

C4	0.2744 (3)	-0.2949 (2)	0.5939 (2)	2.86 (9)
C5	0.2670 (3)	-0.1556 (2)	0.5606 (2)	2.63 (8)
C6	0.2526 (3)	-0.0529 (2)	0.6249 (2)	2.74 (9)
C7	0.2465 (3)	-0.2691 (2)	0.8636 (2)	2.31 (8)
O1'	0.7667 (3)	-0.4619 (2)	0.8154 (1)	3.67 (7)
O2'	0.7247 (3)	-0.0980 (2)	1.0268 (1)	3.58 (7)
N1'	0.7709 (3)	-0.1834 (2)	0.5697 (1)	2.71 (7)
N2'	0.7839 (3)	-0.2675 (2)	0.4956 (1)	3.25 (8)
N3'	0.7834 (3)	-0.3973 (2)	0.5274 (1)	3.28 (8)
N4'	0.7703 (3)	-0.3993 (2)	0.6249 (1)	2.80 (7)
C1'	0.7528 (3)	-0.3247 (2)	0.8293 (1)	2.38 (8)
C2'	0.7501 (3)	-0.2280 (2)	0.7492 (1)	2.16 (8)
C3'	0.7378 (3)	-0.0852 (2)	0.7657 (2)	2.56 (8)
C4'	0.7282 (3)	-0.0422 (2)	0.8578 (2)	2.81 (9)
C5'	0.7320 (3)	-0.1418 (2)	0.9356 (1)	2.46 (8)
C6'	0.7437 (3)	-0.2813 (2)	0.9218 (1)	2.63 (8)
C7'	0.7626 (3)	-0.2683 (2)	0.6506 (1)	2.30 (8)
OW1	0.0840 (3)	0.2550 (2)	0.7173 (1)	3.77 (7)
OW2	0.3167 (3)	0.3281 (2)	0.8345 (1)	3.69 (7)
OW3	-0.3016 (3)	0.3067 (2)	0.6946 (1)	4.02 (7)

Table 2. Selected bond lengths (Å) and angles (°)

N1—N2	1.359 (3)	N1'—N2'	1.360 (3)
N1—C7	1.324 (3)	N1'—C7'	1.324 (3)
N2—N3	1.286 (3)	N2'—N3'	1.290 (3)
N3—N4	1.337 (3)	N3'—N4'	1.343 (3)
N4—C7	1.347 (3)	N4'—C7'	1.343 (3)
O1—C1—C2	118.6 (2)	O1'—C1'—C2'	119.2 (2)
C1—C2—C7	122.1 (2)	C1'—C2'—C7'	122.6 (2)
N1—C7—C2	127.6 (2)	N1'—C7'—C2'	126.4 (2)
N4—C7—C2	125.3 (2)	N4'—C7'—C2'	125.8 (2)

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

D—H...A	D—H	H...A	D...A	D—H...A
OW1—H1W1...N2' <sup>i</sup>	0.89	2.00	2.870 (3)	166
OW1—H2W1...OW3	0.94	1.90	2.786 (3)	156
OW2—H1W2...N1' <sup>ii</sup>	1.00	1.93	2.910 (3)	166
OW2—H2W2...OW1	0.99	1.77	2.747 (3)	167
OW3—H1W3...O2' <sup>iii</sup>	0.94	2.01	2.940 (3)	170
OW3—H2W3...OW2' <sup>iv</sup>	0.95	1.93	2.850 (3)	163
O1—HO1...OW1	0.95	1.78	2.717 (3)	171
O2—HO2...N1' <sup>i</sup>	0.99	1.95	2.941 (3)	177
N4—HN4...O2' <sup>v</sup>	0.99	1.92	2.842 (3)	154
N4—HN4...O1	0.99	2.10	2.631 (3)	112
O1'—HO1'...N4'	1.12	1.66	2.675 (3)	147
O2'—HO2'...OW2' <sup>v</sup>	0.96	1.82	2.784 (3)	173
N4'—HN4'...OW3' <sup>vi</sup>	0.86	2.08	2.943 (3)	170

Symmetry codes: (i) 1 - x, -y, 1 - z; (ii) x, 1 + y, z; (iii) -x, -y, 1 - z; (iv) x - 1, y, z; (v) 1 - x, -y, 2 - z; (vi) 1 + x, y - 1, z.

