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Tetraethylammonium Perchlorate at 150 K

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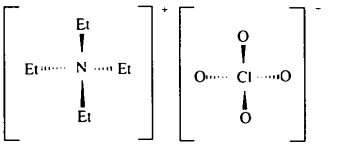
Abstract

Tetraethylammonium perchlorate, $C_8H_{20}N^+.ClO_4^-$, has been crystallized and its crystal structure is reported.

Comment

Both the tetraethylammonium cation and the perchlorate anion are widely used counterions. During the course of work aimed at understanding the structure of complexes formed between tetraalkylammonium ions and lipophilic cyclodextrins (Bates, Kataky & Parker, 1994*a*,*b*), we accidentally managed to co-crystallize tetraethylammonium with perchlorate to form the title compound, (I). Views of the ions and the unit-cell contents are shown in Figs. 1 and 2, respectively.



(I)

The tetraethylammonium ion has S_4 symmetry and its shape is very regular. The symmetry, bond lengths and bond angles agree with those reported previously [for example, by Vicente, Knop, Linden, Cameron & Robertson (1988)]. Two of the O atoms in the perchlorate ion, O2 and O3, are disordered with refined site occupancies of 0.71 (1) and 0.74 (1), respectively. As a result, the tetrahedral shape of the ion is slightly distorted. The bond lengths and angles still have acceptable values. In the crystal lattice the ions are stacked in the direction of the *b* axis. The shortest Cl···N distance is 4.795 (3) Å and the shortest O···N distance is 4.033 (7) Å. There are no significantly short distances between the ions, hence the crystal lattice is held together by weak electrostatic forces.

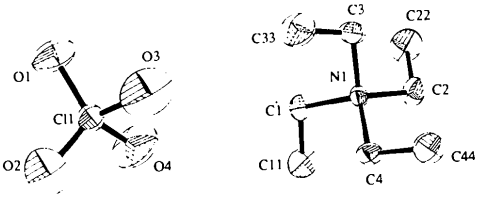


Fig. 1. A view of the cation and anion showing the numbering scheme (O atom disorder and H atoms not shown). Displacement ellipsoids are plotted at the 50% probability level.

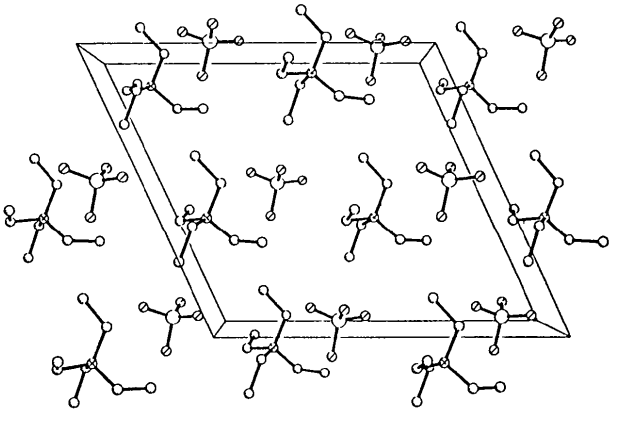


Fig. 2. The unit-cell contents viewed down **b**.

Experimental

Crystal data

$C_8H_{20}N^+.ClO_4^-$
M_r = 229.70

Mo $K\alpha$ radiation
λ = 0.71073 Å

Monoclinic
Cc
a = 12.514 (5) Å
b = 7.433 (4) Å
c = 13.956 (6) Å
β = 114.86 (3)°
V = 1178 (1) Å^3
Z = 4
D_x = 1.30 Mg m^{-3}

Cell parameters from 25 reflections
θ = 8–15°
μ = 0.314 mm^{-1}
T = 150 (2) K
Plate
0.05 × 0.50 × 0.60 mm
Colourless

Data collection

Rigaku AFC-6*S* four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
T_{min} = 0.941, T_{max} = 1.000
2305 measured reflections
1145 independent reflections
1068 observed reflections [$I > 3.0\sigma(I)$]

R_{int} = 0.0273
θ_{max} = 26.00°
$h = 0 \rightarrow 16$
$k = -9 \rightarrow 8$
$l = -8 \rightarrow 8$
3 standard reflections monitored every 150 reflections
intensity decay: 5.4%

