Acta Cryst. (1993). C**49**, 1656–1657

3-Amino-1-propylammonium Perchlorate: an Extensively Hydrogen-Bonded System

Nadeem Aurangzeb, Charles A. McAuliffe,
Robin G. Pritchard* and Michael Watkinson

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

Ana Garcia-Deibe, Antonio Sousa
and Manuel R. Bermejo

Departmento de Chimica Inorganica, Universidad de Santiago, E-15706 Santiago de Compostela, Spain

(*Received* 11 *February* 1993; *accepted* 16 *March* 1993)

Abstract

The structure consists of singly charged 3-amino-1-propylammonium cations and tetrahedral perchlorate counterions. Both of the terminal N atoms form three N—H bonds, each of which is involved in a strong hydrogen bond [N···O 2.739 (5)–2.845 (5) Å]. This requires the lone proton to partially occupy sites on both N atoms, thereby giving each some ammonium character.

Comment

The 3-amino-1-propylammonium dimensions in the title compound match those of 1,3-propanediammonium diperchlorate (Pritchard *et al.*, 1992). However, comparison of the perchlorate anions in the two salts shows a significant bond lengthening in the title compound [1.456 (3)–1.478 (3), average 1.471 Å, compared with 1.406 (4)–1.445 (4), average 1.425 Å]. This is related to improved hydrogen bonding in the monoperchlorate, where five H atoms (disordered over six sites) form hydrogen bonds with N···O separation 2.739 (5)–2.845 (5) Å compared with 2.95 (1)–3.23 (1) Å for the diammonium salt. The title structure also differs from ammonium perchlorate (Lundgren, 1979) and monomethylammonium perchlorate (Zanazzi, 1968) in this respect, both of which have shorter Cl—O bonds and longer, weaker hydrogen bonds.

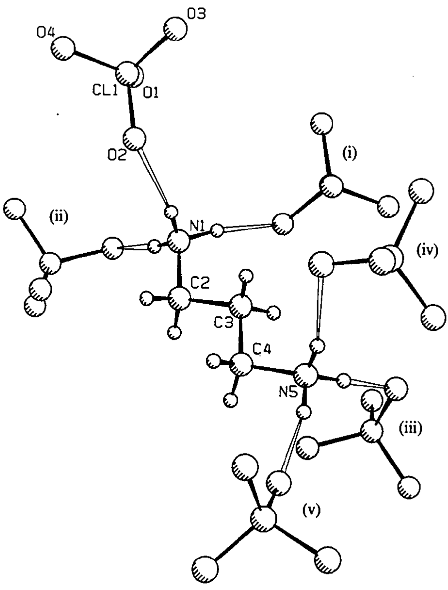

Fig. 1. The title molecule and hydrogen-bonding scheme, drawn using *PLUTO* (Motherwell & Clegg, 1978). Symmetry codes (i)–(v) are given in Table 3.

Experimental

Crystal data

$C_3H_{11}N_2^+.ClO_4^-$
$M_r = 174.58$
Orthorhombic
Pbca
$a = 12.337\ (4)$ Å
$b = 12.888\ (6)$ Å
$c = 9.677\ (7)$ Å
$V = 1538\ (2)$ Å^3
$Z = 8.00$
$D_x = 1.507$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 30.48$–$38.84°$
$\mu = 0.4589$ mm^{-1}
$T = 296$ K
Plate
0.40 × 0.15 × 0.02 mm
Buff

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF
$T_{\min} = 0.92$, $T_{\max} = 1.06$
1595 measured reflections
1595 independent reflections
989 observed reflections [$I > 2\sigma(I)$]
$R_{\text{int}} = 0.00$

$\theta_{\max} = 25.0199°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 150 reflections intensity variation: −1.70%

Refinement

Refinement on F
Final $R = 0.0390$
$wR = 0.0523$
$S = 2.077$
989 reflections
139 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{\max} = 0.0089$
$\Delta\rho_{\max} = -0.40334$ e Å^{-3}
$\Delta\rho_{\min} = 0.31512$ e Å^{-3}
Extinction correction: not applied
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: *MSC/AFC Diffractometer Control Software*. (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molec-

Acta Crystallographica Section C
ISSN 0108-2701 ©1993

ular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *TEXSAN FINISH*. Literature survey: *CSSR* (1984).

