Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A polymorph of tetraethylammonium chloride

Yong Yan, Joel T. Mague and James P. Donahue*

Department of Chemistry, Tulane University, 6400 Freret Street, New Orleans, LA 70118-5698, USA
Correspondence e-mail: donahue@tulane.edu

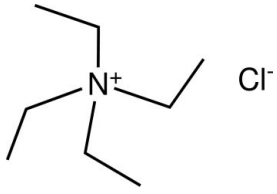
Received 11 May 2009; accepted 28 May 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.083; data-to-parameter ratio = 24.2.

The structure of the title compound, $C_8H_{20}N^+\cdot Cl^-$, is compared with a polymorph that was described earlier in the same space group. Differences in the conformations of the ethyl groups of the cation exist between the polymorphs. This study is given here in order to provide additional unit-cell data for use in qualitative identification of crystalline samples obtained in syntheses in which $Et_4N^+\cdot Cl^-$ is either used or generated.

Related literature

A polymorph with three molecules in the asymmetric unit was earlier solved in the $P2_1/n$ setting of this same space group (Staples, 1999). A discussion of crystal growth conditions that can affect the occurrence of polymorphs has been given by Hulliger (1994). For descriptions of chemistry involving tetraethylammonium chloride, see: McCleverty *et al.* (1967); Lorber *et al.* (1998); Donahue *et al.* (1998).



Experimental

Crystal data

$C_8H_{20}N^+\cdot Cl^-$
M_r = 165.70
Monoclinic, $P2_1/c$
a = 8.429 (2) Å
b = 8.109 (2) Å
c = 14.499 (4) Å
β = 91.378 (3)°
V = 990.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 100 K
0.20 × 0.14 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*b*)
T_{min} = 0.876, T_{max} = 0.963
8314 measured reflections
2302 independent reflections
2038 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.083
S = 1.03
2302 reflections
95 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*a*); molecular graphics: *SHELXTL* (Sheldrick, 2008*a*); software used to prepare material for publication: *SHELXTL*.

JTM gratefully acknowledges Tulane University for support of the Tulane Crystallography Laboratory.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2163).

References

Bruker (2008). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2009). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Donahue, J. P., Goldsmith, C. R., Nadiminti, U. & Holm, R. H. (1998). *J. Am. Chem. Soc.* **120**, 12869–12881.
Hulliger, J. (1994). *Angew. Chem. Int. Ed. Engl.* **33**, 143–162.
Lorber, C., Donahue, J. P., Goddard, C. A., Nordlander, E. & Holm, R. H. (1998). *J. Am. Chem. Soc.* **120**, 8102–8112.
McCleverty, J. A., Atherton, N. M., Locke, J., Wharton, E. J. & Winscom, C. J. (1967). *J. Am. Chem. Soc.* **89**, 6082–6092.
Sheldrick, G. M. (2008*a*). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2008*b*). *SADABS*. University of Göttingen, Germany.
Staples, R. J. (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 231–232.

supporting information

Acta Cryst. (2009). E**65**, o1491 [doi:10.1107/S160053680902042X]

A polymorph of tetraethylammonium chloride

Yong Yan, Joel T. Mague and James P. Donahue

S1. Comment

Tetraethylammonium chloride, $Et_4N^+Cl^-$ (Scheme 1), is frequently employed in inorganic synthesis as a convenient source of soluble countercations for anionic metal species. For instance, $Et_4N^+Cl^-$ is added to the reaction mixture in which $Na_2[Fe_2(mnt)_4]$ is formed from $Na_2(mnt)$ and $FeCl_3$ (mnt = $(CN)_2C_2S_2$(2-) = maleonitriledithiolate(2-)), thereby providing a metallodithiolene product that has useful solubility in common organic solvents (McCleverty *et al.*, 1967). In other instances, $Et_4N^+Cl^-$ is generated as a byproduct of synthesis, as in the preparation of $[Et_4N][M(OSiMe_3)(bdt)_2]$ (M = Mo or W; bdt = benzene-1,2-dithiolate(2-)) by silylation of the corresponding oxo bis(dithiolene) dianion (Lorber *et al.*, 1998; Donahue *et al.*, 1998). The frequency with which $Et_4N^+Cl^-$ is used or otherwise encountered in inorganic synthesis, and the ease with which crystalline samples may be occluded with colored impurities that obscure their identity, make desirable the availability of complete crystallographic data for this compound as a means for qualitatively identifying it and avoiding needless data collections.

White parallelpiped crystals of $Et_4N^+Cl^-$ grew without disorder (Fig. 1) in monoclinic space group $P2_1/c$ with only one formula unit in the asymmetric unit and a Z value of 4 (Fig. 2). A view of the tetraethylammonium cation that is approximately orthogonal to a mean plane projection of the C and N atoms shows a propeller-like disposition of the ethyl groups around the central N atom (Fig. 1).

