

The presence of six F atoms may also make librational motion more difficult due to repulsion effects; in octafluoronaphthalene, in its 1:1 complex with naphthalene (Potenza & Mastropaolo, 1975), there is no apparent libration of the F atoms. The atoms at the outer ends of the present molecule [C(11), C(12), C(13), F(3), F(4) and F(5)] do not have temperature factors significantly larger than those of atoms nearer the centre, indicating that there is no rigid-body motion of each half of the molecule about the central C atom.

Because of the small amount of data relative to the number of parameters, the bond lengths and angles are of limited accuracy (Table 2). However, the variation of C—C bond lengths observed in the rings indicates that there is probably only partial delocalization, with some bonds [e.g. C(1)—C(3) 1.32 (1), C(6)—C(7) 1.33 (2) Å] distinctly shorter than others [e.g. C(7)—C(8) 1.44 (1), C(3)—C(4) 1.44 (1) Å]. The bond angles are those which would be expected for this structure. There is only one close intermolecular contact, a distance of 3.18 (2) Å between F(4) and F(3) of a

neighbouring molecule. This is larger than the sum of the van der Waals radii, but at the lower end of the range of non-bonded F—F distances observed for organic structures in the Cambridge Structural Database (Nyburg & Faerman, 1985).

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Diethylmethyl{2-[*p*-(*o*-octyloxybenzamido)benzoyloxy]ethyl}ammonium Bromide (I) and Diethylmethyl{2-[*p*-(*o*-propyloxybenzamido)benzoyloxy]ethyl}ammonium Bromide (II)

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(Received 8 August 1985; accepted 5 November 1985)

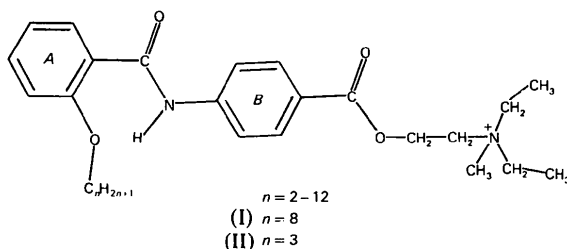
Abstract. (I) $C_{29}H_{43}BrN_2O_4$, $M_r = 563.59$, monoclinic, $P2_1/c$, $a = 19.023$ (6), $b = 16.767$ (5), $c = 9.497$ (4) Å, $\beta = 94.57$ (5)°, $V = 3020$ (2) Å³, $Z = 4$, $D_x = 1.239$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 13.40$ cm⁻¹, $F(000) = 1192$, $T = 293$ K, final conventional R is 0.079 for 1379 unique observed reflections. (II) $C_{24}H_{33}BrN_2O_4$, $M_r = 493.45$, triclinic, $P\bar{1}$, $a = 15.929$ (9), $b = 8.261$ (5), $c = 9.417$ (6) Å, $\alpha = 89.916$ (4), $\beta = 96.954$ (4), $\gamma = 89.188$ (4)°, $V = 1230$ (1) Å³, $Z = 2$, $D_x = 1.332$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 16.40$ cm⁻¹, $F(000) = 516$, $T = 293$ K, final conventional R is 0.047 for 2114 unique observed reflections. The structures of the two compounds are quite similar: the benzoanilide moieties are nearly planar; the quaternary nitrogen side chains show the same *trans-trans-gauche* trend for the corresponding torsional angles. The only remarkable difference lies in

the orientation of their alkyloxy chains. In the octyloxy derivative the ends of the two side chains are spatially closely related giving to the molecule a 'loop' conformation.

Introduction. The failure of atropine-like substances to antagonize increased motility and/or tone of the gastro-intestinal tract without affecting other organs and structures is ascribable to their lack of selectivity. It has been shown that spasmolytics, which present a quaternary ammonium group in their structure, act, after oral administration, at the level of the gastro-intestinal tract with less central effects (Burger, 1970). Following this route a new series of molecules, all of them showing smooth-muscle relaxant activity, has been synthesized (Ghelardoni, Pestellini, Pisanti & Volterra, 1973). In these molecules the influence of the

alkyloxy chain on the activity is particularly marked. The position as well as the length of the chain play a significant role; the activity increases if the alkyloxy chain is *ortho* to the amide group and it reaches a maximum for the octyloxy derivative (I). The octyloxy derivative (I) is almost completely devoid of central, ocular and cardiovascular atropine-like side effects when administered in doses that produce spasmolytic effects comparable with those of *N*-butylscopolammonium bromide (Maggi, Grimaldi, Volterra & Meli, 1983; Maggi & Meli, 1983; Maggi, Manzini & Meli, 1983).

