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2,3-Dihydro-2-methyl-2-indenylmethylammonium Chloride

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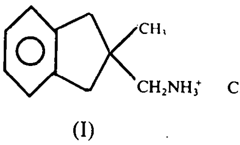
Abstract

The title compound, $C_{11}H_{16}N^+.Cl^-$, packs to give non-polar and polar regions in the crystal. In the polar region each proton of the NH_3^+ group points towards a Cl^- ion; the average N· · ·Cl separation is 3.183 Å.

Comment

The title compound ORG9768 (Organon Laboratories Ltd), (I), was prepared as a potential antidepressant. It showed the characteristic profile of antidepressants in neurotransmitter-uptake studies and in animal models.



(I)

All bond lengths and angles in the cation have typical values. The NH_3^+ group is staggered with respect to C10; the torsion angles C10—C8—C11—N12 and C9—C8—C11—N12 are 58.4 (6) and 178.8 (4)°, respectively. C7 and C9 are 0.032 Å above the plane of the benzene ring, with C8 0.540 Å above this plane; C11 and N12 are displaced by −0.041 and 0.405 Å, respectively, from the plane.

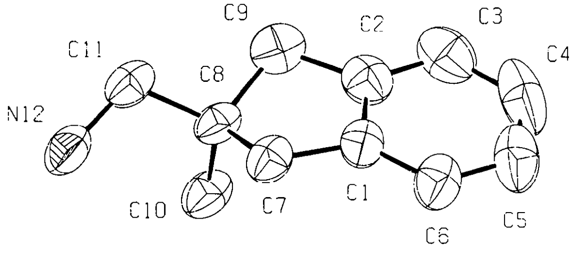


Fig. 1. The 2,3-dihydro-2-methyl-2-indenylmethylammonium cation, depicted with 50% probability displacement ellipsoids.

The molecules pack to give non-polar and polar regions in the crystal. In the latter, N12 has three Cl^- neighbours at distances of 3.15-3.20 Å, with a fourth Cl^- at 3.5 Å. The H atoms were located on difference Fourier maps and lie very close to the N· · ·Cl^- vectors, confirming the stabilization of the lattice by hydrogen bonding.

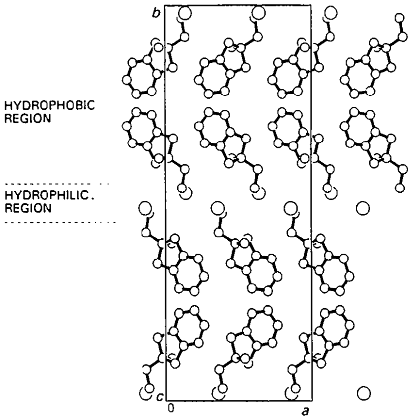


Fig. 2. Packing diagram viewed down the *c* axis showing the polar and non-polar regions to be layers parallel to the *ac* plane. Polar regions occur at *b* = 0 and *b* = 0.5. Each N atom has three equidistant Cl^- neighbours.

Experimental

ORG9768 was prepared by reaction of α,α′-dibromo-*o*-xylene with disodium ethylcyanoacetate to give a cyclized cyano ester, which was decarboxylated to 2,3-dihydro-1*H*-indene-2-carbonitrile, followed by methylation of the nitrile with lithium diisopropylamide and methyl iodide. Reduction of the nitrile gave the desired amine which was crystallized as the hydrochloride.

Crystal data

$C_{11}H_{16}N^+.Cl^-$
$M_r = 197.70$
Orthorhombic
Pbca
$a = 11.151$ (2) Å
$b = 29.244$ (4) Å
$c = 6.666$ (2) Å
$V = 2173.8$ (8) Å^3
$Z = 8$
$D_x = 1.208$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 15 reflections
$\theta = 8$-$12°$
$\mu = 0.307$ mm^{-1}
$T = 293$ (2) K
Transparent blocks
0.43 × 0.35 × 0.20 mm
Colourless

