$a = 12.600\,(3)$ Å
$b = 7.258\,(1)$ Å
$c = 6.162\,(1)$ Å
$\beta = 114.49\,(3)°$
$V = 512.8\,(2)$ Å^3
$Z = 2$
$D_x = 1.698$ Mg m^{-3}
D_m not measured

$\theta = 11–18°$
$\mu = 0.299$ mm^{-1}
$T = 293\,(2)$ K
Transparent plate
0.40 × 0.35 × 0.25 mm
Colourless

Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2941 measured reflections
855 independent reflections
763 observed reflections
[$I > 2\sigma(I)$]
$R_{\text{int}} = 0.0378$

$\theta_{\max} = 31.06°$
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 9$
$l = -8 \rightarrow 8$
2 standard reflections
monitored every 50
reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R(F) = 0.0304$
$wR(F^2) = 0.0620$
$S = 1.040$
855 reflections
54 parameters
H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0003P)^2 + 0.48P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.328$ e Å^{-3}
$\Delta\rho_{\min} = -0.201$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.049 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

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

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

	x	y	z	U_{eq}
Si	0	0	0	0.0276 (2)
F1	−0.01623 (6)	0.16438 (11)	0.1766 (2)	0.0415 (2)
F2	0.14496 (8)	0	0.1630 (2)	0.0385 (3)
N2	0.61924 (15)	0	0.2735 (4)	0.0408 (4)
C1	0.72359 (14)	0	0.2692 (3)	0.0293 (3)
N1	0.77580 (11)	0.1579 (2)	0.2703 (2)	0.0371 (3)

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

Si—F2	1.6805 (12)	N2—C1	1.326 (2)
Si—F1	1.6833 (8)	C1—N1	1.3203 (14)
F2—Si—F2$^{\text{i}}$	180.0	F1$^{\text{i}}$—Si—F1	180.0
F2—Si—F1$^{\text{i}}$	89.61 (4)	N1—C1—N1$^{\text{ii}}$	120.5 (2)
F2—Si—F1	90.39 (4)	N1—C1—N2	119.73 (8)

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

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

D—H···A	H···A	D···A	D—H···A
N1—H11···F2$^{\text{i}}$	2.08 (2)	2.901 (2)	171 (2)
N1—H12···F1$^{\text{ii}}$	2.08 (2)	2.908 (2)	167 (2)
N2—H21···F1$^{\text{iii}}$	2.05 (2)	2.891 (1)	172 (2)

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

Data collection: KM-4 software (version 7.1). Cell refinement: KM-4 software (version 7.1). Data reduction: KM-4 software (version 7.1). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXS*86 (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

The author is grateful to Professor Z. Czapla (Institute of Experimental Physics, Wrocław University) for the crystal sample and to M. Drozd (Institute of Low Temperature and Structure Research, Polish Academy of Sciences) for the DSC measurements.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1247). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 130–132

2-Methylperimidinium Acetate Monohydrate

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(*Received 30 September 1996; accepted 8 October 1996*)

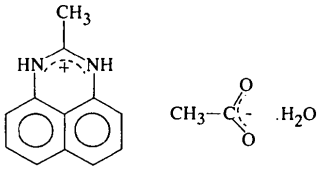
Abstract

The title compound, $C_{12}H_{11}N_2^+.C_2H_3O_2^-.H_2O$, is a hydrated salt of 2-methylperimidinium acetate. The 2-methylperimidinium cations are protonated at the N atoms. In the acetate anions, the C—O bond distances

are 1.238 (3) and 1.265 (3) Å. A hydrogen-bonding network allows the self assembly of the ionic counterparts, together with the water of hydration, along the *c* axis.

Comment

This X-ray diffraction study was carried out in order to determine unambiguously the nature of the structure of the title compound, (I), at room temperature.



(I)

The C—C bond distances in the perimidinium moiety range between 1.356 (4) and 1.417 (4) Å, while the C—N distances are in the range 1.315 (4)–1.409 (3) Å. The least-squares calculation reveals that the perimidinium moiety and the acetate group each adopt planar conformations. The C—O distances in the acetate are 1.238 (3) and 1.265 (3) Å. The crystal structure is held together by hydrogen bonds of the N—H···O and O—H···O types (Table 3). Inversion-related perimidinium planes are stacked along the *b* axis, with plane-to-plane separations of 3.493 (5) and 3.401 (4) Å.