NMR investigations have shown that (I) exists in dimethyl sulfoxide predominantly in conformations where rings *A* and *B* form angles of ± 53 and $\pm 55^\circ$ respectively with the plane of the amide bridge (Sega, Gaggelli & Valensin, 1985). Moreover, a dipolar connection exists between the hydrogens of the terminal methyl of the octyloxy chain and the methyls of the ethyl groups bonded to the quaternary nitrogen (Sega, Gaggelli & Valensin, 1985). These interactions are absent in the propyloxy derivative (II) (Sega, Ghelardoni, Pestellini, Pogliani & Valensin, 1984).



Considering these features, we were interested in determining the molecular structure of (I); in particular in establishing the conformation of the octyloxy chain and comparing it with that found in solution. For the same reasons we also determined the molecular structure of the propyloxy derivative (II).

Experimental. Compound (I): prismatic crystals obtained by slow crystallization from dimethyl sulfoxide. Philips PW 1100 diffractometer; lattice parameters determined using 25 reflections (θ range: $4.30-8.73^\circ$); 4686 unique measured reflections, $2\theta \leq 50^\circ$, $\theta-2\theta$ scan technique, $-17 \leq h \leq 17$, $0 \leq k \leq 18$, $0 \leq l \leq 10$; 1379 unique reflections with $I \geq 3\sigma(I)$; three standard reflections (421 , $4\bar{2}1$, $0\bar{2}\bar{3}$), no significant variation of intensity; Lp correction, absorption correction with numerical method of *SHELX76* program (Sheldrick, 1976), transmission factor between 0.39 and 0.67; parallelepipedal crystal, $0.70 \times 0.60 \times 0.30$ mm, delimited by (100), (010) and (001) and their centrosymmetric faces; scattering factors for non-hydrogen atoms (*International Tables for X-ray Crystallography*, 1974, p. 99) and for H atoms (Stewart, Davidson &

Simpson, 1965), anomalous-dispersion corrections (*International Tables for X-ray Crystallography*, 1974, p. 149), $R_{\text{int}} = 0.052$; Patterson and Fourier methods; full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized; anisotropic thermal parameters only for Br on account of the rather low reflections/parameters ratio, isotropic for other atoms; H atoms (except those of octyloxy chain on account of high thermal factors) introduced in calculated positions with overall U of 0.05 \AA^2 and not refined; 150 refined parameters; max. $\Delta/\sigma = 0.75$, final $R = 0.079$, $wR = 0.079$ (low accuracy of analysis due to high thermal motion of octyloxy chain), goodness of fit $S = 2.75$, $w = 1/\sigma^2$, min. and max. $\Delta\rho$ excursions -0.78 and 0.69 e \AA^{-3} respectively; GOULD-SEL 32/70 computer, *SHELX76* program (Sheldrick, 1976).

Compound (II): prismatic crystals obtained by slow crystallization from dimethyl sulfoxide. Philips PW 1100 diffractometer; lattice parameters determined using 25 reflections (θ range $4.02-8.30^\circ$), 3052 unique measured reflections, $2\theta \leq 50^\circ$, $\theta-2\theta$ scan technique, $-18 \leq h \leq 18$, $-9 \leq k \leq 9$, $0 \leq l \leq 11$; 2114 unique reflections with $I \geq 3\sigma(I)$; three standard reflections (312 , 212 , 401), no appreciable variation of intensity; Lp correction, absorption correction with numerical method of *SHELX76* program (Sheldrick, 1976); transmission factor between 0.72 and 0.89, parallelepipedal crystal, $0.90 \times 0.20 \times 0.07$ mm, delimited by (100), (010) and (001) and their centrosymmetric faces; scattering factors for non-hydrogen atoms (*International Tables for X-ray Crystallography*, 1974, p. 99) and for H atoms (Stewart, Davidson & Simpson, 1965), anomalous-dispersion corrections (*International Tables for X-ray Crystallography*, 1974, p. 149), $R_{\text{int}} = 0.048$; Patterson and Fourier methods; full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized; anisotropic thermal parameters for non-hydrogen atoms; H linked to N(2) (from ΔF synthesis) and other hydrogens (calculated) isotropic U of 0.05 \AA^2 ; max. $\Delta/\sigma = 0.82$, final $R = 0.047$, $wR = 0.044$, goodness of fit $S = 2.40$, $w = 1/\sigma^2$; 280 refined parameters; min. and max. $\Delta\rho$ excursions -0.35 and 0.37 e \AA^{-3} respectively; GOULD-SEL 32/70 computer, *SHELX76* program (Sheldrick, 1976).