H atoms were located from a difference electron density map and were not refined. Most of the calculations were performed on a DEC 3000 AXP computer.

Data collection: *CAD-4-Express* (Enraf-Nonius, 1993). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *SHELX86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEPII*. Software used to prepare material for publication: *MolEN* (Fair, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NS1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## n-Dodecylammonium Chloride

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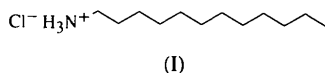
## Abstract

The redetermination of the structure of C<sub>12</sub>H<sub>28</sub>N<sup>+</sup>.Cl<sup>-</sup> corroborates and complements the limited data of a prior less accurate study. The structure consists of C<sub>12</sub>H<sub>28</sub>N<sup>+</sup> chains arranged head-to-tail in layers between layers of Cl<sup>-</sup> ions.

## Comment

The structure of dodecylammonium bromide has been reported previously along with a preliminary (projection) determination of dodecylammonium chloride (Gordon, Stenhagen & Vand, 1953). A more recent study of decylammonium chloride (DACl) and dodecylammonium chloride (DDACl) (Pinto, Vencato, Gallardo & Mascarenhas, 1987) resulted in the latter complex being solved from only 337 observed reflections using isotropic displacement factors. It was suggested (Pinto *et al.*, 1987) that the finding that the reflection intensities were weak for both compounds was perhaps due to the closeness of a phase transition (Gault, Gallardo & Muller, 1985), which may provoke crystalline disorder.

der. Unfortunately, the temperature of the data collection was not reported (Pinto *et al.*, 1987). As part of a programme of work on layer structures, we wished to have a more accurate solution of DDACl, (I), and we report here the results of that study.



The present structure was solved using 1299 observed reflections and 239 variables (reflection to parameter ratio 5.44:1, which is just acceptable). The refinement also included H atoms. The structure of (I) is shown in Fig. 1 and a packing diagram is presented in Fig. 2. The structure is essentially the same in layout as previously reported (Pinto *et al.*, 1987). The bond lengths, however, are more accurate. Refinement of the H atoms led to C—H distances in the range 0.86–1.10 Å and N—H distances of 0.83–1.05 Å. The H-atom *B* values ranged from 2.9 to 9.0 Å<sup>2</sup> with a mean of 5.0 Å<sup>2</sup>.

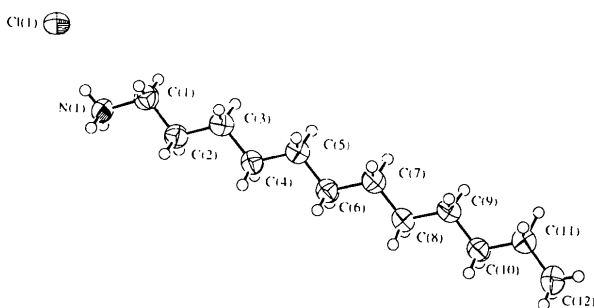


Fig. 1. View of the title molecule showing the atom-labelling scheme. The displacement ellipsoids are drawn at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

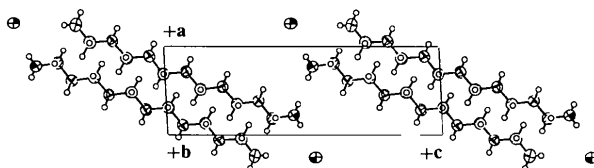


Fig. 2. Projection of the title structure down *b* showing the packing of the chains within the layers.

