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Tris(prop-2-ynyl)ammonium chloride

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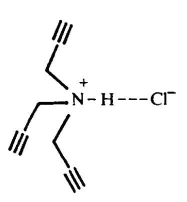
Abstract

The title compound, $C_9H_{10}N^+\cdot Cl^-$, contains two symmetry-independent formula units in the unit cell. The independent organic cations have similar conformations and coordination geometries, whereas the chloride ions have very different coordination spheres. All six independent alkyne groups form $C\equiv C-H\cdots Cl^-$ hydrogen bonds, with $C\cdots Cl^-$ separations in the narrow range 3.46–3.63 Å.

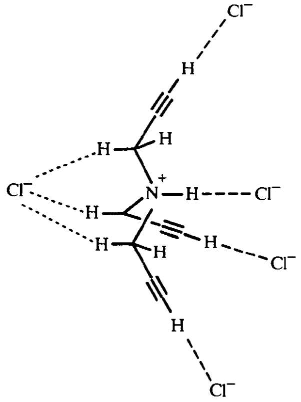
Comment

The title compound, (I), was prepared and its crystal structure determined in order to elucidate the hydrogen-bond interactions. Interest is focused especially on the three alkyne groups of the cation because $C\equiv C-H\cdots X$ hydrogen bonding often plays an important role in terminal alkynes (Steiner, 1998*a*).



(I)

The crystal structure of (I) contains two symmetry-independent formula units, as shown in Fig. 1. The conformations of the two cations are very similar, as are their coordination geometries with the chloride ions (Table 1). The bond lengths and angles are all normal. Each cation is in contact with five chloride ions, as shown in the Scheme below. The dominant $N^+-H\cdots Cl^-$ hydrogen bond and the three $C\equiv C-H\cdots Cl^-$ hydrogen bonds all have distances in the usual ranges (Steiner, 1998*b*). In addition, each cation forms a short contact to a chloride ion positioned opposite the N—H vector, leading to three $C-H\cdots Cl^-$ contacts. The two corresponding $N^+\cdots Cl^-$ separations are $N1\cdots Cl2 = 3.760\,(2)$ and $N2\cdots Cl2 = 3.832\,(2)$ Å, which are in the distance range typical of this interaction (Desiraju & Steiner, 1999).



In structures with more than one molecule or formula unit per asymmetric crystal unit ($Z' > 1$), it is of interest to see if this is associated with different conformations and intermolecular interactions. In (I), the conformation and immediate surroundings of the two organic cations are almost identical, but the two chloride ions have

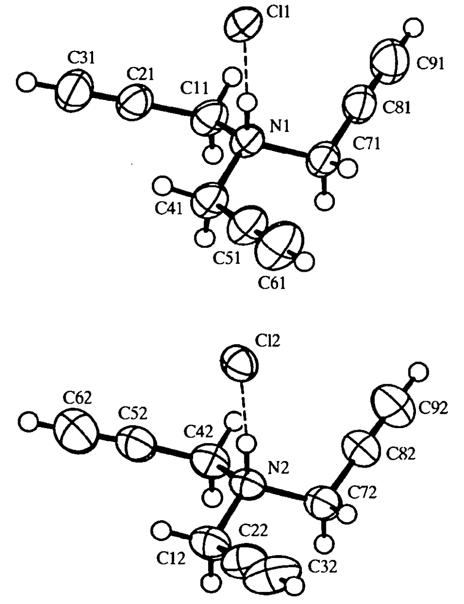


Fig. 1. The molecular structure of the two symmetry-independent formula units in (I), showing 50% probability displacement ellipsoids.

very different coordination spheres (Fig. 2). Cl1 accepts one $N^+—H\cdots Cl^-$ and five $C≡C—H\cdots Cl^-$ hydrogen bonds. Cl2, on the other hand, accepts one $N^+—H\cdots Cl^-$ and only one $C≡C—H\cdots Cl^-$ hydrogen bond. In addition, Cl2 is involved in two $N^+(CH_2)_3\cdots Cl^-$ contacts, neither of which is formed by Cl1. This makes (I) an interesting example of a crystal structure where the fact that $Z' = 2$ is not reflected in the molecular conformation nor in the intermolecular interactions of the largest constituent of the structure, but only in the coordination of the halide ions.

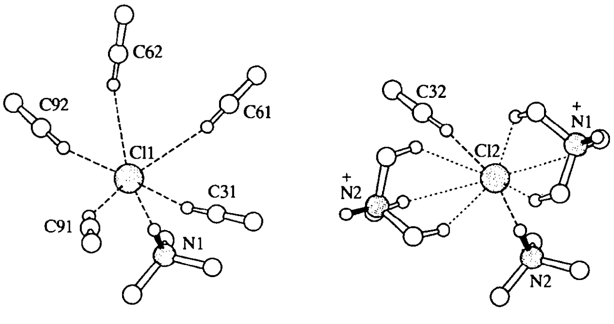


Fig. 2. The coordination of the two symmetry-independent chloride ions in (I), shown in the same projection with respect to the $N^+—H\cdots Cl^-$ hydrogen bonds.

Experimental

Crystals were obtained by slow evaporation of a solution of tris(prop-2-ynyl)amine (Aldrich) in dilute HCl.

