, K. V. & Rao, C. N. R. (2004). *Inorg. Chem.* **43**, 2636–2642.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Bruker (2005). *APEX2*. Version 1.27. Bruker AXS Inc., Madison, Wisconsin, USA.
Dowty, E. (2000). *ATOMS*. Version 6.1. Shape Software, Kingsport, Tennessee, USA.
Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer-Verlag.
Rao, C. N. R., Natarajan, S. & Neeraj, S. (2000). *J. Solid State Chem.* **152**, 302–321.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Watkin, D. J. (1994). *Acta Cryst.* A**50**, 411–437.

supporting information

Acta Cryst. (2006). E**62**, o2632–o2633 [https://doi.org/10.1107/S1600536806020988]

3-[4-(3-Aminopropyl)piperazin-1-yl]propan-1-aminium chloride

Paz Vaqueiro

3-[4-(3-aminopropyl)piperazin-1-yl]propan-1-aminium chloride

Crystal data

$C_{10}H_{25}N_4^+ \cdot Cl^-$
$M_r = 236.79$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.9035$ (9) Å
$b = 15.9679$ (13) Å
$c = 7.8750$ (6) Å
$\beta = 96.693$ (4)°
$V = 1361.74$ (19) Å^3
$Z = 4$
$F(000) = 520$
$D_x = 1.155$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3967 reflections
$\theta = 2$–30°
$\mu = 0.26$ mm^{-1}
$T = 293$ K
Needle, colourless
0.50 × 0.10 × 0.10 mm

Data collection

Bruker-Nonius APEX-2 CCD area-detector diffractometer
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.811$, $T_{max} = 0.974$
18638 measured reflections
3967 independent reflections
2257 reflections with $I > 3.00\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 30.1°$, $\theta_{min} = 1.9°$
$h = -13 \rightarrow 15$
$k = -22 \rightarrow 22$
$l = -11 \rightarrow 9$

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.033$
$S = 1.11$
2257 reflections
211 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
Only H-atom coordinates refined
$W = [1-(\delta F/6\sigma F)^2]^2/[0.491T_0(x) + 0.340T_1(x) + 0.263T_{n-1}(x)]$ where T_i are Chebychev polynomials and $x = F/F_{max}$ (Watkin, 1994; *P*rince, 1982)
$(\Delta/\sigma)_{max} = 0.0004$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.43536 (4)	0.14304 (2)	0.77225 (4)	0.0211
N2	1.37324 (11)	0.16961 (8)	1.37176 (15)	0.0161
N3	1.01135 (10)	0.15643 (7)	0.92003 (14)	0.0139