S2. Experimental

White parallelpiped crystals of $Et_4N^+Cl^-$ grew by diffusion of Et_2O vapor into an acetonitrile solution under a dry, N_2 atmosphere.

S3. Refinement

H atoms were placed in calculated positions (C—H = 0.98–0.99 Å) and included as riding contributions with isotropic displacement parameters 1.2–1.5 times those of the attached C atoms.

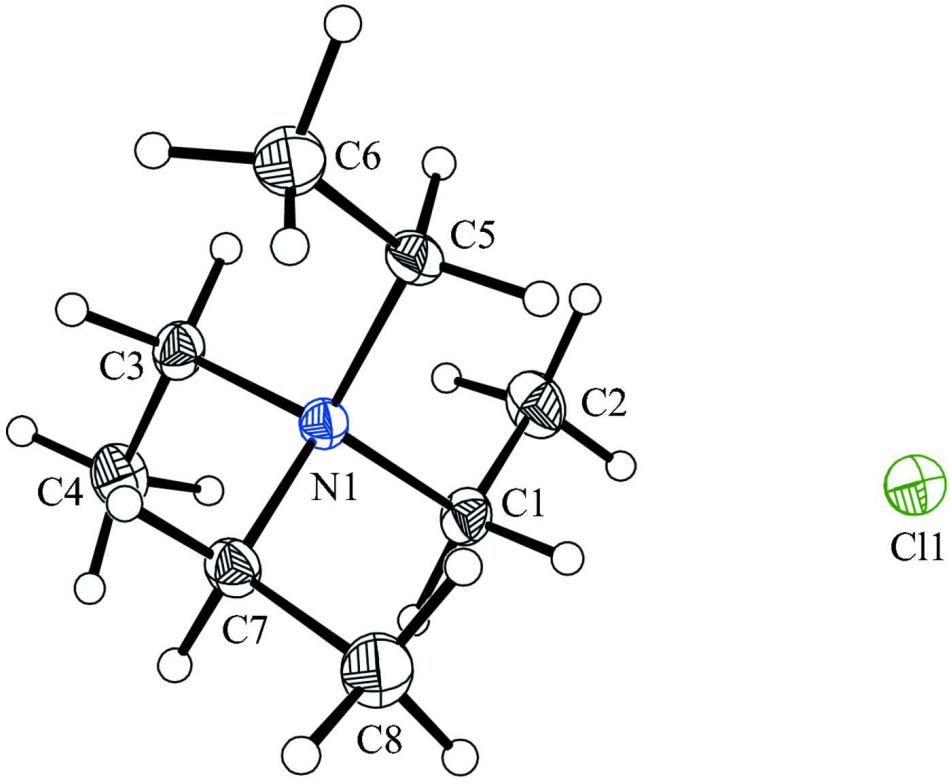


Figure 1

$Et_4N^+Cl^-$ shown with 50% probability ellipsoids.