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

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	0.68223 (7)	0.57826 (6)	0.61354 (9)	0.0269
O(1)	0.6914 (3)	0.4674 (2)	0.5873 (3)	0.0601
O(2)	0.6491 (2)	0.6338 (2)	0.4868 (3)	0.0386
O(3)	0.6034 (2)	0.5973 (2)	0.7248 (3)	0.0365
O(4)	0.7892 (2)	0.6183 (2)	0.6569 (3)	0.0382
N(1)	0.5670 (3)	0.5413 (3)	0.2445 (4)	0.0306
N(5)	0.2974 (3)	0.7947 (3)	0.0770 (4)	0.0292
C(2)	0.5437 (3)	0.6254 (3)	0.1446 (5)	0.0354
C(3)	0.4311 (3)	0.6679 (3)	0.1616 (5)	0.0343
C(4)	0.4103 (3)	0.7561 (3)	0.0657 (4)	0.0307
H(1*A*)	0.505 (3)	0.498 (3)	0.267 (4)	0.03 (1)
H(1*B*)	0.583 (3)	0.564 (3)	0.315 (5)	0.04 (1)
H(1*C*)	0.616 (4)	0.493 (4)	0.210 (5)	0.06 (1)
H(2*A*)	0.594 (3)	0.673 (3)	0.151 (4)	0.024 (10)
H(2*B*)	0.550 (3)	0.601 (3)	0.057 (4)	0.04 (1)
H(3*A*)	0.378 (4)	0.612 (3)	0.138 (4)	0.05 (1)
H(3*B*)	0.425 (4)	0.690 (4)	0.236 (5)	0.06 (2)
H(4*A*)	0.423 (2)	0.738 (3)	−0.024 (4)	0.022 (10)
H(4*B*)	0.452 (3)	0.807 (3)	0.085 (4)	0.04 (1)
H(5*A*)	0.237 (3)	0.741 (4)	0.056 (5)	0.06 (1)
H(5*B*)	0.284 (3)	0.816 (3)	0.152 (5)	0.03 (1)
H(5*C*)	0.290 (3)	0.849 (3)	0.020 (4)	0.04 (1)

Table 2. *Geometric parameters* (Å, °)

Cl(1)—O(1)	1.456 (3)	N(1)—C(2)	1.480 (5)
Cl(1)—O(2)	1.478 (3)	N(5)—C(4)	1.483 (5)
Cl(1)—O(3)	1.472 (3)	C(2)—C(3)	1.503 (6)
Cl(1)—O(4)	1.478 (3)	C(3)—C(4)	1.491 (6)
O(1)—Cl(1)—O(2)	110.6 (2)	O(3)—C(1)—O(4)	108.9 (2)
O(1)—Cl(1)—O(3)	110.1 (2)	N(1)—C(2)—C(3)	112.0 (3)
O(1)—C(1)—O(4)	108.8 (2)	C(2)—C(3)—C(4)	111.6 (3)
O(2)—C(1)—O(3)	110.1 (2)	N(5)—C(4)—C(3)	111.8 (3)
O(2)—Cl(1)—O(4)	108.3 (2)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)	H(1*A*)	O(3$^{\rm i}$)	0.97 (4)	1.82 (4)	2.775 (5)	168 (3)
N(1)	H(1*B*)	O(2)	0.76 (4)	2.06 (5)	2.819 (5)	170 (4)
N(1)	H(1*C*)	O(4$^{\rm ii}$)	0.93 (5)	1.92 (5)	2.845 (5)	172 (4)
N(5)	H(5*A*)	O(2$^{\rm iii}$)	1.04 (5)	1.80 (5)	2.833 (5)	171 (4)
N(5)	H(5*B*)	O(4$^{\rm iv}$)	0.79 (4)	2.04 (5)	2.811 (5)	165 (4)
N(5)	H(5*C*)	O(1$^{\rm v}$)	0.90 (5)	1.86 (5)	2.739 (5)	166 (4)

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

Buff plate-like crystals of the title compound were formed as a by-product when the sodium salt of 3-(2-pyrrolmethylideneamino)propylamine (0.0033 mol) in 100 ml of methanol was reacted with manganese(II) perchlorate (0.0011 mol) also in methanol (50 ml). The resultant red-brown precipitate from this reaction was collected and dried. A sample was then redissolved in methanol and the resultant solution layered with *n*-hexane. This resulted in crystal formation after approximately 2 weeks of standing at ambient temperature.

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Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71193 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1052]

Acta Cryst. (1993). C**49**, 1657–1659

Structures of the Diels–Alder Reaction Products of Thymoquinone and 1-Vinylcyclohexene. I. 2-Isopropyl-5-methyltricyclo[8.4.0.0^{2,7}]tetradeca-4,9-diene-3,6-dione

J. IULEK† AND J. ZUKERMAN-SCHPECTOR*

Instituto de Física e Química de São Carlos,
Universidade de São Paulo, Caixa Postal 369,
13560-970 – São Carlos – SP, Brazil

T. J. BROCKSOM AND F. SILVA JR

Universidade Federal de São Carlos,
Departamento de Química, Caixa Postal 676,
13565-905 São Carlos SP, Brazil

(*Received* 16 *December* 1992; *accepted* 2 *March* 1993)

Abstract

The title molecule has an all-*cis* ring-fusion tricyclic structure, with the angular isopropyl group next to the two cyclohexene ring substituents. The cyclohexenedione ring is in a sofa conformation, the middle cyclohexene ring adopts a half-chair conformation and the cyclohexane ring adopts an almost-ideal chair conformation. The molecule adopts an overall distorted-hemispherical conformation.

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† Permanent address: Departamento de Química, Universidade Estadual de Ponta Grossa, PR, Brazil.

Acta Crystallographica Section C
ISSN 0108-2701 ©1993