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Synthesis and Crystal Structure of Ammonium (Picrate) (Dibenzo-18-crown-6)

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Abstract. NH_4(Pic)(DB18C6) (Pic = picrate and DB18C6 = dibenzo-18-crown-6), ($C_{26}H_{30}N_4O_{13}$) FW 606.56, orthorhombic, $Pmn2_1$, a = 26.045(5), b = 12.055(3), c = 8.982(3) Å, V = 2820(1) Å^3, Z = 4, D_c = 1.429 g/cm^3, Cu$K\alpha$, λ = 1.54184 Å, μ(Cu$K\alpha$) = 9.5 cm^{-1}, F(000) = 1272, T = 298 K. The structure has been refined to R = 0.0475 for 2617 unique observed reflections. In the lattice the 1 : 1 complex exists as a 2 : 2 dimer in which the crowns are coupled through the Pic anions and NH_4^+ cations. The asymmetric unit consists of two independent half crown ethers of which two opposite O atoms are on the mirror plane, two half ammonium cations of which the N and two H atoms are also on the mirror plane while the Pic anion is in a general position. Relative to each other, the crown ethers are shifted by about 7.3 Å along b and 1 Å along c. The 1 : 1 sandwich of NH_4 with DB18C6 and Pic on dimerisation becomes a 'club pseudo-sandwich' with three phenyl rings on either side of the mirror plane, thus forming a nearly parallel stack with a 3.6 Å inter-ring distance. The NH_4 ions hold the structure; two H atoms on the mirror plane are hydrogen-bonded to the opposite oxygens of the crown located on the purely aliphatic part of the ring (2.10(1), 2.06(3) and 2.26(3), 2.05(1) Å) for the two independent crowns, respectively, while the other two H atoms form mirror-related bifurcated hydrogen bonds with the phenoxide oxygen (1.99(1) and 2.01(1) Å) and the *o*-nitrogen oxygen (2.44(2) and 2.34(1) Å) of the picrates.

Key words: Ammonium, picrate, dibenzo-18-crown-6, molecular structure.

Supplementary Data relating to this article are deposited with the British Library as Supplementary Publication No. SUP 82037 (29 pages).

1. Introduction

With a view to follow potassium in biological systems, the NH_4^+ ion has been recommended [1] as a probe because of its comparable size and its NMR properties. This led one of us [2] to an extensive examination of the Lewis acid status of these two cations under different chemical (nucleophilic) environments and to the crystallographic behavior in salts and complexes of NH_4^+. In this paper we describe the crystallographic features of the 1 : 1 complex NH_4(Pic)(DB18C6) which exists as a 2 : 2 dimer in the crystal lattice, with each NH_4^+ ion effectively encapsulated within the pseudo-sandwich of one crown molecule and two Pic anions.

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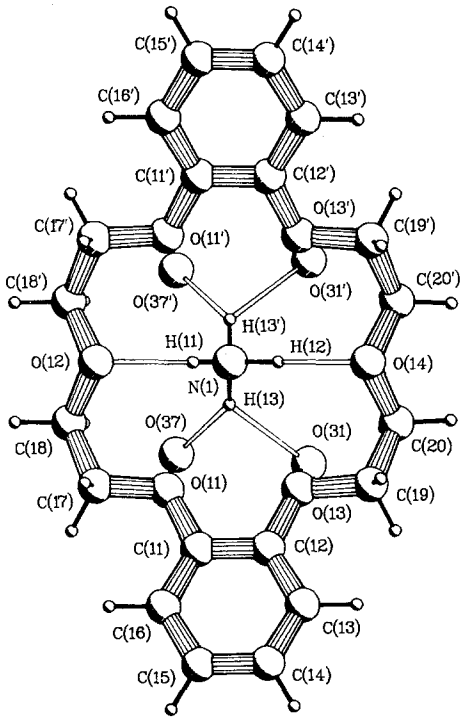

Fig. 1(a)

