

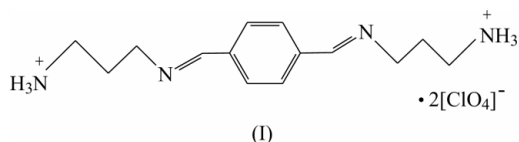
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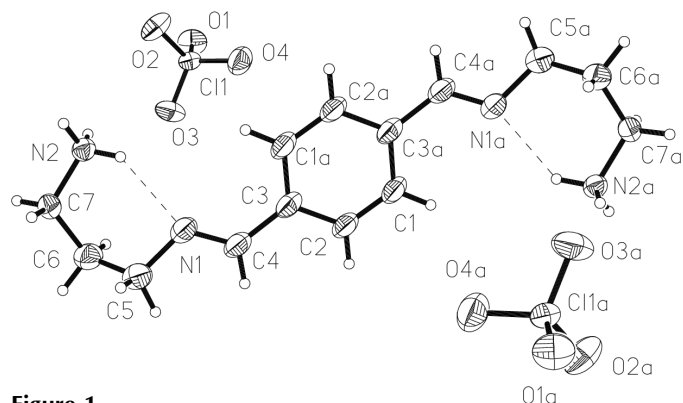
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.049
 wR factor = 0.138
Data-to-parameter ratio = 15.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N,N'*-Bis(aminopropyl)-1,4-benzylidene-
diammonium diperchlorateThe structure determination of the title compound,
[C₁₄H₂₄N₄](ClO₄)₂, indicates that intermolecular hydrogen
bonds between the N atom of the aminopropyl group and an
O atom of the perchlorate result in a two-dimensional layer
structure. The cation has crystallographic inversion symmetry.

Comment

Recently, we have synthesized a series of imidazole-containing
Schiff base compounds, which can be used in crystal engi-
neering or in the synthesis of transition metal complexes to
obtain structural models of some metalloenzymes (Long *et al.*,
1999; Yang *et al.*, 2000, 2001). As a continuation of our studies,
we have isolated the title compound, (I) (Fig. 1), and report
here its preparation and structure. All bond lengths and angles
in (I) are normal (Table 1). The crystal structure of (I) consists
of *N,N'*-bis(aminopropyl)-1,4-benzylidenediammonium
cations and perchlorate anions. The center of the benzene ring
is located at a crystallographic inversion center. The crystal
structure is stabilized by intermolecular N—H···O hydrogen
bonding (Table 2), which produces a two-dimensional network
(Fig. 2).

Experimental

A solution of propane-1,3-diamine (10 mmol) in ethanol (30 ml) was
heated at 333 K and a solution of terephthalaldehyde (5 mmol) in**Figure 1**
View of (I), with the atom-numbering scheme and 50% probability
displacement ellipsoids. The suffix *a* corresponds to symmetry code (i) in
Table 1.

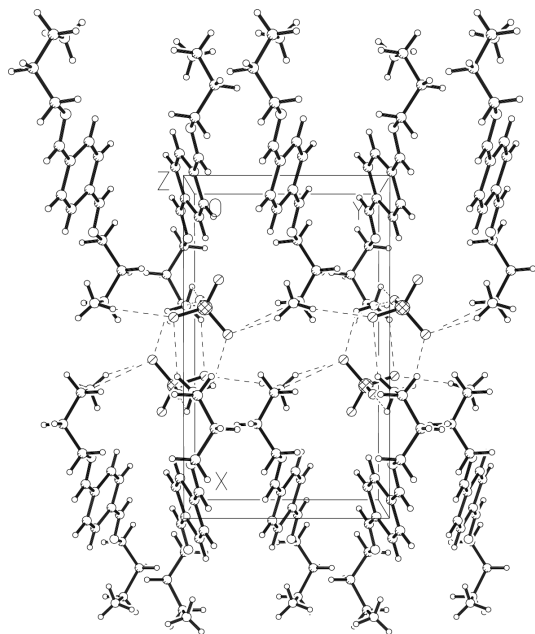


Figure 2
A perspective view of the crystal packing of (I), viewed down the c axis.

methanol (30 ml) added dropwise with stirring over a 2 h period. The mixture was then refluxed for 2.5 h, a solution of sodium perchlorate (10 mmol) in methanol (20 ml) was added and the pH adjusted to ca 6.5. Evaporation at low pressure affording a colorless powder. A crystal suitable for X-ray analysis was obtained by recrystallization from ethyl alcohol over two weeks.