Crystalline cohesion is achieved by ionic bonds between the —NH<sub>3</sub><sup>+</sup> cations and the Cl<sup>−</sup> anions, and van der Waals interactions between the carbon chains. There are three close Cl(1)⋯N(1) interactions of 3.142 (3), 3.159 (4) and 3.167 (3) Å, so that each Cl<sup>−</sup> anion is surrounded by three NH<sub>3</sub><sup>+</sup> groups and each NH<sub>3</sub><sup>+</sup> by three Cl<sup>−</sup> ions. The C—C bond lengths in the C<sub>12</sub>N chain are in the range 1.491 (6)–1.526 (5) Å. The C—C bond lengths in the isostructural DACl structure (Pinto *et al.* 1987) are in the range 1.506 (8)–1.534 (9) Å. The average distance found between alternate C atoms is 2.54 Å, which is the same as that found for isostructural

DACl and similar to that found for lauric acid (Vand, Morley & Lomer, 1951). The chain length as measured from N(1) to C(12) is 15.155 (5) Å.

The carbon chains form a layer so that the tail of one stops just short of the next Cl<sup>−</sup> layer and the NH<sub>3</sub><sup>+</sup> heads of the three neighbouring chains overlap this tail balancing the charge in the Cl<sup>−</sup> layer. Thus, the packing of the carbon chains can be considered as two interdigitated layers (Fig. 2). At 318 K, DDACl is found to undergo a phase transition to an interlayer spacing of 30 Å (Gault *et al.*, 1985). This spacing is about double the chain length and could be explained by the two interdigitated layers in the low-temperature phase moving apart in the high-temperature phase allowing the chains the freedom to rotate.

## Experimental

The title compound was prepared from *n*-dodecylamine and HCl. Crystals were grown from an ethanol solution.

### Crystal data

C<sub>12</sub>H<sub>28</sub>N<sup>+</sup>·Cl<sup>−</sup>

*M<sub>r</sub>* = 221.81

Monoclinic

*P*2<sub>1</sub>

*a* = 5.682 (2) Å

*b* = 7.186 (6) Å

*c* = 17.775 (2) Å

β = 92.75 (2)°

*V* = 725.0 (6) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.016 Mg m<sup>−3</sup>

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 25 reflections

θ = 15.3–17.5°

μ = 0.235 mm<sup>−1</sup>

*T* = 294.2 K

Plate

0.60 × 0.35 × 0.09 mm

Colourless

### Data collection

Rigaku AFC-7R diffractometer

ω/2θ scans

Absorption correction: analytical

*T<sub>min</sub>* = 0.924, *T<sub>max</sub>* = 0.978

1888 measured reflections

1718 independent reflections

1299 observed reflections

[*I* > 2.0σ(*I*)]

*R<sub>int</sub>* = 0.023

θ<sub>max</sub> = 26.99°

*h* = 0 → 7

*k* = 0 → 9

*l* = −22 → 22

3 standard reflections

monitored every 150

reflections

intensity decay: 0.86%

### Refinement

Refinement on *F*

*R* = 0.0350

*wR* = 0.0307

*S* = 1.928

1299 reflections

239 parameters

All H-atom parameters refined

Weighting scheme based on measured e.s.d.'s

(Δ/σ)<sub>max</sub> = 0.0001

Δρ<sub>max</sub> = 0.15 e Å<sup>−3</sup>

Δρ<sub>min</sub> = −0.12 e Å<sup>−3</sup>

Extinction correction:

Zachariasen (1967) type

2 Gaussian isotropic

Extinction coefficient:

65.30354

Atomic scattering factors

from *International Tables for X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

