

Molecular graphics: *ORTEPII* (Johnson, 1976) in *NRCVAX*. Software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993). Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1150). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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n-Undecylammonium Chloride Monohydrate

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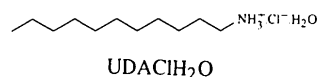
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Abstract

The structure of $C_{11}H_{26}N^+.Cl^-.H_2O$ is the first of a new class of monoalkylammonium halide compounds containing a water molecule. The latter is hydrogen bonded both to the N and Cl atoms. The structure may be a useful model for the interface between biological membranes and aqueous solutions.

Comment

Complex polymorphism has been described in long-chain monoalkylammonium chlorides (Gilson, Kertes, Manley, Tsau & Donnay, 1976). The structures of *n*-dodecylammonium chloride (DACl; Schenk & Chapuis, 1986; Pinto, Vencato, Gallardo & Mascarenhas, 1987) and *n*-dodecylammonium chloride (DDACl; Pinto, Vencato, Gallardo & Mascarenhas, 1987; Silver, Marsh & Frampton, 1995) have been described and used to interpret solid-solid phase transitions (Gault, Gallardo & Muller, 1985; Kind *et al.*, 1982; Seliger, Žagra, Blinc, Arend & Chapuis, 1983; Silver, Marsh & Frampton, 1995). The title compound, *n*-undecylammonium chloride monohydrate, UDAClH₂O, represents the first of what appears to be a new class of these compounds containing a hydrogen-bonded water molecule.



The structure of a single molecule of UDAClH₂O is shown in Fig. 1. The C—C distances vary between 1.514 (2) and 1.526 (2) Å, the C—N distance is 1.494 (2) Å and the C—C—C(N) angles are in the range 111.3 (1)–113.9 (1)°. Unlike previous findings for DACl and DDACl (Pinto, Vencato, Gallardo & Mascarenhas, 1987; Silver, Marsh & Frampton, 1995), there is no evidence of stress in the bond lengths and angles, although the torsion angles and Fig. 2 show that the UDA chains are distorted from a fully extended conformation on packing.

The structure of UDAClH₂O consists of layers built of N, Cl and O atoms alternating with layers of UDA cations (Fig. 2). The two layers are linked by hydrogen bonds between the N atoms of the UDA chains and the Cl and water O atoms, such that each N atom

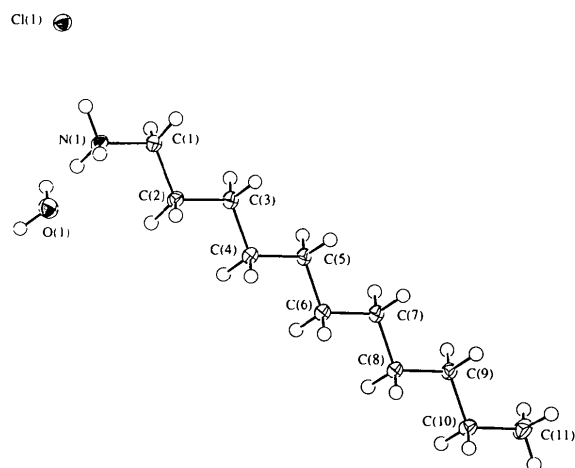


Fig. 1. View of the title molecule showing the atom-labelling scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

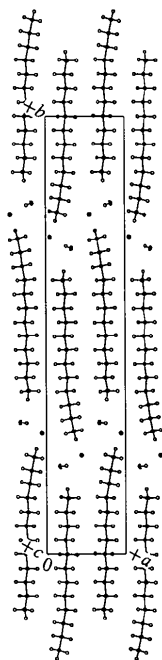


Fig. 2. Projection of the title structure down *c*. The intercalated packing of the UDA chains is shown and the offset of this packing caused by the presence of the water molecules is apparent. The ammonium group of the cation points directly at the chloride anion.