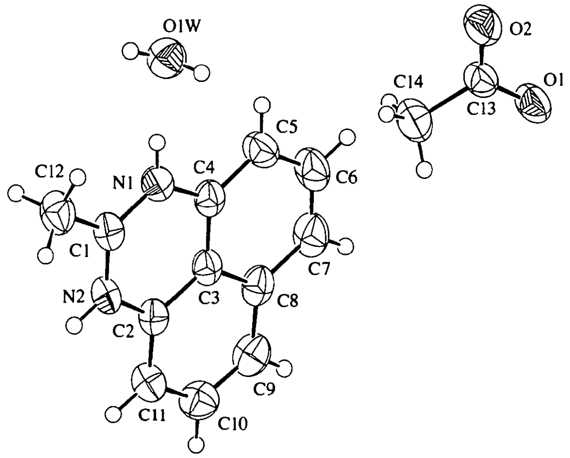


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of (I) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 40% probability level.

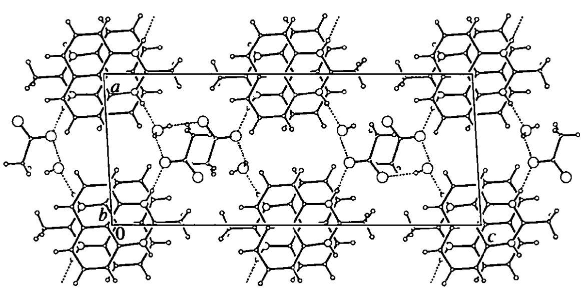


Fig. 2. Packing of the molecules in the unit cell shown down the *b* axis.

Experimental

A solution of 1,8-diaminonaphthalene (6.33 mmol, 1 g) in ether (20 ml) at 273 K was treated with acetic anhydride (13 mmol, 1.326 g) in ether (20 ml) at such a rate that the ether refluxed gently. The resulting yellow precipitate was stirred for 15 min, filtered off, washed with ether and recrystallized from chloroform. Suitable crystals (m.p. 367–369 K) were isolated by slow evaporation from chloroform.

Crystal data

$C_{12}H_{11}N_2^+.C_2H_3O_2^-.H_2O$
$M_r = 260.29$
Monoclinic
$P2_1/c$
$a = 8.480\,(2)$ Å
$b = 7.436\,(2)$ Å
$c = 20.783\,(2)$ Å
$\beta = 93.50\,(2)°$
$V = 1308.1\,(5)$ Å^3
$Z = 4$
$D_x = 1.322$ Mg m^{-3}
D_m not measured

Cu K_α radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 7–26°$
$\mu = 0.773$ mm^{-1}
$T = 293\,(2)$ K
Needle
$0.6 \times 0.1 \times 0.1$ mm
Yellow

Data collection

Enraf Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2051 measured reflections
1910 independent reflections
1500 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0270$

$\theta_{\rm max} = 60.04°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 7$
$l = -23 \rightarrow 23$
3 standard reflections monitored every 99 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0531$
$wR(F^2) = 0.1656$
$S = 1.093$
1900 reflections
169 parameters
H atoms refined using a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2 + 1.0843P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.264$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.278$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
N1	0.1189 (3)	0.1447 (3)	0.41911 (11)	0.0444 (6)
N2	−0.1388 (3)	0.2392 (3)	0.41091 (10)	0.0432 (6)
C1	−0.0145 (3)	0.1723 (4)	0.38452 (13)	0.0436 (7)

C2	−0.1373 (3)	0.2843 (4)	0.47678 (13)	0.0411 (7)
C3	0.0062 (3)	0.2610 (4)	0.51362 (12)	0.0398 (7)
C4	0.1387 (3)	0.1887 (4)	0.48489 (13)	0.0425 (7)
C5	0.2797 (4)	0.1658 (4)	0.51948 (15)	0.0529 (8)
C6	0.2906 (4)	0.2180 (5)	0.5846 (2)	0.0606 (9)
C7	0.1649 (4)	0.2875 (4)	0.61389 (15)	0.0576 (9)
C8	0.0176 (4)	0.3088 (4)	0.57953 (13)	0.0467 (7)
C9	−0.1201 (4)	0.3771 (4)	0.60619 (14)	0.0557 (8)
C10	−0.2570 (4)	0.3957 (5)	0.56952 (15)	0.0573 (8)
C11	−0.2681 (3)	0.3501 (4)	0.50381 (14)	0.0506 (8)
C12	−0.0239 (4)	0.1291 (5)	0.31447 (14)	0.0577 (9)
O1	0.5870 (2)	0.2011 (3)	0.84757 (9)	0.0587 (6)
O2	0.6843 (2)	0.0797 (3)	0.76138 (11)	0.0656 (7)
C13	0.5721 (3)	0.1223 (4)	0.79357 (14)	0.0476 (7)
C14	0.4064 (4)	0.0808 (5)	0.7681 (2)	0.0645 (9)
O1*W*	0.3749 (2)	0.0269 (3)	0.36254 (10)	0.0582 (6)