Data collection

Stoe Stadi 2 two-circle diffractometer
ω scans
Absorption correction: none
2078 measured reflections
1529 independent reflections
1264 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.023$
$\theta_{\rm max} = 25.59°$
$h = 0 \rightarrow 10$
$k = -1 \rightarrow 34$
$l = 0 \rightarrow 7$
2 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0612$
$wR(F^2) = 0.1734$
$S = 1.393$
1264 reflections
182 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.310$
$\Delta\rho_{\max} = 0.224$ e Å^{-3}
$\Delta\rho_{\min} = -0.211$ 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	$U_{\rm eq}$
Cl1	0.35996 (10)	0.01963 (4)	0.2306 (2)	0.0551 (5)
C1	0.6211 (4)	0.3608 (2)	−0.0293 (6)	0.0504 (12)
C2	0.5537 (5)	0.3244 (2)	0.0372 (7)	0.0594 (13)
C3	0.5904 (7)	0.2804 (2)	0.0039 (9)	0.078 (2)
C4	0.6995 (8)	0.2731 (3)	−0.0942 (11)	0.096 (3)
C5	0.7648 (7)	0.3094 (3)	−0.1633 (9)	0.080 (2)
C6	0.7265 (5)	0.3531 (2)	−0.1319 (7)	0.0640 (15)
C7	0.5635 (4)	0.4045 (2)	0.0274 (7)	0.0510 (13)
C8	0.4782 (4)	0.3911 (2)	0.1984 (6)	0.0481 (12)
C9	0.4452 (5)	0.3410 (2)	0.1457 (10)	0.0653 (15)
C10	0.5426 (5)	0.3922 (3)	0.3990 (9)	0.061 (2)
C11	0.3649 (4)	0.4197 (2)	0.2042 (8)	0.0537 (13)
N12	0.3864 (4)	0.4677 (2)	0.2559 (9)	0.0627 (13)

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

C1—C2	1.377 (7)	C5—C6	1.364 (8)
C1—C6	1.378 (7)	C7—C8	1.536 (6)
C1—C7	1.481 (7)	C8—C11	1.515 (6)
C2—C3	1.366 (8)	C8—C10	1.518 (7)
C2—C9	1.490 (7)	C8—C9	1.551 (8)
C3—C4	1.398 (11)	C11—N12	1.466 (7)
C4—C5	1.367 (11)		
C2—C1—C6	120.0 (5)	C1—C7—C8	103.7 (4)
C2—C1—C7	110.5 (4)	C11—C8—C10	111.1 (4)
C6—C1—C7	129.5 (5)	C11—C8—C7	113.2 (4)
C3—C2—C1	120.7 (6)	C10—C8—C7	110.8 (4)
C3—C2—C9	129.0 (6)	C11—C8—C9	109.2 (4)
C1—C2—C9	110.3 (5)	C10—C8—C9	109.4 (5)
C2—C3—C4	118.7 (7)	C7—C8—C9	102.7 (4)
C5—C4—C3	120.2 (7)	C2—C9—C8	103.1 (4)
C6—C5—C4	120.5 (7)	N12—C11—C8	113.5 (4)
C5—C6—C1	119.7 (6)		

All e.s.d.'s were estimated using the full covariance matrix. Data collection: Stoe Stadi diffractometer control. Cell refinement: local program. Data reduction: *SHELX*76 (Sheldrick, 1976). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1994). Molecular graphics: *PLUTON*, *PLATON* (Spek, 1990, 1992). Software used to prepare material for publication: *SHELXL*92.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71755 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1078]

References

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Spek, A. L. (1992). *PLUTON92. Molecular Graphics Program*. Univ. of Utrecht, The Netherlands.

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Dimethyl [3,3,4,4,5,5-Hexamethyl-1(*E*),2(*E*)-cyclopentandiylidene]diacetate, $C_{17}H_{26}O_4$

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Abstract

To establish the conformation of the diene system of the title compound, its structure was determined by X-ray analysis. The non-planar butadiene fragment is in a *gauche* conformation [torsion angle 44.8 (9)°]. The methoxycarbonyl groups are fully eclipsed with respect to the single bond in the diene system. The five-membered ring adopts a distorted envelope conformation with the apical C atom at position 3. In the cyclopentane ring, elongations of C—C bonds [1.587 (2) and 1.575 (2) Å] and contractions of C—C—C bond angles [99.9 (2), 104.0 (2), 103.3 (2)°] involving atoms at positions 3, 4 and 5 are observed.

Comment

Several unconventional products have been isolated from the combination of 3,3,4,4,5,5-hexamethyl-1,2-bis(methylene)cyclopentane with cycloaddition partners, which normally react in a 1,2 manner with other conjugated dienes (Baran & Mayr, 1987, and references therein). Therefore, this diene has been employed as a probe for the study of cycloaddition mechanisms; the results of these investigations have been reviewed recently (Mayr, Baran & Heigl, 1991). Studies of muconic acid derivatives, structurally related to this diene and possessing known (*E*,*E*) configuration at the 1,3-diene termini, should enable the elucidation of the stereochemistry of these cycloadditions. To obtain unequivocal proof

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