Refinement

Refinement on F
R = 0.0489
wR = 0.0517
S = 1.05
1068 reflections
125 parameters
H-atom parameters not refined
$w = w'[1 - (\Delta F/6\sigma F)^2]^2$ where w' is a five-term Chebyshev polynomial (Prince, 1982) for F_c with coefficients of 2.27, 1.51, 2.35, 0.492 and 0.783

$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl1	0.5364 (2)	0.1269 (1)	0.3694 (2)	0.0268
O1	0.5470 (4)	0.0104 (6)	0.4535 (3)	0.0470
O2	0.5828 (7)	0.297 (1)	0.4009 (6)	0.0736
O3	0.4121 (7)	0.148 (1)	0.3011 (6)	0.0809
O4	0.5778 (5)	0.0405 (8)	0.3011 (4)	0.0701
O22	0.6427 (8)	0.243 (1)	0.4213 (8)	0.0189
O33	0.437 (1)	0.245 (2)	0.348 (1)	0.0397
N1	0.9060 (3)	0.1206 (5)	0.1181 (3)	0.0224
C1	1.0298 (4)	0.1220 (6)	0.2058 (3)	0.0267
C2	0.8954 (4)	−0.0075 (6)	0.0297 (3)	0.0326
C3	0.8222 (4)	0.0585 (6)	0.1641 (3)	0.0310
C4	0.8764 (4)	0.3094 (5)	0.0726 (3)	0.0272
C11	1.1259 (5)	0.1669 (9)	0.1707 (5)	0.0431
C22	0.9356 (7)	−0.2004 (8)	0.0641 (5)	0.0493
C33	0.8156 (6)	0.180 (1)	0.2473 (5)	0.0487
C44	0.7517 (5)	0.3345 (9)	−0.0102 (5)	0.0395

Table 2. *Selected geometric parameters* (Å, °)

Cl1—O1	1.420 (4)	N1—C3	1.515 (5)
Cl1—O2	1.383 (8)	N1—C4	1.521 (5)
Cl1—O3	1.452 (8)	C1—C11	1.515 (7)
Cl1—O4	1.416 (4)	C2—C22	1.528 (7)
N1—C1	1.518 (5)	C3—C33	1.503 (8)
N1—C2	1.519 (5)	C4—C44	1.513 (7)
O1—Cl1—O2	114.5 (4)	C2—N1—C3	108.3 (3)
O1—Cl1—O3	108.0 (3)	C1—N1—C4	108.5 (3)
O2—Cl1—O3	107.6 (6)	C2—N1—C4	108.5 (3)
O1—Cl1—O4	110.3 (3)	C3—N1—C4	111.5 (3)
O2—Cl1—O4	113.9 (4)	N1—C1—C11	114.8 (4)
O3—Cl1—O4	101.6 (4)	N1—C2—C22	115.3 (4)
C1—N1—C2	111.7 (3)	N1—C3—C33	114.7 (4)
C1—N1—C3	108.3 (3)	N1—C4—C44	115.1 (4)
C11—C1—N1—C2	56.1 (5)	C33—C3—N1—C1	63.2 (5)
C11—C1—N1—C3	175.3 (4)	C33—C3—N1—C2	−175.5 (4)
C11—C1—N1—C4	−63.5 (5)	C33—C3—N1—C4	−56.2 (5)
C22—C2—N1—C1	53.9 (5)	C44—C4—N1—C1	−175.3 (4)
C22—C2—N1—C3	−65.3 (5)	C44—C4—N1—C2	63.2 (5)
C22—C2—N1—C4	173.5 (4)	C44—C4—N1—C3	−56.1 (5)

The low-temperature experiment was carried out using a Cryostream (Oxford Cryosystems) open-flow gas cryostat (Cosier & Glaser, 1986). *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988) was used for data collection and cell determination, and *TEXSAN* (Molecular Structure Corporation, 1989) was used for data reduction and the application of an absorption correction (North, Phillips & Mathews, 1968) based on 36 ψ scans of three reflections. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990), and refined by full-matrix least squares and prepared for publication using *CRYSTALS* (Watkin, Carruthers & Betteridge, 1993). *SHELXTL-Plus* (Sheldrick, 1989) was used for molecular graphics.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1122). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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