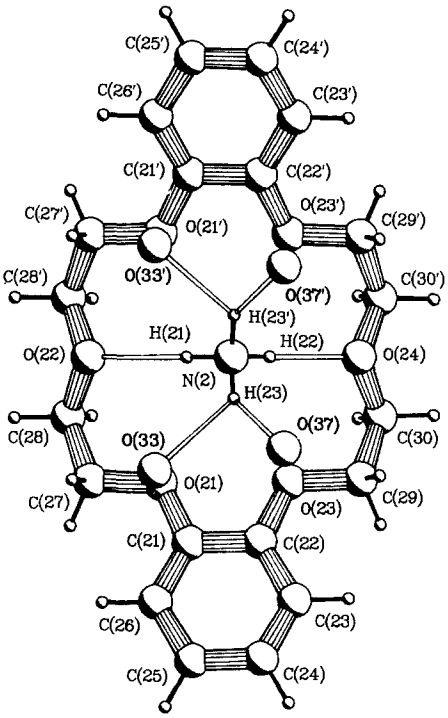

Fig. 1(b)

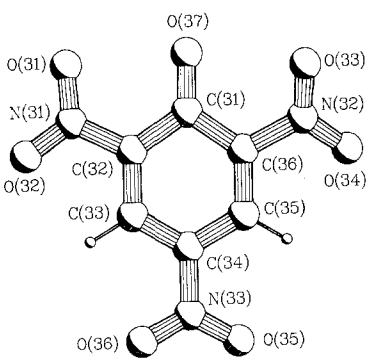

Fig. 1(c)

Fig. 1. Perspective view of the crown and picrate moieties with atom designations. The asymmetric unit consists of two half crowns and NH_4^+ cations, crown (**I**) and NH_4^+ (**I**) shown on the left, crown (**II**) and NH_4^+ (**II**) on the right and one picrate molecule shown below.

2. Experimental

2.1. SYNTHESIS OF NH_4(Pic)(DB18C6)

A 1 : 1 reaction mixture of NH_4(Pic) and DB18C6 (0.2 mM each) in ethanol (5 ml) was subjected to slow evaporation at room temperature until orange yellow crystals of the complex (m.p. 170–180 °C) were obtained.

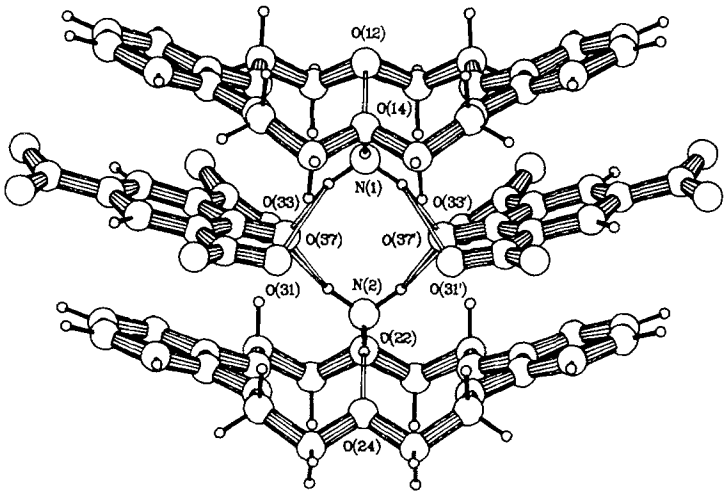

Fig. 2. Perspective view of the molecule with hydrogen bonds.