Discussion. Final atomic coordinates are given in Tables 1 and 2;* bond lengths and angles in Tables 3 and 4 and Tables 5 and 6, respectively. The *ORTEPII* (Johnson, 1976) plots of the molecules are shown in Figs. 1 and 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42629 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for compound (I)

For Br $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$.

	x	y	z	U_{eq}/U_{iso}
Br	-344 (1)	3831 (1)	-2503 (2)	82 (1)
O(1)	-2055 (6)	3313 (6)	3691 (10)	75 (3)
O(2)	-2407 (6)	2427 (8)	5246 (12)	93 (4)
O(3)	-5892 (7)	3790 (9)	4982 (16)	139 (5)
O(4)	-5905 (8)	5535 (8)	1903 (14)	120 (5)
N(1)	-636 (6)	3965 (8)	2553 (12)	71 (4)
N(2)	-5244 (8)	4404 (8)	3514 (15)	87 (4)
C(1)	-250 (10)	4053 (12)	1257 (18)	116 (7)
C(2)	-137 (10)	4073 (12)	3797 (19)	122 (7)
C(3)	404 (12)	3484 (16)	3954 (26)	196 (11)
C(4)	-1165 (10)	4641 (12)	2628 (20)	113 (7)
C(5)	-1611 (12)	4658 (15)	1230 (22)	181 (10)
C(6)	-1002 (8)	3155 (9)	2477 (15)	72 (5)
C(7)	-1363 (8)	2944 (10)	3802 (16)	77 (5)
C(8)	-2534 (10)	2999 (11)	4442 (17)	77 (5)
C(9)	-3231 (9)	3381 (9)	4287 (16)	63 (5)
C(10)	-3347 (10)	3962 (10)	3249 (17)	88 (6)
C(11)	-4043 (10)	4279 (11)	3091 (18)	84 (6)
C(12)	-4556 (9)	4053 (10)	3823 (17)	69 (5)
C(13)	-4449 (10)	3465 (10)	4840 (17)	76 (5)
C(14)	-3771 (9)	3145 (9)	5054 (17)	72 (5)
C(15)	-5840 (11)	4255 (12)	4024 (21)	92 (6)
C(16)	-6510 (10)	4721 (11)	3501 (19)	94 (6)
C(17)	-7072 (12)	4512 (14)	4143 (23)	121 (7)
C(18)	-7745 (12)	4934 (13)	3729 (23)	129 (8)
C(19)	-7681 (12)	5482 (13)	2803 (21)	112 (7)
C(20)	-7133 (12)	5704 (14)	2125 (23)	121 (7)
C(21)	-6461 (11)	5301 (12)	2507 (21)	102 (7)
C(22)	-5902 (13)	6181 (16)	917 (24)	137 (8)
C(23)	-5245 (14)	6217 (16)	358 (26)	163 (9)
C(24)	-4656 (17)	6515 (17)	1376 (31)	173 (11)
C(25)	-4005 (27)	6513 (25)	431 (46)	268 (19)
C(26)	-3487 (33)	6589 (30)	1100 (54)	284 (24)
C(27)	-2826 (26)	6666 (28)	-118 (50)	288 (22)
C(28)	-2376 (38)	6849 (36)	938 (70)	393 (33)
C(29)	-1827 (32)	7028 (32)	-570 (62)	413 (30)

Table 2. Positional ($\times 10^4$) and thermal ($\text{\AA}^2 \times 10^3$) parameters for compound (II)

