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2-[2-(4-Ethoxyphenyl)methyl-5-nitro-1*H*-benzimidazolyl]-*N*,*N*-diethylethanaminium Chloride–Acetic Acid: Etonitazene

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Abstract. $C_{22}H_{28}N_4O_3.HCl.C_2H_4O_2$, $P2_1/c$, a = 9·721 (5), b = 13·588 (5), c = 19·673 (5) Å, β = 92·47 (5)°, D_m = 1·25, D_c = 1·26 g cm^{-3}, Z = 4. The planes of the benzimidazole groups are stacked parallel.

Introduction. The present study was undertaken as part of an investigation of the structure–activity relationship in narcotic analgesics. The title compound is a thousand times more potent than morphine.

Colourless prismatic crystals were obtained from ethyl acetate. The hydrochloride form of the starting compound could explain the solvant hydrolysis which gave rise to the acetic acid found in the crystal structure.

The space group was determined from photographs. Cell dimensions and intensities were measured on a Nonius CAD-4 diffractometer with the experimental conditions given in Table 1. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable E map showed all atoms except H.

Full-matrix least-squares refinement with XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) converged at R = 0·14. A difference map revealed disorder at the end C of one of the N-ethyl chains; the two positions selected and the corresponding population factors were included in the refinement. The H atoms of the two hydrogen bonds were observed on this map. Refinement with anisotropic temperature factors for Cl, O, N and C and isotropic for H converged at a final R = 0·061 (for observed reflexions). The final positional parameters are given in Table 2.*

Table 1. *Experimental conditions*

Source: Cu $K\bar{\alpha}$, λ = 1·54178 Å	
Scan: ω–2θ	
Graphite monochromator	
Confidence level: 2·5σ, with $\sigma^2(I) = S + B + (0{\cdot}03S)^2$ (S being the scan and B the background count)	
$2{\cdot}0 \le \theta \le 70{\cdot}0°$	
Scanning angle: 0·8 + 0·2 tan θ (°)	
Aperture	: 2·5 + 0·5 tan θ (mm)
Total number of independent reflexions: 3282	
Number observed: 2236	

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33852 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final positional parameters* (× 10^4) *with e.s.d.'s in parentheses*

	x	y	z
C(1)	−186 (11)	1383 (8)	920 (7)
C(2)	1075 (11)	1866 (8)	1269 (6)
O(3)	1863 (5)	1046 (4)	1546 (3)
C(4)	3063 (7)	1247 (5)	1911 (4)
C(5)	3755 (7)	435 (5)	2196 (4)
C(6)	4967 (7)	568 (5)	2580 (3)
C(7)	5503 (6)	1505 (5)	2698 (3)
C(8)	4811 (7)	2300 (5)	2406 (4)
C(9)	3598 (7)	2191 (5)	2013 (4)
C(10)	6804 (6)	1650 (5)	3150 (3)
C(11)	6618 (6)	2334 (5)	3734 (3)
N(12)	7271 (5)	3178 (4)	3806 (3)
C(13)	6839 (6)	3567 (5)	4414 (3)
C(14)	7196 (6)	4454 (5)	4731 (3)
C(15)	6605 (7)	4619 (5)	5339 (3)
N(16)	6911 (7)	5565 (5)	5685 (4)
O(17)	7717 (7)	6121 (4)	5418 (3)
O(18)	6319 (7)	5763 (4)	6204 (3)
C(19)	5693 (7)	3990 (5)	5645 (3)
C(20)	5319 (7)	3108 (5)	5326 (3)
C(21)	5907 (6)	2927 (5)	4700 (3)
N(22)	5767 (5)	2147 (4)	4252 (2)
C(23)	4875 (6)	1293 (5)	4335 (3)
C(24)	3420 (6)	1525 (5)	4061 (3)
N(25)	2420 (5)	752 (4)	4261 (3)
C(26)	938 (8)	1048 (7)	4092 (5)
C(27)	409 (14)	1838 (12)	4420 (9)
C(271)	671 (18)	1392 (17)	3421 (12)
C(28)	2746 (8)	−242 (6)	3969 (4)
C(29)	1923 (10)	−1059 (6)	4280 (5)
Cl(60)	2785 (2)	691 (1)	5798 (1)
C(50)	11170 (9)	4369 (8)	2528 (4)
C(51)	9841 (9)	3914 (7)	2720 (4)
O(52)	9671 (5)	3877 (4)	3352 (3)
O(53)	8950 (7)	3660 (7)	2318 (3)

Discussion. The atomic numbering, bond distances and angles are given in Fig. 1. The main torsion angles are listed in Table 3. Fig. 2 is a stereoview of the molecule.