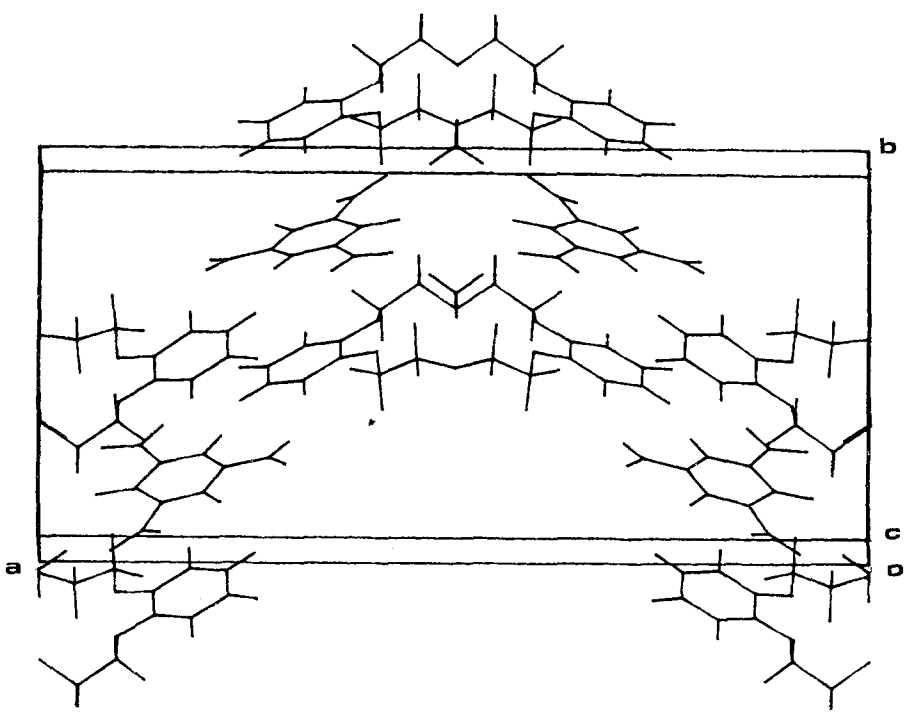

Fig. 3. View of the unit cell along *c*, showing the molecular packing.

2.2. STRUCTURAL ANALYSIS

An orange-yellow crystal measuring approximately 0.2 × 0.2 × 0.6 mm was used in data collection. Reflections were measured with a Enraf-Nonius CAD-4F diffractometer with Ni-filtered Cu$K\alpha$ radiation (λ = 1.54184 Å). Lattice parameters were determined from 21 reflections (θ-range 13.4–18.2°). Intensity data were collected using the $\omega/2\theta$-scan technique [$\omega = (0.60 + 0.15 \tan \theta)°$]. In one octant 3068 reflections were collected with $2\theta_{max} = 140°$ of which 2617 reflections with $I > 2.5\sigma(I)$ were considered observed. Standard reflections $\bar{3}\,2\,\bar{2}$, $3\,2\,\bar{2}$, $\bar{3}\,\bar{2}\,\bar{2}$ and $3\,\bar{2}\,\bar{2}$ were measured every 60 reflections (variation 0.7%). Lorentz polarization corrections were applied, but absorption was neglected.

Table I. Geometry of the hydrogen bonds

N—H···O	N···O (Å)	N—H (Å)	H···O (Å)	N—H···O (°)
N(1)—H(11)···O(12)	3.062(6)	1.01(2)	2.10(1)	158(3)
N(1)—H(12)···O(14)	3.067(6)	1.01(3)	2.06(3)	177(2)
N(1)—H(13)···O(31)	3.184(5)	1.012(7)	2.44(2)	130(1)
N(1)—H(13)···O(37)	2.908(4)	1.012(7)	1.99(1)	149(1)
N(2)—H(21)···O(22)	3.260(5)	1.00(3)	2.26(3)	180(2)
N(2)—H(22)···O(24)	3.036(6)	1.01(1)	2.05(1)	166(3)
N(2)—H(23)···O(33)	3.118(4)	1.012(7)	2.34(1)	133(1)
N(2)—H(23)···O(37)	2.916(4)	1.012(7)	2.01(1)	148(1)

Table II. Bond distances (Å), bond angles (°) and selected torsion angles (°) with e.s.d.'s

Crown(I)		Crown(II)	
O(11)—C(11)	1.373(4)	O(21)—C(21)	1.361(4)
O(11)—C(17)	1.430(4)	O(21)—C(27)	1.420(4)
O(12)—C(18)	1.431(4)	O(22)—C(28)	1.431(3)
O(13)—C(12)	1.366(4)	O(23)—C(22)	1.355(4)
O(13)—C(19)	1.438(5)	O(23)—C(29)	1.426(5)
O(14)—C(20)	1.416(4)	O(24)—C(30)	1.408(5)
C(11)—C(12)	1.390(5)	C(21)—C(22)	1.406(5)
C(11)—C(16)	1.390(4)	C(21)—C(26)	1.376(4)
C(12)—C(13)	1.390(4)	C(22)—C(23)	1.405(4)
C(13)—C(14)	1.390(5)	C(23)—C(24)	1.361(5)
C(14)—C(15)	1.375(8)	C(24)—C(25)	1.390(8)
C(15)—C(16)	1.387(6)	C(25)—C(26)	1.409(5)
C(17)—C(18)	1.477(4)	C(27)—C(28)	1.494(4)
C(19)—C(20)	1.476(6)	C(29)—C(30)	1.490(7)
Picrate			
O(37)—C(31)	1.239(4)	N(31)—O(31)	1.220(4)
N(31)—O(32)	1.210(5)	N(31)—C(32)	1.440(5)
N(32)—C(36)	1.468(4)	N(32)—O(33)	1.216(4)
N(32)—O(34)	1.216(4)	N(33)—O(36)	1.226(5)
C(31)—C(32)	1.462(4)	C(31)—C(36)	1.457(5)
C(32)—C(33)	1.377(4)	C(33)—C(34)	1.372(5)
C(34)—C(35)	1.388(4)	C(35)—C(36)	1.372(4)
N(33)—C(34)	1.437(4)	N(33)—O(35)	1.219(5)