	x	y	z	U_{eq}
Br	-5614 (1)	2248 (1)	-7636 (1)	67 (1)
O(1)	-3576 (5)	3135 (8)	-3400 (7)	66 (5)
O(2)	-2948 (5)	2801 (10)	-5380 (10)	86 (7)
O(3)	872 (7)	6340 (13)	-2682 (13)	123 (9)
O(4)	254 (6)	8309 (10)	1094 (10)	86 (7)
N(1)	-5482 (6)	3003 (12)	-2492 (9)	55 (6)
N(2)	-158 (7)	6695 (12)	-1289 (13)	70 (8)
C(1)	-4827 (9)	3097 (19)	-1216 (16)	96 (12)
C(2)	-6544 (13)	1145 (28)	-3835 (23)	154 (21)
C(3)	-5849 (11)	1352 (21)	-2541 (19)	109 (14)
C(4)	-6750 (9)	4041 (18)	-1252 (15)	92 (11)
C(5)	-6175 (8)	4282 (15)	-2388 (13)	72 (9)
C(6)	-5088 (7)	3375 (13)	-3806 (11)	55 (8)
C(7)	-4327 (7)	2374 (14)	-4074 (12)	58 (8)
C(8)	-2934 (8)	3317 (14)	-4188 (14)	57 (9)
C(9)	-2219 (7)	4238 (12)	-3438 (11)	52 (7)
C(10)	-2256 (8)	4927 (13)	-2095 (12)	66 (8)
C(11)	-1562 (8)	5747 (14)	-1413 (13)	68 (9)
C(12)	-833 (7)	5865 (13)	-2064 (12)	58 (8)
C(13)	-801 (8)	5215 (14)	-3418 (13)	66 (9)
C(14)	-1491 (8)	4410 (14)	-4099 (13)	65 (9)
C(15)	634 (10)	6892 (16)	-1623 (16)	75 (10)
C(16)	1213 (9)	7897 (16)	-628 (15)	75 (10)
C(17)	2014 (10)	8136 (17)	-1038 (16)	89 (11)
C(18)	2606 (11)	9084 (19)	-177 (19)	100 (13)
C(19)	2414 (11)	9752 (19)	1065 (19)	103 (13)
C(20)	1629 (11)	9540 (18)	1520 (18)	93 (13)
C(21)	1049 (10)	8587 (17)	670 (16)	77 (11)
C(22)	73 (9)	8857 (17)	2478 (15)	81 (11)
C(23)	-791 (9)	8254 (18)	2723 (15)	89 (12)
C(24)	-1041 (10)	8833 (20)	4167 (16)	122 (14)

The interactions between the organic cation and the bromine anion occur through hydrogens; the interactions involving Br...H distances lower than the sum of van der Waals radii [3.15 Å from $f_H = 1.20$ and $f_{Br} = 1.95$ Å; Pauling (1960)] are reported in Table 7. In this table are also reported the intra- and intermolecular C—H...O contacts that could be considered as hydrogen bonds. Intermolecular hydrogen bonds involving the most electronegative atoms (nitrogen and oxygen) as donors and acceptors are not present.

Table 3. Bond lengths (Å) for compound (I)

O(1)—C(7)	1.45 (2)	C(10)—C(11)	1.42 (2)
O(1)—C(8)	1.31 (2)	C(11)—C(12)	1.30 (2)
O(2)—C(8)	1.24 (2)	C(12)—C(13)	1.38 (2)
O(3)—C(15)	1.21 (2)	C(13)—C(14)	1.39 (2)
O(4)—C(21)	1.30 (2)	C(15)—C(16)	1.54 (2)
O(4)—C(22)	1.43 (2)	C(16)—C(17)	1.32 (2)
N(1)—C(1)	1.49 (2)	C(16)—C(21)	1.36 (2)
N(1)—C(2)	1.47 (2)	C(17)—C(18)	1.49 (2)
N(1)—C(4)	1.52 (2)	C(18)—C(19)	1.28 (2)
N(1)—C(6)	1.53 (2)	C(19)—C(20)	1.32 (2)
N(2)—C(12)	1.44 (2)	C(20)—C(21)	1.46 (2)
N(2)—C(15)	1.29 (2)	C(22)—C(23)	1.40 (3)
C(2)—C(3)	1.42 (2)	C(23)—C(24)	1.51 (3)
C(4)—C(5)	1.52 (2)	C(24)—C(25)	1.59 (4)
C(6)—C(7)	1.52 (2)	C(25)—C(26)	1.14 (6)
C(8)—C(9)	1.47 (2)	C(26)—C(27)	1.78 (6)
C(9)—C(10)	1.39 (2)	C(27)—C(28)	1.30 (7)
C(9)—C(14)	1.36 (2)	C(28)—C(29)	1.86 (7)

Table 4. Bond lengths (Å) for compound (II)