N4	0.87488 (11)	0.09259 (7)	0.60663 (14)	0.0143
N5	0.50436 (12)	0.06571 (8)	0.17912 (15)	0.0177
C6	1.24057 (12)	0.14862 (11)	1.32522 (16)	0.0170
C7	1.19916 (13)	0.16654 (10)	1.13825 (17)	0.0171
C8	1.06443 (12)	0.14127 (10)	1.09743 (16)	0.0153
C9	1.07007 (13)	0.10325 (9)	0.79997 (18)	0.0152
C10	1.00747 (13)	0.11313 (9)	0.61937 (18)	0.0152
C11	0.87922 (12)	0.13626 (10)	0.90503 (17)	0.0156
C12	0.81727 (12)	0.14716 (10)	0.72436 (16)	0.0160
C13	0.81963 (13)	0.10454 (10)	0.42929 (17)	0.0164
C14	0.68553 (14)	0.07703 (10)	0.39558 (18)	0.0191
C15	0.63633 (14)	0.08777 (11)	0.20778 (18)	0.0203
H21	1.3947 (17)	0.1627 (11)	1.486 (2)	0.0196*
H22	1.3889 (16)	0.2220 (12)	1.346 (2)	0.0196*
H23	1.4214 (17)	0.1352 (11)	1.312 (2)	0.0196*
H52	0.4953 (17)	0.0114 (13)	0.197 (2)	0.0212*
H53	0.4781 (17)	0.0758 (12)	0.075 (2)	0.0212*
H61	1.1909 (17)	0.1794 (12)	1.393 (2)	0.0208*
H62	1.2318 (17)	0.0890 (12)	1.348 (2)	0.0208*
H71	1.2089 (16)	0.2262 (12)	1.116 (2)	0.0207*
H72	1.2516 (16)	0.1385 (12)	1.070 (2)	0.0207*
H81	1.0146 (17)	0.1737 (11)	1.169 (2)	0.0185*
H82	1.0563 (16)	0.0845 (12)	1.126 (2)	0.0185*
H91	1.1566 (17)	0.1191 (11)	0.802 (2)	0.0185*
H92	1.0675 (16)	0.0438 (11)	0.833 (2)	0.0185*
H101	1.0199 (16)	0.1713 (12)	0.581 (2)	0.0188*
H102	1.0471 (16)	0.0765 (12)	0.544 (2)	0.0188*
H111	0.8371 (16)	0.1737 (11)	0.983 (2)	0.0188*
H112	0.8672 (17)	0.0789 (12)	0.937 (2)	0.0188*
H121	0.8258 (16)	0.2078 (12)	0.692 (2)	0.0194*
H122	0.7347 (17)	0.1342 (12)	0.721 (2)	0.0194*
H131	0.8670 (17)	0.0735 (12)	0.359 (2)	0.0198*
H132	0.8277 (17)	0.1639 (12)	0.398 (2)	0.0198*
H141	0.6795 (16)	0.0183 (12)	0.423 (2)	0.0230*
H142	0.6324 (18)	0.1086 (12)	0.464 (2)	0.0230*
H151	0.6878 (18)	0.0556 (12)	0.137 (2)	0.0245*
H152	0.6447 (17)	0.1454 (13)	0.174 (2)	0.0245*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.03055 (18)	0.01941 (15)	0.01294 (14)	0.00514 (16)	0.00035 (11)	0.00047 (15)
N2	0.0184 (6)	0.0186 (6)	0.0111 (5)	0.0001 (5)	0.0004 (4)	−0.0006 (4)
N3	0.0122 (5)	0.0168 (6)	0.0130 (5)	−0.0006 (4)	0.0021 (4)	−0.0011 (4)
N4	0.0128 (5)	0.0170 (6)	0.0133 (5)	−0.0009 (4)	0.0019 (4)	−0.0012 (4)
N5	0.0192 (6)	0.0206 (6)	0.0123 (5)	0.0026 (5)	−0.0027 (4)	−0.0017 (4)
C6	0.0159 (6)	0.0222 (7)	0.0130 (5)	−0.0020 (6)	0.0014 (4)	−0.0008 (5)
C7	0.0165 (6)	0.0212 (7)	0.0136 (6)	−0.0013 (5)	0.0019 (5)	0.0026 (5)

C8	0.0151 (6)	0.0163 (6)	0.0142 (5)	−0.0020 (6)	0.0013 (4)	0.0012 (6)
C9	0.0128 (6)	0.0162 (6)	0.0170 (6)	−0.0007 (5)	0.0033 (5)	−0.0020 (5)
C10	0.0131 (6)	0.0184 (6)	0.0149 (6)	−0.0014 (5)	0.0050 (5)	−0.0025 (5)
C11	0.0125 (6)	0.0184 (7)	0.0161 (6)	0.0000 (5)	0.0029 (4)	0.0001 (5)
C12	0.0132 (6)	0.0188 (6)	0.0161 (6)	0.0015 (5)	0.0025 (5)	−0.0005 (5)
C13	0.0173 (6)	0.0197 (7)	0.0125 (6)	−0.0005 (5)	0.0023 (5)	−0.0012 (5)
C14	0.0173 (7)	0.0251 (8)	0.0144 (6)	−0.0022 (5)	−0.0007 (5)	0.0008 (5)
C15	0.0199 (7)	0.0281 (8)	0.0130 (6)	0.0017 (6)	0.0019 (5)	0.0005 (5)

Geometric parameters (Å, º)