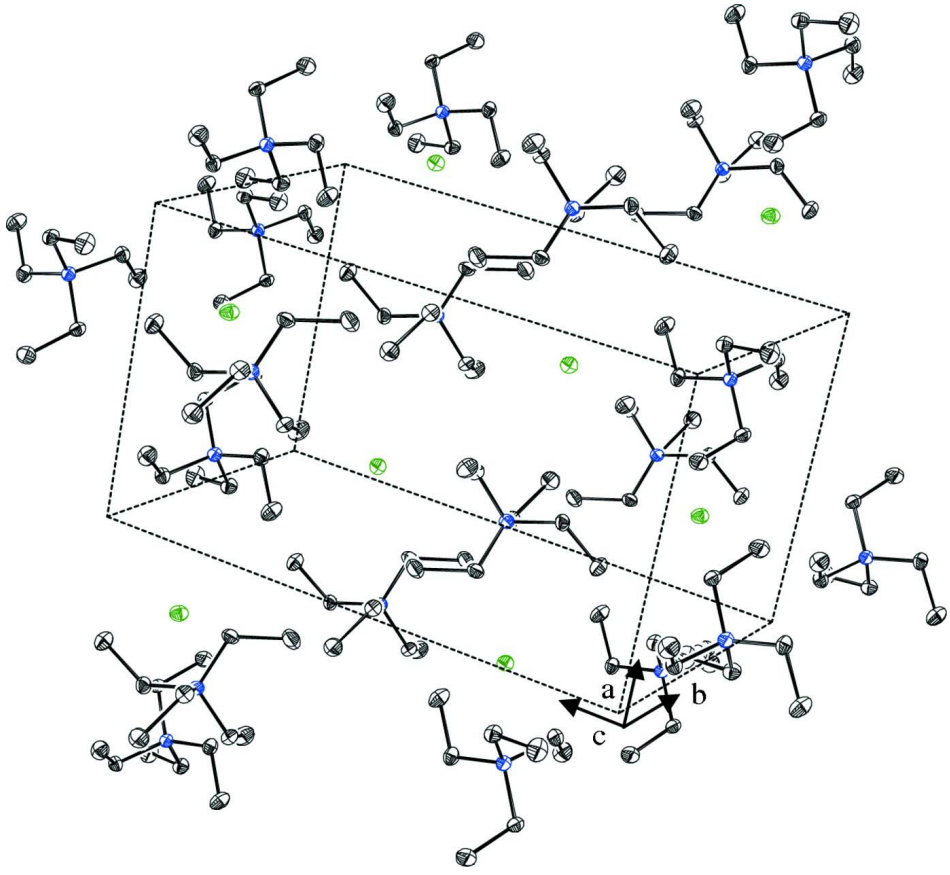


Figure 2

Unit cell of $Et_4N^+Cl^-$ in $P2_1/c$.

Tetraethylammonium chloride

Crystal data

$C_8H_{20}N^+\cdot Cl^-$
$M_r = 165.70$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.429\ (2)$ Å
$b = 8.109\ (2)$ Å
$c = 14.499\ (4)$ Å
$\beta = 91.378\ (3)°$
$V = 990.7\ (4)$ Å^3
$Z = 4$

$F(000) = 368$
$D_x = 1.111$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5930 reflections
$\theta = 2.4–28.5°$
$\mu = 0.32$ mm^{-1}
$T = 100$ K
Parallelepiped, colourless
0.20 × 0.14 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008a)
$T_{min} = 0.876$, $T_{max} = 0.963$

8314 measured reflections
2302 independent reflections
2038 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.8°$, $\theta_{min} = 2.4°$
$h = -10\rightarrow10$
$k = -10\rightarrow10$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.083$
$S = 1.03$
2302 reflections
95 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.2706P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. The diffraction data were collected in three sets of 606 frames (0.3 °. width in ω) at φ = 0, 120 and 240 °. A scan time of 30 sec/frame was used.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.24449 (3)	0.49267 (3)	0.660880 (18)	0.02030 (10)
N1	0.25555 (10)	0.10663 (10)	0.86208 (6)	0.01401 (19)
C1	0.30638 (13)	0.09217 (13)	0.76286 (7)	0.0177 (2)
H1A	0.4027	0.0225	0.7610	0.021*
H1B	0.3350	0.2032	0.7402	0.021*
C2	0.18066 (15)	0.01931 (15)	0.69818 (8)	0.0252 (3)
H2A	0.1584	−0.0945	0.7166	0.038*
H2B	0.2191	0.0203	0.6349	0.038*
H2C	0.0833	0.0850	0.7012	0.038*
C3	0.19934 (13)	−0.05808 (13)	0.90007 (8)	0.0182 (2)
H3A	0.1879	−0.0473	0.9676	0.022*
H3B	0.0930	−0.0825	0.8730	0.022*
C4	0.30819 (14)	−0.20341 (14)	0.88139 (8)	0.0232 (2)
H4A	0.3011	−0.2321	0.8157	0.035*
H4B	0.2758	−0.2983	0.9183	0.035*
H4C	0.4178	−0.1735	0.8981	0.035*
C5	0.11843 (12)	0.22872 (13)	0.86575 (7)	0.0176 (2)
H5A	0.1432	0.3248	0.8265	0.021*
H5B	0.0218	0.1756	0.8393	0.021*
C6	0.08328 (14)	0.28976 (15)	0.96222 (8)	0.0251 (3)
H6A	0.0840	0.1963	1.0051	0.038*
H6B	−0.0213	0.3426	0.9619	0.038*
H6C	0.1644	0.3697	0.9819	0.038*