is hydrogen bonded to three Cl atoms at distances of 3.209 (1), 3.235 (2) and 3.353 (1) Å; the last of these interactions is a bifurcated hydrogen bond which interacts also with the O atom of the water molecule at a distance of 2.845 (2) Å. There are also two close Cl...O contacts of 3.180 (1) and 3.226 (1) Å, which suggests further possible hydrogen-bonding interactions. These N...Cl hydrogen-bond lengths are between 0.03 and 0.18 Å longer than those found in both DACl (Schenk & Chapuis, 1986) and DDACl (Silver, Marsh & Frampton, 1995), as would be expected where a more extensive hydrogen-bonding network occurs (caused by the incorporation of water molecules into the structure). It is worth noting that the N...Cl distances in UDACl.H₂O are shorter than those found for the Cl-to-DDA chains in (C₁₂H₂₅NH₃)₂[MnCl₄] layer structures, which are 3.27, 3.28 and 3.38 Å (Ciajolo, Corradini & Pavone, 1976).

The UDA chains are intercalated (Fig. 2) and held together by van der Waals forces in a manner similar to that found in both DACl and DDACl (Pinto, Ven- cato, Gallardo & Mascarenhas, 1987; Silver, Marsh & Frampton, 1995), except that the water molecule causes displacement of the methyl terminal of the nearest UDA chain so that intercalation is less than that found in the former anhydrous structures.

Structures of long-chain monoalkylammonium salts have often been suggested as models for biological membranes (Schenk & Chapuis, 1986). The present

structure further extends such ideas, as the incorporation of a water molecule into the hydrogen bonding in the ionic layer extends our understanding of how the hydrophilic ends of biological molecules may interact with surrounding water molecules at the membrane-aqueous interface.

Experimental

Crystals of the title compound (commercial sample from the Aldrich Chemical Co. Ltd) were grown from an aqueous ethanol solution in the presence of ferrous chloride.

Crystal data

C₁₁H₂₆N⁺.Cl⁻.H₂O

M_r = 225.80

Monoclinic

*P*2₁/*a*

a = 7.701 (2) Å

b = 40.020 (5) Å

c = 4.6437 (6) Å

β = 107.34 (1)°

V = 1366.1 (4) Å³

Z = 4

D_x = 1.098 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 20

reflections

θ = 18.8–20.8°

μ = 0.256 mm⁻¹

T = 123.0 K

Prism

0.34 × 0.28 × 0.12 mm

Colourless

Data collection

Rigaku AFC-7R diffractometer

ω scans

Absorption correction:

none

3227 measured reflections

3023 independent reflections

2301 observed reflections

[*I* > 2.5σ(*I*)]

R_{int} = 0.0213

θ_{max} = 27.0°

h = 0 → 9

k = 0 → 51

l = -5 → 5

3 standard reflections

monitored every 150

reflections

intensity decay: none

Refinement

Refinement on *F*

R = 0.0293

wR = 0.0324

S = 1.670

2301 reflections

239 parameters

All H-atom parameters

refined

w = 1/σ²(*F*)

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.23 e Å⁻³

Δρ_{min} = -0.17 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 (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Cl(1)	0.94496 (5)	0.275428 (9)	0.73053 (8)	0.0218 (1)
O(1)	1.1789 (2)	0.19908 (3)	0.1058 (3)	0.0263 (3)
N(1)	0.8487 (2)	0.22028 (3)	0.2049 (3)	0.0195 (3)
C(1)	0.8150 (2)	0.18983 (4)	0.3663 (3)	0.0191 (4)
C(2)	0.7934 (2)	0.15907 (4)	0.1690 (3)	0.0183 (4)
C(3)	0.7611 (2)	0.12778 (4)	0.3331 (3)	0.0198 (4)
C(4)	0.7714 (2)	0.09565 (4)	0.1645 (3)	0.0186 (4)
C(5)	0.7455 (2)	0.06414 (4)	0.3313 (3)	0.0185 (4)

C(6)	0.7629 (2)	0.03190 (4)	0.1665 (3)	0.0185 (4)
C(7)	0.7390 (2)	0.00035 (4)	0.3338 (3)	0.0184 (4)
C(8)	0.7566 (2)	-0.03184 (4)	0.1694 (3)	0.0185 (4)
C(9)	0.7315 (2)	-0.06341 (4)	0.3348 (3)	0.0183 (4)
C(10)	0.7481 (2)	-0.09552 (4)	0.1699 (3)	0.0215 (4)
C(11)	0.7212 (2)	-0.12701 (4)	0.3342 (4)	0.0274 (5)