N2—C6	1.4884 (18)	C8—H82	0.942 (19)
N2—H21	0.911 (19)	C9—C10	1.512 (2)
N2—H22	0.882 (19)	C9—H91	0.975 (18)
N2—H23	0.927 (19)	C9—H92	0.984 (18)
N3—C8	1.4686 (17)	C10—H101	0.991 (18)
N3—C9	1.4717 (17)	C10—H102	0.972 (18)
N3—C11	1.4676 (17)	C11—C12	1.5122 (19)
N4—C10	1.4744 (18)	C11—H111	1.005 (18)
N4—C12	1.4659 (17)	C11—H112	0.962 (19)
N4—C13	1.4675 (18)	C12—H121	1.009 (18)
N5—C15	1.473 (2)	C12—H122	0.921 (18)
N5—H52	0.88 (2)	C13—C14	1.520 (2)
N5—H53	0.853 (19)	C13—H131	0.941 (19)
C6—C7	1.5162 (19)	C13—H132	0.986 (18)
C6—H61	0.942 (19)	C14—C15	1.523 (2)
C6—H62	0.975 (19)	C14—H141	0.967 (19)
C7—C8	1.5212 (19)	C14—H142	0.978 (19)
C7—H71	0.978 (18)	C15—H151	0.979 (19)
C7—H72	0.943 (18)	C15—H152	0.97 (2)
C8—H81	0.974 (18)		
C6—N2—H21	110.3 (11)	H91—C9—H92	107.6 (15)
C6—N2—H22	111.4 (12)	C9—C10—N4	111.86 (11)
H21—N2—H22	108.1 (16)	C9—C10—H101	108.7 (10)
C6—N2—H23	109.4 (11)	N4—C10—H101	110.8 (10)
H21—N2—H23	109.7 (16)	C9—C10—H102	109.3 (10)
H22—N2—H23	107.9 (16)	N4—C10—H102	109.0 (10)
C8—N3—C9	111.30 (11)	H101—C10—H102	107.0 (14)
C8—N3—C11	108.35 (10)	N3—C11—C12	112.16 (11)
C9—N3—C11	108.55 (11)	N3—C11—H111	109.6 (10)
C10—N4—C12	108.29 (11)	C12—C11—H111	108.7 (10)
C10—N4—C13	109.05 (11)	N3—C11—H112	110.5 (11)
C12—N4—C13	111.47 (11)	C12—C11—H112	107.2 (11)
C15—N5—H52	109.6 (12)	H111—C11—H112	108.7 (15)
C15—N5—H53	108.4 (13)	C11—C12—N4	110.45 (12)
H52—N5—H53	107.6 (17)	C11—C12—H121	107.8 (10)
N2—C6—C7	111.56 (11)	N4—C12—H121	110.5 (10)

N2—C6—H61	110.4 (11)	C11—C12—H122	109.3 (11)
C7—C6—H61	109.1 (11)	N4—C12—H122	109.7 (11)
N2—C6—H62	106.8 (11)	H121—C12—H122	109.1 (15)
C7—C6—H62	109.6 (11)	N4—C13—C14	114.11 (11)
H61—C6—H62	109.4 (15)	N4—C13—H131	107.4 (11)
C6—C7—C8	109.03 (11)	C14—C13—H131	109.2 (11)
C6—C7—H71	109.4 (11)	N4—C13—H132	108.8 (11)
C8—C7—H71	110.1 (11)	C14—C13—H132	110.3 (11)
C6—C7—H72	109.5 (11)	H131—C13—H132	106.8 (15)
C8—C7—H72	112.8 (11)	C13—C14—C15	111.09 (12)
H71—C7—H72	105.8 (15)	C13—C14—H141	109.3 (11)
C7—C8—N3	114.90 (11)	C15—C14—H141	107.6 (11)
C7—C8—H81	109.1 (11)	C13—C14—H142	112.0 (11)
N3—C8—H81	106.0 (11)	C15—C14—H142	108.3 (11)
C7—C8—H82	108.7 (11)	H141—C14—H142	108.4 (16)
N3—C8—H82	110.6 (11)	C14—C15—N5	110.43 (12)
H81—C8—H82	107.4 (15)	C14—C15—H151	109.3 (11)
N3—C9—C10	111.19 (12)	N5—C15—H151	113.7 (11)
N3—C9—H91	109.4 (11)	C14—C15—H152	110.0 (11)
C10—C9—H91	108.5 (11)	N5—C15—H152	107.9 (11)
N3—C9—H92	111.2 (10)	H151—C15—H152	105.3 (15)
C10—C9—H92	108.8 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H23···N5^{i}	0.926 (18)	1.834 (17)	2.7574 (18)	174.7 (17)
N2—H21···Cl1^{i}	0.910 (16)	2.268 (16)	3.1761 (12)	176.3 (17)
N2—H22···Cl1^{ii}	0.882 (19)	2.304 (19)	3.1853 (13)	177.8 (14)
N5—H52···Cl1^{iii}	0.89 (2)	2.58 (2)	3.4104 (13)	155.9 (16)
N5—H53···Cl1^{iv}	0.852 (16)	2.607 (16)	3.4344 (13)	164.3 (16)

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