C7	0.39616 (12)	0.16349 (13)	0.92106 (7)	0.0167 (2)
H7A	0.4806	0.0789	0.9185	0.020*
H7B	0.3629	0.1720	0.9859	0.020*
C8	0.46431 (14)	0.32803 (14)	0.89185 (8)	0.0243 (3)
H8A	0.5208	0.3142	0.8340	0.036*
H8B	0.5382	0.3684	0.9400	0.036*
H8C	0.3780	0.4078	0.8827	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01788 (16)	0.02294 (16)	0.02008 (16)	−0.00039 (9)	0.00052 (11)	0.00271 (9)
N1	0.0142 (4)	0.0146 (4)	0.0132 (4)	0.0006 (3)	0.0001 (3)	0.0003 (3)
C1	0.0206 (5)	0.0200 (5)	0.0125 (5)	0.0022 (4)	0.0023 (4)	−0.0006 (4)
C2	0.0280 (6)	0.0301 (6)	0.0174 (6)	0.0023 (5)	−0.0054 (5)	−0.0035 (4)
C3	0.0202 (5)	0.0158 (5)	0.0185 (5)	−0.0023 (4)	0.0002 (4)	0.0027 (4)
C4	0.0281 (6)	0.0158 (5)	0.0256 (6)	0.0021 (4)	−0.0023 (5)	0.0008 (4)
C5	0.0158 (5)	0.0183 (5)	0.0187 (5)	0.0045 (4)	0.0008 (4)	0.0006 (4)
C6	0.0265 (6)	0.0276 (6)	0.0214 (6)	0.0090 (5)	0.0053 (4)	−0.0007 (4)
C7	0.0164 (5)	0.0182 (5)	0.0153 (5)	−0.0009 (4)	−0.0029 (4)	−0.0002 (4)
C8	0.0249 (6)	0.0224 (6)	0.0253 (6)	−0.0062 (5)	−0.0035 (5)	0.0013 (4)

Geometric parameters (Å, º)

N1—C1	1.5153 (13)	C4—H4B	0.9800
N1—C7	1.5165 (13)	C4—H4C	0.9800
N1—C5	1.5238 (13)	C5—C6	1.5197 (16)
N1—C3	1.5246 (13)	C5—H5A	0.9900
C1—C2	1.5178 (16)	C5—H5B	0.9900
C1—H1A	0.9900	C6—H6A	0.9800
C1—H1B	0.9900	C6—H6B	0.9800
C2—H2A	0.9800	C6—H6C	0.9800
C2—H2B	0.9800	C7—C8	1.5170 (15)
C2—H2C	0.9800	C7—H7A	0.9900
C3—C4	1.5219 (15)	C7—H7B	0.9900
C3—H3A	0.9900	C8—H8A	0.9800
C3—H3B	0.9900	C8—H8B	0.9800
C4—H4A	0.9800	C8—H8C	0.9800
C1—N1—C7	108.90 (8)	C3—C4—H4C	109.5
C1—N1—C5	108.39 (8)	H4A—C4—H4C	109.5
C7—N1—C5	111.46 (8)	H4B—C4—H4C	109.5
C1—N1—C3	111.88 (8)	C6—C5—N1	114.12 (9)
C7—N1—C3	107.94 (8)	C6—C5—H5A	108.7
C5—N1—C3	108.30 (8)	N1—C5—H5A	108.7
N1—C1—C2	114.05 (9)	C6—C5—H5B	108.7
N1—C1—H1A	108.7	N1—C5—H5B	108.7
C2—C1—H1A	108.7	H5A—C5—H5B	107.6

N1—C1—H1B	108.7	C5—C6—H6A	109.5
C2—C1—H1B	108.7	C5—C6—H6B	109.5
H1A—C1—H1B	107.6	H6A—C6—H6B	109.5
C1—C2—H2A	109.5	C5—C6—H6C	109.5
C1—C2—H2B	109.5	H6A—C6—H6C	109.5
H2A—C2—H2B	109.5	H6B—C6—H6C	109.5
C1—C2—H2C	109.5	N1—C7—C8	113.96 (8)
H2A—C2—H2C	109.5	N1—C7—H7A	108.8
H2B—C2—H2C	109.5	C8—C7—H7A	108.8
C4—C3—N1	114.85 (9)	N1—C7—H7B	108.8
C4—C3—H3A	108.6	C8—C7—H7B	108.8
N1—C3—H3A	108.6	H7A—C7—H7B	107.7
C4—C3—H3B	108.6	C7—C8—H8A	109.5
N1—C3—H3B	108.6	C7—C8—H8B	109.5
H3A—C3—H3B	107.5	H8A—C8—H8B	109.5
C3—C4—H4A	109.5	C7—C8—H8C	109.5
C3—C4—H4B	109.5	H8A—C8—H8C	109.5
H4A—C4—H4B	109.5	H8B—C8—H8C	109.5

C7—N1—C1—C2	174.07 (9)	C1—N1—C5—C6	−164.83 (9)
C5—N1—C1—C2	−64.51 (11)	C7—N1—C5—C6	−45.00 (12)
C3—N1—C1—C2	54.83 (11)	C3—N1—C5—C6	73.60 (11)
C1—N1—C3—C4	47.32 (11)	C1—N1—C7—C8	58.94 (11)
C7—N1—C3—C4	−72.47 (11)	C5—N1—C7—C8	−60.59 (11)
C5—N1—C3—C4	166.71 (9)	C3—N1—C7—C8	−179.40 (9)