Table II (continued)

Crown(I)		Crown(II)	
C(11)—O(11)—C(17)	116.6(3)	C(21)—O(21)—C(27)	116.5(3)
C(18)—O(12)—C(18′)	110.4(3)	C(28)—O(22)—C(28′)	110.0(2)
C(12)—O(13)—C(19)	116.3(3)	C(22)—O(23)—C(29)	117.7(3)
C(20)—O(14)—C(20′)	114.0(3)	C(30)—O(24)—C(30′)	111.1(3)
O(11)—C(11)—C(12)	116.7(3)	O(21)—C(21)—C(22)	114.7(3)
O(11)—C(11)—C(16)	123.5(3)	O(21)—C(21)—C(26)	125.1(3)
C(12)—C(11)—C(16)	119.8(3)	C(22)—C(21)—C(26)	120.3(3)
C(11)—C(12)—O(13)	116.6(2)	C(21)—C(22)—O(23)	116.1(2)
C(11)—C(12)—C(13)	119.5(3)	C(21)—C(22)—C(23)	118.7(3)
O(13)—C(12)—C(13)	123.9(3)	O(23)—C(22)—C(23)	125.2(3)
C(12)—C(13)—C(14)	120.3(4)	C(22)—C(23)—C(24)	121.3(4)
C(13)—C(14)—C(15)	120.1(4)	C(23)—C(24)—C(25)	119.9(3)
C(14)—C(15)—C(16)	119.9(5)	C(24)—C(25)—C(26)	120.1(3)
C(11)—C(16)—C(15)	120.3(4)	C(21)—C(26)—C(25)	119.7(4)
O(11)—C(17)—C(18)	109.2(3)	O(21)—C(27)—C(28)	107.6(3)
C(17)—C(18)—O(12)	110.5(3)	C(27)—C(28)—O(22)	111.0(2)
O(13)—C(19)—C(20)	108.7(4)	O(23)—C(29)—C(30)	107.7(4)
C(19)—C(20)—O(14)	110.5(3)	C(29)—C(30)—O(24)	110.2(3)
Picrate			
O(31)—N(31)—O(32)	121.0(4)	O(31)—N(31)—C(32)	119.9(3)
O(32)—N(31)—C(32)	119.0(3)	O(33)—N(32)—C(36)	119.9(3)
O(34)—N(32)—C(36)	117.3(3)	O(33)—N(32)—O(34)	122.8(3)
O(35)—N(33)—C(34)	119.0(3)	O(36)—N(33)—C(34)	118.3(3)
O(35)—N(33)—O(36)	122.7(3)	O(37)—C(31)—C(32)	124.8(3)
C(32)—C(31)—C(36)	111.4(2)	O(37)—C(31)—C(36)	123.8(3)
N(31)—C(32)—C(31)	120.5(2)	N(31)—C(32)—C(33)	116.1(3)
C(31)—C(32)—C(33)	123.4(3)	C(32)—C(33)—C(34)	120.3(3)
N(33)—C(34)—C(33)	120.2(3)	C(33)—C(34)—C(35)	121.0(2)
N(33)—C(34)—C(35)	118.8(3)	C(34)—C(35)—C(36)	119.0(3)
C(31)—C(36)—C(35)	124.7(3)	N(32)—C(36)—C(31)	118.9(2)
N(32)—C(36)—C(35)	116.4(3)		