Crystal data

$C_9H_{10}N^+\cdot Cl^-$
$M_r = 167.63$
Monoclinic
$P2_1/c$
$a = 13.730\ (5)$ Å
$b = 11.334\ (3)$ Å
$c = 13.848\ (5)$ Å
$\beta = 109.15\ (2)°$
$V = 2035.7\ (14)$ Å^3
$Z = 8$
$D_x = 1.094$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 36 reflections
$\theta = 4.4–16.8°$
$\mu = 0.317$ mm^{-1}
$T = 293\ (2)$ K
Block
0.3 × 0.3 × 0.3 mm
Colourless

Data collection

Nonius Kappa CCD diffractometer
ψ ($\kappa = 0$) and ω ($\kappa = 147.78$) scans
Absorption correction: none
11 299 measured reflections
4661 independent reflections
3164 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\rm max} = 27.5°$
$h = -15 \rightarrow 17$
$k = -14 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.130$
$S = 1.118$
4661 reflections
225 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.8112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.218$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.189$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$N1—H1\cdots Cl1$	0.97 (3)	2.03 (3)	3.001 (2)	177 (2)
$N2—H2\cdots Cl2$	1.03 (3)	1.99 (3)	3.015 (2)	177 (2)
$C31—H31\cdots Cl1^{\rm i}$	0.97	2.70	3.557 (3)	153
$C61—H61\cdots Cl1^{\rm ii}$	0.97	2.74	3.625 (4)	159
$C91—H91\cdots Cl1^{\rm iii}$	0.97	2.66	3.508 (3)	152
$C32—H32\cdots Cl2^{\rm iv}$	0.97	2.57	3.459 (3)	161
$C62—H62\cdots Cl1^{\rm v}$	0.97	2.74	3.634 (4)	161
$C92—H92\cdots Cl1^{\rm vi}$	0.97	2.60	3.482 (3)	160
$C11—H11B\cdots Cl2$	0.97	2.71	3.554 (3)	148
$C41—H41B\cdots Cl2$	0.97	2.85	3.668 (3)	143
$C71—H71B\cdots Cl2$	0.97	2.71	3.554 (3)	146
$C12—H12B\cdots Cl2^{\rm vii}$	0.97	2.89	3.720 (3)	144
$C42—H42B\cdots Cl2^{\rm vii}$	0.97	2.78	3.640 (3)	148
$C72—H72B\cdots Cl2^{\rm vii}$	0.97	2.76	3.621 (3)	148

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

The area-detector data are 100% complete to $2\theta = 55°$. H atoms bonded to C were refined using a riding model with a C—H bond length of 0.97 Å, and their isotropic displacement parameters were allowed to vary. H atoms bonded to N atoms were located in difference Fourier calculations and refined isotropically. All H-atom displacement parameters refined to realistic values.

Cell refinement: *EVAL*14 (Duisenberg, 1998). Data reduction: *EVAL*14. Program(s) used to solve structure: *SHELXS*86

(Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1347). Services for accessing these data are described at the back of the journal.

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(±)-(3′aα,4′α,6′aα)-3′,3′a,4′,6′a-Tetrahydro-5,5,*N*,*N*-tetramethylspiro[1,3-dioxane-2,2′(1′*H*)-pentalene]-4′-acetamide, a triclinic crystal structure with local monoclinic pseudosymmetry

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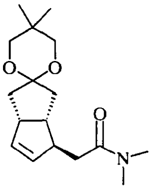
Abstract

The crystal packing in the title compound, $C_{17}H_{27}NO_3$, shows layers with local pseudo-monoclinic symmetry. The two independent molecules are related by local non-crystallographic glide planes and screw axes. Adjacent layers are displaced by a distance of about 1.61 Å in the *a* direction, resulting in an overall triclinic symmetry of the structure.

Comment

The structure of the title compound, (I), contains two crystallographically independent molecules (1 and 2). The geometries of both molecules are very similar. Inspection of the fractional coordinates shows that both independent molecules are related by a local non-crystallographic glide plane almost perpendicular to the *a* axis, with a translation vector of 0.5**b**. The equation of this pseudo-glide plane in crystal coordinates is approximately $9.48x + 0y - 1.61z = 1.56$. The angle between the *a* axis and the normal to this pseudo-glide plane is only 0.6°. The pseudo-glide plane is continuous in the crystallographic *b* direction. In the *c* direction, however, the pseudo-glide plane only acts in one unit cell and shows a shift of about one sixth of the length of the *a* axis between adjacent cells. The pseudo-glide symmetry can be combined with the crystallographic inversion center to give an additional pseudo-twofold screw axis parallel to **a** and displaced from the origin by ±**b**/4 and +**c**/2. These pseudosymmetry elements can be easily identified using the program *BUNYIP* (Hester & Hall, 1996) or by a combined coordinate midpoint and translation analysis as described by Kálmán & Argay (1998).



(I)

The crystal packing (Fig. 2) shows layers parallel to the *ab* plane with approximate monoclinic symmetry. The symmetry of the layer corresponds to structural class $P_{l(XY)}2_1/b$, $Z = 4\,(1)$, using the nomenclature of Zorky (1996). Adjacent layers are displaced by a distance of about 1.61 Å along the *a* axis. This displacement results in a triclinic symmetry of the three-dimensional crystal structure. Without this displacement the structure would be monoclinic with space group $P2_1/b$ (*a* axis unique), but with intermolecular H···H contacts between adjacent layers as short as 2.04 Å, which, obviously, is too short. A similar pseudo-symmetric structure has been observed by Bats *et al.* (1999). The five-membered rings are *cis*-connected about the C1—C5 and the C18—C22 bonds. These bonds have lengths of 1.563 (2) Å and are slightly lengthened with respect to a normal C—C single bond of 1.54 Å. The cyclopentene ring has an envelope con-