Crystal data

$C_{14}H_{24}N_4^{2+} \cdot 2ClO_4^-$	$D_x = 1.519 \text{ Mg m}^{-3}$
$M_r = 447.27$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4254 reflections
$a = 13.358 (3) \text{ \AA}$	$\theta = 3.1\text{--}52.0^\circ$
$b = 7.864 (2) \text{ \AA}$	$\mu = 0.38 \text{ mm}^{-1}$
$c = 9.313 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 91.714 (3)^\circ$	Block, colorless
$V = 977.9 (4) \text{ \AA}^3$	$0.38 \times 0.16 \times 0.16 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	1915 independent reflections
ω scans	1691 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$R_{\text{int}} = 0.015$
$T_{\text{min}} = 0.868$, $T_{\text{max}} = 0.941$	$\theta_{\text{max}} = 26.0^\circ$
4254 measured reflections	$h = -16 \rightarrow 16$
	$k = -9 \rightarrow 4$
	$l = -10 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.8221P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.138$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.63 \text{ e \AA}^{-3}$
1915 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
128 parameters	Extinction correction: SHELXL97
H-atoms parameters constrained	Extinction coefficient: 0.035 (4)

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—C4	1.261 (4)	C3—C1 ⁱ	1.394 (4)
N1—C5	1.460 (4)	C3—C4	1.456 (4)
N2—C7	1.480 (3)	C5—C6	1.471 (4)
C1—C2	1.356 (4)	C6—C7	1.506 (4)
C2—C3	1.391 (4)		
C4—N1—C5	118.0 (3)	C1 ⁱ —C3—C4	122.3 (3)
C2—C1—C3 ⁱ	120.5 (3)	N1—C4—C3	124.2 (3)
C1—C2—C3	121.3 (3)	N1—C5—C6	112.2 (3)
C2—C3—C1 ⁱ	118.2 (3)	C5—C6—C7	115.8 (3)
C2—C3—C4	119.5 (3)	N2—C7—C6	112.3 (2)
C3 ⁱ —C1—C2—C3	−0.5 (5)		

Symmetry code: (i) $-x, 2-y, 1-z$.

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N2—H2C \cdots O1 ⁱⁱ	0.89	2.42	3.176 (3)	143
N2—H2C \cdots O2 ⁱⁱⁱ	0.89	2.33	2.968 (3)	129
N2—H2B \cdots O1 ⁱⁱⁱ	0.89	2.47	3.199 (3)	139
N2—H2B \cdots O2 ^{iv}	0.89	2.20	2.953 (3)	142
N2—H2A \cdots N1	0.89	2.15	2.879 (3)	139

Symmetry codes: (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $1-x, 1-y, 1-z$; (iv) $x, \frac{3}{2}-y, z-\frac{1}{2}$.

All H atoms were positioned geometrically ($N-H = 0.89$ and $C-H = 0.93$ or 0.97 \AA) and refined using the riding-model approximation, with $U_{\text{iso}}(N) = 1.5U_{\text{eq}}$ and $U_{\text{iso}}(C) = 1.2U_{\text{eq}}$ of the parent atom. The choice between two possible protonation sites, N2 and N1, was made in favor of N2, which provided better R factors, *viz.* $R = 0.049$ and $wR = 0.055$ versus $R = 0.055$ and $wR = 0.061$ for N1 protonation, using data with $F^2 > 2\sigma(F^2)$. The highest peak in the difference Fourier map is located 0.99 \AA from atom C5.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998) and SHELXTL (Sheldrick, 1998); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXL97.

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