There are two strong hydrogen bonds from the analgesic molecule to the Cl^- ion and acetic acid molecule: Cl(60)···N(25) 3·034, N(25)–H(601) 1·002, Cl(60)···H(601) 2·036 Å, Cl(60)···H(601)–N(25) 173·49°, O(52)···N(12) 2·692, O(52)–H(521) 1·152, N(12)···H(521) 1·546 Å, O(52)–H(521)···N(12) 172·23°.

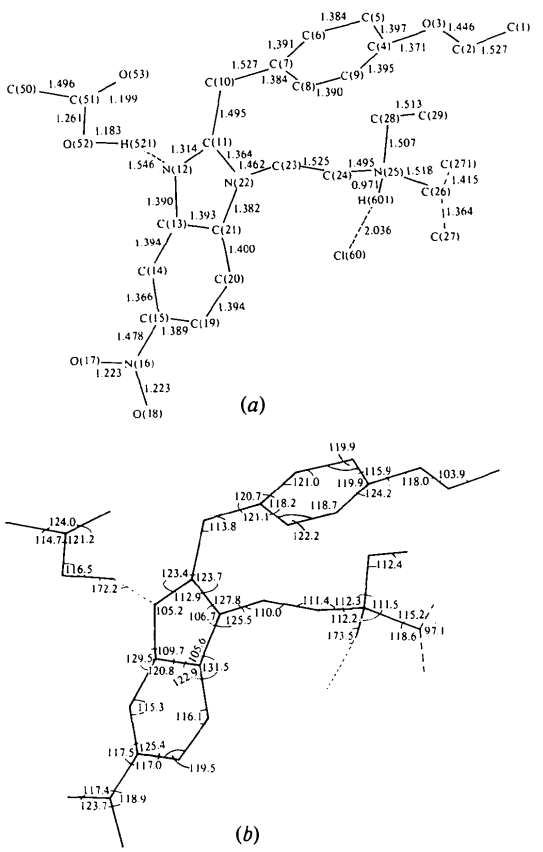

Fig. 1. (*a*) Bond distances (Å) and (*b*) angles (°). The average e.s.d.'s for the lengths are 0·009 Å and for angles 0·6° except for the terminal ethyl groups for which e.s.d.'s range between 0·026–0·012 Å and 0·8–1·3°.

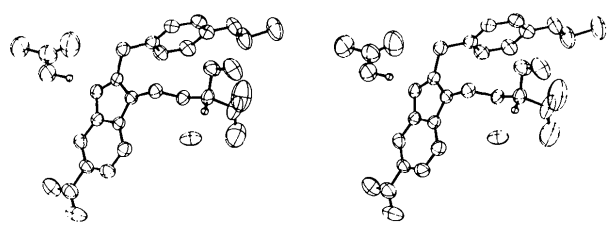

Fig. 2. Stereoscopic view (Johnson, 1965) of the molecule with 50% probability thermal ellipsoids.

Table 3. *Main torsion angles* (°)

C(6)–C(7)–C(10)–C(11)	−123·5
C(8)–C(7)–C(10)–C(11)	55·2
C(7)–C(10)–C(11)–N(22)	−116·2
C(10)–C(11)–N(22)–C(23)	−1·5
C(11)–N(22)–C(23)–C(24)	−95·5
N(22)–C(23)–C(24)–N(25)	−168·6
C(23)–C(24)–N(25)–C(26)	170·8
C(23)–C(24)–N(25)–C(28)	−62·6
C(24)–N(25)–C(28)–C(29)	168·5
C(24)–N(25)–C(26)–C(27)	−64·8
C(1)–C(2)–O(3)–C(4)	−178·4
C(2)–O(3)–C(4)–C(5)	177·0
C(2)–O(3)–C(4)–C(9)	−2·3
C(14)–C(15)–N(16)–O(17)	−2·9
C(19)–C(15)–N(16)–O(18)	−2·5

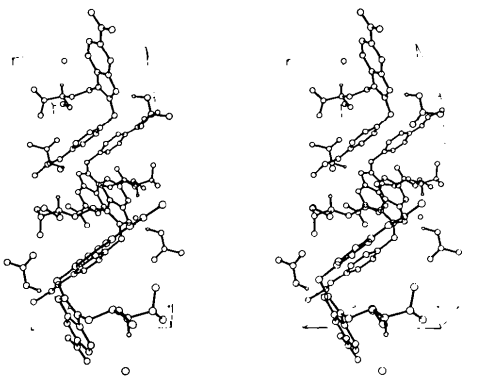

Fig. 3. Stereoscopic view of the packing.

The packing is essentially due to van der Waals interactions between the almost parallel benzimidazole groups of the (x,y,z) and $(1-x, 1-y, 1-z)$ equivalent molecules, the interplanar distances ranging from 3·4 to 3·5 Å. The resulting shape of the packing is a pleated sheet (Fig. 3).

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