Crown(I)		Crown(II)	
C(17)—O(11)—C(11)—C(12)	−169.5(3)	C(27)—O(21)—C(21)—C(22)	−177.1(3)
C(17)—O(11)—C(11)—C(16)	9.4(4)	C(27)—O(21)—C(21)—C(26)	5.2(4)
C(11)—O(11)—C(17)—C(18)	−173.6(3)	C(21)—O(21)—C(27)—C(28)	−169.7(2)
C(17)—C(18)—O(12)—C(18′)	−178.6(3)	C(27)—C(28)—O(22)—C(28′)	178.4(3)
C(19)—O(13)—C(12)—C(11)	170.6(3)	C(29)—O(23)—C(22)—C(21)	−178.1(3)
C(19)—O(13)—C(12)—C(13)	−9.3(4)	C(29)—O(23)—C(22)—C(23)	2.4(5)
C(12)—O(13)—C(19)—C(20)	171.2(3)	C(22)—O(23)—C(29)—C(30)	166.1(3)
C(19)—C(20)—O(14)—C(20′)	171.6(4)	C(29)—C(30)—O(24)—C(30′)	176.7(4)
O(11)—C(11)—C(12)—O(13)	−1.514)	O(21)—C(21)—C(22)—O(23)	−0.3(4)
O(11)—C(17)—C(18)—O(12)	−67.4(4)	O(21)—C(27)—C(28)—O(22)	−67.0(4)
O(13)—C(19)—C(20)—O(14)	62.8(5)	O(23)—C(29)—C(30)—O(24)	64.2(5)

Table II (continued)

Picrate			
O(31)—N(31)—C(32)—C(31)	6.6(4)	O(31)—N(31)—C(32)—C(33)	−173.2(3)
O(32)—N(31)—C(32)—C(31)	−177.9(4)	O(32)—N(31)—C(32)—C(33)	2.3(5)
O(33)—N(32)—C(36)—C(31)	21.2(4)	O(33)—N(32)—C(36)—C(35)	−159.5(3)
O(34)—N(32)—C(36)—C(31)	−158.8(3)	O(34)—N(32)—C(36)—C(35)	20.4(4)
O(35)—N(33)—C(34)—C(33)	170.2(3)	O(35)—N(33)—C(34)—C(35)	−11.9(4)
O(36)—N(33)—C(34)—C(33)	−9.5(4)	O(36)—N(33)—C(34)—C(35)	168.4(3)
O(37)—C(31)—C(32)—N(31)	−2.9(4)	O(37)—C(31)—C(36)—N(32)	5.9(4)
O(37)—C(31)—C(32)—C(33)	176.9(3)	O(37)—C(31)—C(36)—C(35)	−173.4(3)

Table III. Final fractional atomic coordinates and equivalent isotropic thermal parameters (Å^2) with e.d.s.'s

Crown(I)	x	y	z	U^a_{eq}(or U)
O(11)	0.5946(1)	0.5907(2)	0.4534(3)	0.0613(6)
O(12)	0.5000	0.6305(3)	0.3055(4)	0.063(1)
O(13)	0.5943(1)	0.4942(2)	0.7147(3)	0.0637(6)
O(14)	0.5000	0.4444(4)	0.8501(4)	0.086(1)
C(11)	0.6385(1)	0.5353(2)	0.4940(4)	0.060(1)
C(12)	0.6380(1)	0.4830(2)	0.6319(4)	0.0590(8)
C(13)	0.6808(1)	0.4231(3)	0.6777(5)	0.073(1)
C(14)	0.7243(1)	0.4182(4)	0.5882(7)	0.091(2)
C(15)	0.7252(2)	0.4725(3)	0.4536(7)	0.084(1)
C(16)	0.6818(1)	0.5281(3)	0.4041(5)	0.073(1)
C(17)	0.5917(1)	0.6279(3)	0.3025(4)	0.069(1)
C(18)	0.5451(1)	0.6962(3)	0.2833(4)	0.070(1)
C(19)	0.5908(2)	0.4262(3)	0.8458(5)	0.074(1)
C(20)	0.5456(1)	0.4615(4)	0.9328(4)	0.078(1)
Ammonium(I)				
N(1)	0.5000	0.6482(3)	0.6456(5)	0.061(1)
H(11)	0.5000	0.621(3)	0.539(1)	0.061
H(12)	0.5000	0.583(2)	0.716(3)	0.061
H(13)	0.5316	0.6949(9)	0.665(2)	0.061
H(13′)	0.4684	0.6949(9)	0.665(2)	0.061
Crown(II)				
O(21)	0.5941(1)	1.1981(2)	0.5692(3)	0.0580(6)
O(22)	0.500	1.2493(2)	0.4218(4)	0.0553(8)
O(23)	0.5931(1)	1.1010(2)	0.8239(3)	0.0653(6)
O(24)	0.500	1.0595(3)	0.9691(4)	0.070(1)
C(21)	0.6386(1)	1.1461(2)	0.6072(4)	0.0590(8)
C(22)	0.6376(1)	1.0930(2)	0.7465(4)	0.0590(8)
C(23)	0.6816(1)	1.0358(3)	0.7943(5)	0.074(1)
C(24)	0.7255(1)	1.0359(3)	0.7121(7)	0.080(1)
C(25)	0.7271(1)	1.0925(3)	0.5773(7)	0.083(2)
C(26)	0.6828(1)	1.1462(3)	0.5232(5)	0.072(1)
C(27)	0.5926(1)	1.2484(3)	0.4263(4)	0.0597(8)
C(28)	0.5450(1)	1.3174(2)	0.4187(4)	0.061(1)
C(29)	0.5912(2)	1.0510(4)	0.9677(5)	0.082(1)
C(30)	0.5446(2)	1.0938(4)	1.0449(4)	0.080(1)