Table 2. Selected geometric parameters (\AA , $^\circ$)

N(1)—C(1)	1.494 (2)	C(6)—C(7)	1.522 (2)
C(1)—C(2)	1.514 (2)	C(7)—C(8)	1.524 (2)
C(2)—C(3)	1.525 (2)	C(8)—C(9)	1.521 (2)
C(3)—C(4)	1.520 (2)	C(9)—C(10)	1.521 (2)
C(4)—C(5)	1.524 (2)	C(10)—C(11)	1.519 (2)
C(5)—C(6)	1.526 (2)		
N(1)—C(1)—C(2)	111.3 (1)	C(5)—C(6)—C(7)	113.8 (1)
C(1)—C(2)—C(3)	111.7 (1)	C(6)—C(7)—C(8)	113.8 (1)
C(2)—C(3)—C(4)	113.3 (1)	C(7)—C(8)—C(9)	113.9 (1)
C(3)—C(4)—C(5)	113.8 (1)	C(8)—C(9)—C(10)	113.9 (1)
C(4)—C(5)—C(6)	113.6 (1)	C(9)—C(10)—C(11)	113.8 (1)
N(1)—C(1)—C(2)—C(3)	179.4 (1)		
C(1)—C(2)—C(3)—C(4)	-170.0 (1)		
C(2)—C(3)—C(4)—C(5)	178.1 (1)		
C(3)—C(4)—C(5)—C(6)	-177.9 (1)		
C(4)—C(5)—C(6)—C(7)	179.4 (1)		
C(5)—C(6)—C(7)—C(8)	-179.9 (1)		
C(6)—C(7)—C(8)—C(9)	-179.6 (1)		
C(7)—C(8)—C(9)—C(10)	179.7 (1)		
C(8)—C(9)—C(10)—C(11)	-179.4 (1)		

Table 3. Contact distances (\AA)

Cl(1)···N(1)	3.209 (1)	O(1)···N(1)	2.845 (2)
Cl(1)···N(1 ⁱ)	3.235 (2)	Cl(1)···O(1 ⁱⁱⁱ)	3.180 (1)
Cl(1)···N(1 ⁱⁱ)	3.353 (1)	Cl(1)···O(1 ^{iv})	3.226 (1)

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

The crystal used for analysis was cooled with an Oxford Cryostream system (Cosier & Glazer, 1986).

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1993a). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Guaianolide 11 β H,13-Dihydro-micheliolide

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Abstract

The lactone ring of the guaianolide-class sesquiterpene lactone [3S-(3 α ,3a α ,9 α ,9a α ,9b β)]-3a,4,5,7,8,9,9a,9b-octa-hydro-9-hydroxy-3,6,9-trimethylazuleno[4,5-*b*]furan-2-(3H)-one, C₁₅H₂₂O₃, is *trans*-fused to the seven-membered ring. Two molecules are present in the asymmetric unit; they differ only slightly in conformation. The seven-membered ring is in the chair conformation, with the local mirror bisecting the double bond. The lactone is in the envelope conformation, while the other five-membered ring adopts the half chair. The two independent molecules form a hydrogen-bonded dimer, with O···O distances 2.907 (3) and 2.966 (2) \AA .

Comment

11 β H,13-Dihydromicheliolide, (1), is the major compound obtained from the BF₃-mediated rearrangement of 11 β H,13-dihydroparthenolide (Parodi, Fronczek & Fischer, 1989). The crystal structure of (1) was determined in order to confirm the relative configurations of the five asymmetric C atoms, and to compare its conformation and hydrogen bonding with those of micheliolide (Castañeda-Acosta, Fronczek & Fischer, 1991). The two independent molecules of the asymmetric unit have very similar conformations. The 17 endocyclic torsion angles of the two exhibit a mean difference of 3.4 $^\circ$, with the largest deviation, about C9—C10, of 8.4 (2) $^\circ$. The most notable conformational difference between the two molecules involves the OH group, in which the O—H bond is *anti* to C3 in the A molecule and *anti* to C5 in