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# *N*,*N*,*N*′-Trimethylethylenediammonium dichloride

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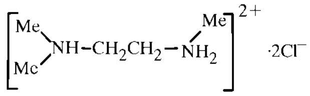
The title compound, $C_5H_{16}N_2^{2+}\cdot 2Cl^-$, was isolated as a by-product from the reaction between trimethylethylenediamine and germanium tetrachloride in the presence of triethylamine. The asymmetric unit contains two cations, one in the *gauche* and the other in the *trans* conformation; these conformations are stabilized by hydrogen-bonding interactions between the N—H moieties and the chloride anions.

## Comment

The use of polydentate N-donor ligands to supplant the classic cyclopentadienyl ($C_5H_5$) ligand in the development of new catalytic metal complexes (*p*-block, early *d*-block and *f*-block) is of great topical interest (*e.g.* Schrock, 1997; Gade, 2000; Skinner *et al.*, 2000). In the search for a potential catalyst system based on $[Ge^{IV}—N]_x$, we have studied the reaction system $GeCl_4/Me_2N—CH_2—CH_2—N(H)Me/Et_3N/CH_3CN$. The title compound, (I), was obtained as a side product (12% yield based on the parent amine), as colourless needle crystals.



$$\left[\mathrm{Me_2NH{-}CH_2CH_2{-}NH_2Me}\right]^{2+}\cdot 2Cl^-$$

(I)

In gross terms, both of the N centres of the parent amine ligand have been protonated, with limited hydrolysis of the Ge—Cl bond(s) (from traces of water impurities) as the most likely source of the acid (HCl).

The asymmetric unit of the title compound is illustrated in Fig. 1. All bond lengths and angles are largely unremarkable. The two cations in the asymmetric unit differ in that one has a *trans* conformation, whilst the other approximates to a *gauche* arrangement (see torsion angles in Table 1). These differences have a dramatic effect upon the hydrogen-bonding interactions within the lattice. The cation with the *gauche* conformation is able to mimic a bidentate ligand by forming two hydrogen bonds from opposite ends of the molecule to a single chloride ion (*i.e.* to Cl1); a third hydrogen bond links this cation to a second chloride ion. In contrast, the '*trans*' cation is only able to hydrogen bond to three separate chloride ions (see Fig. 1). All of these hydrogen-bonding interactions are conventional two-centre (*i.e.* linear) interactions, with N—H···Cl angles (Table 2) ranging from 163 to 179°. The hydrogen bonding described above does not extend in a polymeric sense throughout the lattice, but is confined to small clusters corresponding to the asymmetric unit. The most important lattice forces are undoubtedly ionic interactions.

The cationic component of this structure does not appear to have been the subject of any previous X-ray investigation. The closest comparisons involve the ethylenediammonium (as the citrate; Gavrushenko *et al.*, 1977) and *N*,*N*,*N*′,*N*′-tetramethylethylenediammonium cations. The ethylenediammonium unit was observed to have a *gauche* conformation [with a torsion angle of 71.4 (2)°], whilst the two reported structures [with $Br^-$ (Annan *et al.*, 1991) and with $I_3^-$ (Robertson *et al.*, 1996)] involving the *N*,*N*,*N*′,*N*′-tetramethylethylenediammonium ion both have *trans* conformations. In all of these structures, including the title compound, hydrogen bonding appears to be important in the stabilization of the observed conformations.