Table III (continued)

Ammonium(II)				
N(2)	0.500	1.0296(3)	0.6335(5)	0.058(1)
H(21)	0.500	1.097(2)	0.568(3)	0.058
H(22)	0.500	1.053(3)	0.741(1)	0.058
H(23)	0.5316	0.9835(9)	0.612(2)	0.058
H(23′)	0.4684	0.9835(9)	0.612(2)	0.058
Picrate	x	y	z	U_{eq}[a](or U)
O(31)	0.5757(1)	0.7362(3)	0.8977(4)	0.095(1)
O(32)	0.6483(1)	0.6826(5)	0.9747(5)	0.127(2)
O(33)	0.5848(1)	0.9670(2)	0.4033(4)	0.0733(8)
O(34)	0.6438(1)	0.9075(3)	0.2588(3)	0.097(1)
O(35)	0.7950(1)	0.7450(3)	0.4579(4)	0.093(1)
O(36)	0.7991(1)	0.7031(3)	0.6898(4)	0.100(1)
O(37)	0.5686(1)	0.8385(2)	0.6392(4)	0.0703(8)
N(31)	0.6220(1)	0.7271(2)	0.8811(4)	0.0673(8)
N(32)	0.6232(1)	0.9123(2)	0.3801(3)	0.0600(6)
N(33)	0.7753(1)	0.7357(2)	0.5804(4)	0.0653(8)
C(31)	0.6153(1)	0.8198(2)	0.6305(4)	0.0557(6)
C(32)	0.6455(1)	0.7623(2)	0.7439(4)	0.0520(6)
C(33)	0.6969(1)	0.7382(2)	0.7280(4)	0.0543(6)
C(34)	0.7220(1)	0.7634(2)	0.5980(4)	0.0540(8)
C(35)	0.6971(1)	0.8191(2)	0.4836(3)	0.0537(6)
C(36)	0.6466(1)	0.8485(2)	0.5017(4)	0.0543(6)

[a] $U_{eq} = 1/3(U_{11} + U_{22} + U_{33})$

The structure was solved with direct methods and difference Fourier methods. All H atoms were located from difference syntheses and included in the refinement with isotropic thermal parameters of the carrier atoms and treated as 'riding-atoms' (C—H = 1.08 Å). In order to preserve tetrahedral symmetry of the ammonium cations it was necessary to apply an additional constraint for the H atoms outside the mirror plane for which the z coordinate was kept fixed at the value corresponding with that of tetrahedral symmetry. Anisotropic, weighted blocked full-matrix least-squares refinement on F gave $R = 0.0475$ and $wR = \Sigma w^{1/2}||F_o| - |F_c||/\Sigma w^{1/2}|F_o| = 0.0634$ with $w = [\sigma^2(F_o) + 0.012042\,F_o^2]^{-1}$. The goodness of fit was 1.41. The mean Δ/σ ratio was 0.4. A final difference synthesis revealed maximal electron densities of 0.30 eÅ^{-3}. Atomic scattering factors were taken from the *International Tables for X-ray Crystallography* [3]. Calculations were performed with MULTAN [4], SHELX-76 [5a], SHELX-84 [5b] and the EUCLID-package [6] on the CDC-Cyber-175 of the University of Utrecht.

