Acta Crystallographica Section C
**Crystal Structure
Communications**
ISSN 0108-2701

N,*N*,*N'*,*N'*-Tetramethylethylenediammonium dichloride

Mehmet Kabak, Yalçin Elerman, Canan Ünaleroglu, Yüksel Mert and T. Nuri Durlu

Copyright © International Union of Crystallography

This paper is published electronically. It meets the data-validation criteria for publication in *Acta Crystallographica Section C*. The submission has been checked by a Section C Co-editor though the text in the "Comments" section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

N,*N*,*N*′,*N*′-Tetramethylethylenediammonium dichloride

Mehmet Kabak,[a]* Yalçin Elerman,[a] Canan Ünaleroglu,[b] Yüksel Mert[b] and T. Nuri Durlu[c]

[a]Department of Engineering Physics, Faculty of Sciences, University of Ankara, 06100 Beşevler, Ankara, Turkey, [b]Department of Chemistry, Faculty of Art and Sciences, University of Hacettepe, 06532 Beytepe, Ankara, Turkey, and [c]Department of Physics, Faculty of Art and Sciences, Kırıkkale University, 71450 Yahşihan, Kırıkkale, Turkey
Correspondence e-mail: kabak@science.ankara.edu.tr

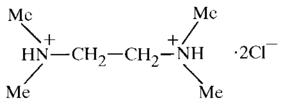
Received 21 December 1999
Accepted 18 January 200

Data validation number: IUC0000016

The structure of the title compound, $C_6H_{18}N_2^{2+}.2Cl^-$, has been determined and has a centre of symmetry. The molecule has strong intermolecular hydrogen bonding between each Cl^- and an N—H bond [Cl···N = 3.012 (3) Å].

Comment

The title compound, (I), was formed as an unexpected by-product of the reaction of L-ascorbic acid (vitamin C) with tmed (tmed = *N*,*N*,*N*′,*N*′-tetramethylethanediamine) in CH_2Cl_2 and also from the reaction of ascorbic acid with tmed and copper(II) methoxide in CH_2Cl_2. The reaction of $SnCl_2$with tmen and tetrachloro-*o*-quinone to form $Cl_4C_6O_2SnCl_2$.tmen complexes in methanol has been reported to give *N*,*N*,*N*′,*N*′-tetramethylethylenediamonium dichloride as a by-product (Annan *et al.*, 1987). However the structure of this compound has not been determined by X-ray crystallography. A related compound of *N*,*N*,*N*′,*N*′-tetamethylenediamonium dibromide was formed as a by-product during the crystallization of the compound $Ph_3SnInBr_2$.tmen in a mixture of $CHCl_3/CH_3OH$ and has been determined crystallograplically (Annan *et al.*, 1991; Annan & Tuck, 1987).



(I)

The geometry around each N atom is essentially tetrahedral and the average C—N—C bond angle of 111.3 (9)° is nearly the same as in the previous work of Annan *et al.* (1991) [111.7 (4)°]. The C—C and average C—N bond distances of 1.526 (5) and 1.491 (7) Å, respectively, compare favourably with those in the compound of Annan *et al.* (1991), where corresponding values are 1.57 (1) and 1.467 (7) Å. The N1—C1 bond length [1.501 (3) Å] is longer than in the previous work of Annan *et al.* (1991) [1.442 (7) Å].

A strong intermolecular hydrogen bond occurs between the Cl1(2 −*x*, 2-*y*, 2-*z*) and N1 atoms, the H atom being essentially bonded to the N1 atom. The Cl1···N1, Cl1···H1 and N1—H1 distances are 3.012 (3), 2.00 (4) and 1.02 (3) Å, respectively. The sum of the van der Waals radii of Cl and N (3.30 Å) is significantly longer than the intramolecular Cl···N hydrogen-bond length (Bondi, 1964).

Experimental

The title compound was prepared by magnetic stirring of L-ascorbic acid (1 g, 5.68 mmol) and tmed (0.667 g, 5.68 mmol) in 50 ml CH_2Cl_2 for 48 h under N_2 to give a yellow solution and a gummy solid. The precipitate was separated by filtration. The filtrate was evaporated to dryness at room temperature giving a mixture of white and yellow powder. Crystallization of this powder from a mixture of methanol/acetone (50:50, *v*/*v*) gave colourless single crystals of the title compound.

Crystal data

$C_6H_{18}N_2^{2+}\cdot 2Cl^-$
$M_r = 189.12$
Triclinic, $P\bar{1}$
$a = 6.1467\ (12)$ Å
$b = 8.075\ (3)$ Å
$c = 5.770\ (2)$ Å
$\alpha = 101.93\ (3)°$
$\beta = 108.27\ (2)°$
$\gamma = 76.65\ (2)°$
$V = 261.77\ (14)$ Å^3
$Z = 1$
$D_x = 1.200$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.00–14.39°$
$\mu = 0.564$ mm^{-1}
$T = 293\ (2)$ K
Prismatic, colorless
0.50 × 0.30 × 0.10 mm

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.927$, $T_{max} = 0.992$
1257 measured reflections
1149 independent reflections
1001 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 27.49°$
$h = 0 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -7 \rightarrow 7$
3 standard reflections every 150 reflections intensity decay: 4.72%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.127$
$S = 1.355$
1149 reflections
49 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.1206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.481$ e Å^{-3}
$\Delta\rho_{min} = -0.331$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.486 (3)	N1—H1	1.02 (3)
N1—C3	1.487 (3)	C1—C1^i	1.526 (5)
N1—C1	1.501 (3)		
C1—N1—C2	112.52 (19)	C2—N1—C3	110.7 (2)
C1—N1—C3	110.6 (2)	N1—C1—C1^i	110.3 (2)

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

All the H atoms (except the H1 atom which was fixed from the difference Fourier to the N atom) were fixed at ideal positions with

e66 Mehmet Kabak *et al.* • $C_6H_{18}N_2^{2+}\cdot 2Cl^-$ *Acta Cryst.* (2000). C**56**, e66–e67

isotropic displacement parameters $1.5U_{\rm eq}$ of the related C and N atoms.

Data collection: *MSC/AFC Diffractomer Control Software* (MSC, 1994); cell refinement: *MSC/AFC Diffractomer Control Software*; data reduction: *teXsan* (MSC, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

References

Annan, T. A., Chadha, R. K. & Tuck, D. G. (1991). *Acta Cryst.* C**47**, 151–153.
Annan, T. A., Chadha, R. K., Tuck, D. G. & Watson, K. D. (1987). *Can. J. Chem.* **65**, 2670–2676.
Annan, T. A. & Tuck, D. G. (1987). *J. Organomet. Chem.* **325**, 83–89.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Molecular Structure Corporation (1994). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1997). *teXsan for Windows*. Version 1.03. Version 1.03. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.