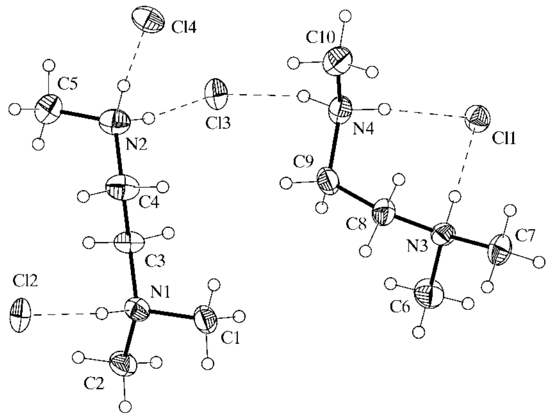


**Figure 1**
View of the asymmetric unit of (I) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radii.

## Experimental

A solution of $Me_2NCH_2CH_2N(H)Me$ (2.33 g, 22.8 mmol) in $CH_3CN$ (10 ml) was added dropwise to a chilled (273 K) solution of $GeCl_4$ (2.51 g, 11.7 mmol) in $CH_3CN$ (10 ml). After stirring the mixture for 15 min, a solution of $Et_3N$ (5.0 g, 49.4 mmol) in $CH_3CN$ was added and the resulting solution was warmed at 323 K for 3 h. The solution was filtered whilst still hot, concentrated (*ca* 50% by volume) and then cooled to ice-cold temperature whereupon colourless needle crystals slowly deposited (yield 0.96 g, 12%). Found: C 34.1, H 9.1, N 15.8%; calculated for $C_5H_{16}Cl_2N_2$: C 34.3, H 9.2, N 16.0%.

*Crystal data*

$C_5H_{16}N_2^{2+}\cdot 2Cl^-$
$M_r$ = 350.20
Monoclinic, $P2_1/c$
$a$ = 11.1419 (12) Å
$b$ = 20.2089 (14) Å
$c$ = 8.7321 (9) Å
$\beta$ = 110.644 (4)°
$V$ = 1839.9 (3) Å$^3$
$Z$ = 4
$D_x$ = 1.264 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2396 reflections
$\theta$ = 1.95–25.00°
$\mu$ = 0.636 mm$^{-1}$
$T$ = 180 (2) K
Needle, colourless
0.24 × 0.12 × 0.12 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.862$, $T_{\max} = 0.928$
8986 measured reflections
3236 independent reflections
1857 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.084$
$\theta_{\max} = 25°$
$h = -13 \rightarrow 12$
$k = -24 \rightarrow 18$
$l = -10 \rightarrow 10$
Intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.096$
$S = 0.980$
3236 reflections
193 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.30$ e Å$^{-3}$

**Table 1**
Selected torsion angles (°).

| | | | |
|---|---|---|---|
| N1—C3—C4—N2 | 179.9 (3) | N3—C8—C9—N4 | −78.9 (4) |

The H atoms linked directly to N atoms were located from electron-density maps, but then restrained under refinement to have equal N—H bond lengths; all other H atoms were added at calculated positions and refined using a riding model, with C—H = 0.98–0.99 Å. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of their parent atoms.

Data collection: *SMART* (Siemens, 1994*a*); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL*/*PC* (Siemens, 1994*b*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC*; software used to prepare material for publication: *SHELXTL*/*PC*.

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1′···Cl2 | 1.02 (2) | 2.05 (2) | 3.062 (3) | 171 (3) |
| N2—H2′···Cl4 | 1.04 (2) | 1.97 (2) | 3.009 (4) | 176 (4) |
| N2—H2″···Cl3 | 1.03 (2) | 2.11 (2) | 3.134 (4) | 171 (4) |
| N3—H3′···Cl1 | 1.01 (2) | 2.07 (2) | 3.080 (3) | 179 (3) |
| N4—H4′···Cl3 | 1.03 (2) | 2.11 (2) | 3.110 (3) | 163 (3) |
| N4—H4″···Cl1 | 1.02 (2) | 2.11 (2) | 3.132 (3) | 173 (3) |

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1121). Services for accessing these data are described at the back of the journal.

## References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Annan, T. A., Chadha, R. K. & Tuck, D. G. (1991). *Acta Cryst.* C**47**, 151–153.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Gade, L. H. (2000). *J. Chem. Soc. Chem. Commun.* pp. 173–181.
Gavrushenko, N., Carrell, H. L., Stallings, W. C. & Glusker, J. P. (1977). *Acta Cryst.* B**33**, 3936–3939.
Robertson, K. N., Cameron, T. S. & Knop, O. (1996). *Can. J. Chem.* **74**, 1572–1591.
Schrock, R. R. (1997). *Acc. Chem. Res.* **30**, 9–16.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994*a*). *SMART Software Reference Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*b*). *SHELXTL*/*PC Reference Manual.* Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1995). *SAINT*. Version 4.021. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Skinner, M. E. G., Cowhig, D. A. & Mountford, P. (2000). *J. Chem. Soc. Chem. Commun.* pp. 1167–1168.

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