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

N1—C1	1.318 (3)	C5—C6	1.406 (4)
N1—C4	1.406 (3)	C6—C7	1.360 (5)
N2—C1	1.315 (4)	C7—C8	1.410 (4)
N2—C2	1.409 (3)	C8—C9	1.417 (4)
C1—C12	1.488 (4)	C9—C10	1.356 (4)
C2—C11	1.365 (4)	C10—C11	1.405 (4)
C2—C3	1.409 (4)	O1—C13	1.265 (3)
C3—C4	1.410 (4)	O2—C13	1.238 (3)
C3—C8	1.413 (4)	C13—C14	1.504 (4)
C4—C5	1.368 (4)		
C1—N1—C4	122.8 (2)	N1—C4—C3	116.7 (2)
C1—N2—C2	122.4 (2)	C4—C5—C6	118.4 (3)
N2—C1—N1	120.9 (2)	C7—C6—C5	121.9 (3)
N2—C1—C12	119.5 (2)	C6—C7—C8	120.7 (3)
N1—C1—C12	119.5 (3)	C7—C8—C3	117.9 (3)
C11—C2—N2	121.8 (2)	C7—C8—C9	124.6 (3)
C11—C2—C3	121.2 (3)	C3—C8—C9	117.4 (3)
N2—C2—C3	117.0 (2)	C10—C9—C8	120.9 (3)
C2—C3—C4	120.1 (2)	C9—C10—C11	121.8 (3)
C2—C3—C8	120.1 (3)	C2—C11—C10	118.6 (3)
C4—C3—C8	119.9 (3)	O2—C13—O1	124.0 (3)
C5—C4—N1	122.0 (3)	O2—C13—C14	119.3 (3)
C5—C4—C3	121.2 (3)	O1—C13—C14	116.6 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1*W*	0.86	1.82	2.678 (3)	173
N2—H2···O1^{i}	0.86	1.78	2.639 (3)	172
O1*W*—H1*W*···O1^{ii}	0.77	2.08	2.737 (3)	143
O1*W*—H2*W*···O2^{iii}	0.77	1.97	2.712 (3)	161

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

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTO*82 (Motherwell, Sussman & Beebf, 1982) and *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983).

The authors thank the Council of Scientific and Industrial Research (CSIR) and the University Grants Commission (UGC), India, for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1235). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 132–134

N,*N*′-Bis(1-pyrenylmethyl)-1,4,10,13-tetraoxa-7,16-diazacyclooctadecane

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(*Received 23 July 1996; accepted 23 September 1996*)

Abstract

Molecules of the title compound, $C_{46}H_{46}N_2O_4$, are centrosymmetric and the two pyrene rings are *anti* with respect to one another across the crown ether ring. No packing *via* π–π interaction between the pyrene planes is observed.

Comment

Charge-transfer (CT) molecular complexes have found wide application in the field of electronic devices as well as in chemistry. X-ray crystallographic studies of these complexes may provide direct information on the charge-transfer interaction in the ground and excited states (Winnik, 1993; Fabrizzi & Poggi, 1995; Petrov, Borisenko, Alfimov, Fiebig & Staerk, 1996). Interesting types of electron-acceptor and electron-donor molecules have recently been discovered which have aroused interest in CT complexes and their possible applications as chromoionophores and fluoroionophores (Löhr & Vögtle, 1985). It has been found that the azacrown ethers act as electron donors in a typical exciplex-forming system involving an appropriate electron acceptor (Petrov *et al.*, 1996). We have investigated the complexation behavior of the title compound, (1), with various metal ions in the excited singlet state

Acta Crystallographica Section C
ISSN 0108-2701 © 1997