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

	x	y	z	U <sub>eq</sub>
Cl(1)	1.2593 (1)	1/4	-0.53474 (4)	0.0693 (2)
N(1)	0.7727 (5)	0.1887 (5)	-0.4616 (2)	0.063 (1)
C(1)	0.8101 (5)	0.262 (1)	-0.3835 (2)	0.0609 (9)
C(2)	0.6135 (6)	0.2025 (6)	-0.3349 (2)	0.055 (1)
C(3)	0.6485 (5)	0.268 (1)	-0.2544 (2)	0.060 (1)
C(4)	0.4546 (6)	0.2097 (8)	-0.2042 (2)	0.059 (1)
C(5)	0.4815 (6)	0.276 (1)	-0.1237 (2)	0.059 (1)
C(6)	0.2874 (6)	0.2106 (7)	-0.0750 (2)	0.054 (1)
C(7)	0.3089 (6)	0.277 (1)	0.0066 (2)	0.058 (1)
C(8)	0.1148 (6)	0.2108 (7)	0.0546 (2)	0.057 (1)
C(9)	0.1345 (5)	0.2761 (9)	0.1359 (2)	0.057 (1)
C(10)	-0.0594 (6)	0.2076 (7)	0.1840 (2)	0.056 (1)
C(11)	-0.0446 (6)	0.274 (1)	0.2646 (2)	0.068 (1)
C(12)	-0.2387 (8)	0.210 (1)	0.3119 (2)	0.082 (2)

Table 2. Selected geometric parameters (Å, °)

N(1)—C(1)	1.492 (5)	C(6)—C(7)	1.526 (5)
C(1)—C(2)	1.507 (5)	C(7)—C(8)	1.505 (5)
C(2)—C(3)	1.510 (5)	C(8)—C(9)	1.517 (5)
C(3)—C(4)	1.509 (5)	C(9)—C(10)	1.510 (5)
C(4)—C(5)	1.510 (5)	C(10)—C(11)	1.508 (5)
C(5)—C(6)	1.510 (5)	C(11)—C(12)	1.491 (6)
N(1)—C(1)—C(2)	110.9 (4)	C(6)—C(7)—C(8)	114.3 (3)
C(1)—C(2)—C(3)	112.7 (3)	C(7)—C(8)—C(9)	114.8 (3)
C(2)—C(3)—C(4)	113.9 (4)	C(8)—C(9)—C(10)	114.6 (3)
C(3)—C(4)—C(5)	115.4 (3)	C(9)—C(10)—C(11)	115.3 (3)
C(4)—C(5)—C(6)	113.8 (4)	C(10)—C(11)—C(12)	115.3 (4)
C(5)—C(6)—C(7)	114.8 (3)		
N(1)—C(1)—C(2)—C(3)	178.0 (5)		
C(1)—C(2)—C(3)—C(4)	-179.8 (5)		
C(2)—C(3)—C(4)—C(5)	-178.9 (5)		
C(3)—C(4)—C(5)—C(6)	-178.6 (5)		
C(4)—C(5)—C(6)—C(7)	-179.4 (5)		
C(5)—C(6)—C(7)—C(8)	-179.8 (5)		
C(6)—C(7)—C(8)—C(9)	179.9 (5)		
C(7)—C(8)—C(9)—C(10)	-179.5 (5)		
C(8)—C(9)—C(10)—C(11)	-179.0 (5)		
C(9)—C(10)—C(11)—C(12)	178.8 (5)		

The scan width was  $(1.37 + 0.35 \tan \theta)^\circ$  with an  $\omega$ -scan rate of  $16.0^\circ \text{ min}^{-1}$ . The weak reflections [ $I < 25.0\sigma(I)$ ] were rescanned (maximum of four rescans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *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: BK1118). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## An Almost Planar Hydrazine Moiety in a Diels–Alder Adduct

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## Abstract

The structure of the product of the reaction of 1,3-cyclooctadiene and *N*-phenyl-1,2,4-triazoline-3,5-dione has been identified as 4-phenyl-2,4,6-triazatricyclo-[5.4.2.0<sup>2,6</sup>]tridec-12-ene-3,5-dione (C<sub>16</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>). The distinction between *endo* and *exo* addition cannot be made because the hydrazine moiety in the product molecule is almost planar.

## Comment

In the course of developing a new reaction for the direct conversion of epoxides to dienes, we investigated the thermal reaction of 1,2-epoxycyclooctane, (1), with