3. Structure and Chemical Significance

The atom numbering scheme for the crown and Pic moieties is shown in Figure 1, the molecular structure in Figure 2 and the molecular packing in Figure 3. The N—H and H···O hydrogen-bond distances are indexed in Table I; bond distances, bond angles and torsion angles in Table II, and atomic coordinates with their equivalent isotropic thermal parameters in Table III. The 1 : 1 pseudo-sandwich of NH_4^+ with a DB18C6 crown molecule and two

Pic anions is dimerized (2 : 2) through Pic anions constituting a 'club pseudo-sandwich' as shown in Figure 2. The structure is held together by the NH_4^+ ions through hydrogen bonding of all four hydrogen atoms displaying a rather unique host–guest relationship with the crown and Pic.

The crown and Pic moieties are stacked in a compact way so that the three benzene rings on each side of the mirror plane are nearly parallel (interplane angles 3.5(1), 4.6(1) and 6.3(1)°). The conformation of each crown with pseudo symmetry C_{2v} is regular and ideal 'round' (torsion code *asa ag*$^+$ *a ag*$^-$ *a asa ag*$^+$ *a ag*$^-$ *a*; *a* = anti, *g* = gauche, *s* = syn) [7]. The distances between the two crown molecules, of which the donor rings are coplanar within 0.08 (I) and 0.06 Å (II), is about 7.3 Å in the *b* direction. The Pic rings are deformed somewhat as is known for this anion in the chelated state [8] so that the phenoxide oxygen is out of the ring plane while the NO_2 groups are rotated to different degrees.

In (I) the N atom of the NH_4^+ ion is displaced by 1.415(6) Å from the mean oxygen plane and located near the centre at the less-hindered side of the crown. In (II) the N atom is displaced by 1.565(6) Å and located near the centre of the most-hindered side. The two mirror plane hydrogens of the NH_4^+ ions are donated to the most highly basic oxygens of the crown ring, O(12) and O(14) [2.10(1) and 2.06(3) Å] and O(22) and O(24) [2.26(3) and 2.05(1) Å] while each of the other two mirror-related hydrogens is donated to the Pic phenoxide, O(37) [1.99(1) and 2.01(1) Å] as well as a suited *o*-nitro oxygen, O(31) [2.44(2) Å] and O(33) [2.34(1) Å].

The N—H···O$^-$ hydrogen-bond angles are 149(1)° (I) and 148(1)° (II) and the corresponding donor-acceptor N···O$^-$ distances are short (2.908(4) (I) and 2.916(4) (II) Å) (Table I) indicating that Pic binds to the NH_4^+ H atoms essentially through the phenoxide O atoms. The N—H···O(NO) angles are much more bent (130(1) (I) and 133(1)° (II) and the pertaining N···O distances are much longer (3.184(5) (I) and 3.118(4) (II) Å), suggesting that the latter interactions represent the weak part of this bifurcated hydrogen-bond configuration. The H atoms of NH_4^+ lying in the mirror plane are involved in single, almost linear hydrogen bonds.

Table I shows that the N···O distances of the hydrogen bonds cover a wide range (2.980(4)–3.260(5) Å. A survey on N···O hydrogen-bond distances in seven crown ethers with NH_4^+, RNH_3^+ or RNH_2 species (9) reports a range of 2.86–3.11 Å for 20 hydrogen bonds, which encompasses six of the eight hydrogen bonds of Table I. In recent studies N—H···O (ether) hydrogen bonds have been observed with N···O distances of 3.00 and 3.04 Å for (18-crown-6) (formamide)$_2$ [7, 10] and (1,3-xylyl-18-crown-5) (tert-butylammonium perchlorate) [11], which are close to the values for three of the N···O (ether) distances involving O(12), O(14) and O(24). Though two of the N···O distances are very long (3.184(5) and 3.260(5) Å), these values are not exceptional, as comparable, very weak hydrogen bonds with N···O distances of 3.21 and 3.28 Å have been observed in (18-crown-6) (urea)$_5$ [7, 12].

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