O(1)—C(7)	1.438 (10)	C(9)—C(10)	1.395 (12)
O(1)—C(8)	1.343 (11)	C(9)—C(14)	1.390 (12)
O(2)—C(8)	1.199 (11)	C(10)—C(11)	1.394 (13)
O(3)—C(15)	1.197 (12)	C(11)—C(12)	1.381 (13)
O(4)—C(21)	1.394 (13)	C(12)—C(13)	1.391 (12)
O(4)—C(22)	1.441 (12)	C(13)—C(14)	1.383 (13)
N(1)—C(1)	1.496 (13)	C(15)—C(16)	1.494 (14)
N(1)—C(3)	1.490 (14)	C(16)—C(17)	1.392 (15)
N(1)—C(5)	1.529 (12)	C(16)—C(21)	1.400 (14)
N(1)—C(6)	1.487 (11)	C(17)—C(18)	1.414 (15)
N(2)—C(12)	1.411 (13)	C(18)—C(19)	1.360 (16)
N(2)—C(15)	1.350 (14)	C(19)—C(20)	1.383 (16)
C(2)—C(3)	1.555 (17)	C(20)—C(21)	1.399 (16)
C(4)—C(5)	1.504 (13)	C(22)—C(23)	1.513 (14)
C(6)—C(7)	1.504 (12)	C(23)—C(24)	1.537 (14)
C(8)—C(9)	1.486 (13)		

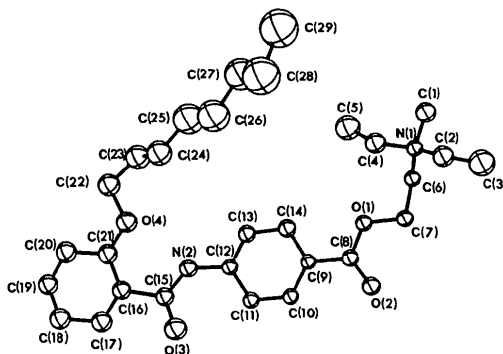


Fig. 1. View of the octyloxy derivative (I) with the atomic labelling system.

In the molecular structure of (I) the largest displacements of the atoms defining the two benzene planes are 0.012 (21) and 0.009 (17) Å for rings *A* and *B*, respectively; in the molecular structure of (II), the same displacements are -0.012 (14) and

Table 7. Intramolecular and intermolecular Br...H interactions (Å) and C—H...O contacts

(I)		(II)	
Br...H(11 ^b)	2.80 (17)	Br...H(11 ^{bb})	2.73 (16)
Br...H(13 ^b)	2.89 (19)	Br...H(31 ^{vb})	2.70 (17)
Br...H(21 ^{bb})	2.65 (18)	Br...H(42 ^{vb})	2.95 (14)
Br...H(22 ^{bb})	2.84 (20)	Br...H(61 ^{vb})	2.97 (11)
Br...H(41 ^b)	2.88 (20)	Br...H(62 ^b)	2.86 (10)
Br...H(61 ^b)	2.62 (15)	Br...H(71 ^b)	2.91 (11)
Br...H(191 ^b)	2.90 (23)	Br...H(191 ^{vb})	2.84 (17)
H(42)...O(1 ^b)	2.35 (20)	H(13)...O(1 ^b)	2.53 (16)
H(52)...O(1 ^b)	2.83 (22)	H(71)...O(2 ^b)	2.23 (11)
H(72)...O(2 ^b)	2.39 (16)	H(101)...O(1 ^b)	2.42 (11)
H(101)...O(1 ^b)	2.37 (17)	H(131)...O(3 ^b)	2.18 (12)
H(131)...O(3 ^b)	2.16 (18)	H(141)...O(2 ^b)	2.53 (13)
H(171)...O(3 ^b)	2.24 (23)	H(171)...O(3 ^b)	2.36 (15)
H(62)...O(2 ^b)	2.44 (15)		

Symmetry code: (i) *x*, *y*, *z*; (ii) $-x$, $-y+1$, $-z$; (iii) *x*, *y*, $z-1$; (iv) *x*, $-y+\frac{1}{2}$, $z-\frac{1}{2}$; (v) $-x-1$, $-y+1$, $-z$; (vi) $-x-1$, $-y$, $-z-1$; (vii) $-x-1$, $-y+1$, $-z-1$; (viii) $x-1$, $y-1$, $z-1$.

Table 5. Bond angles (°) for compound (I)

C(8)—O(1)—C(7)	117 (1)	C(13)—C(12)—C(11)	120 (2)
C(22)—O(4)—C(21)	124 (2)	C(14)—C(13)—C(12)	117 (2)
C(2)—N(1)—C(1)	109 (1)	C(13)—C(14)—C(9)	122 (2)
C(4)—N(1)—C(1)	110 (1)	N(2)—C(15)—O(3)	122 (2)
C(4)—N(1)—C(2)	105 (1)	C(16)—C(15)—O(3)	117 (2)
C(6)—N(1)—C(1)	108 (1)	C(16)—C(15)—N(2)	121 (2)
C(6)—N(1)—C(2)	114 (1)	C(17)—C(16)—C(15)	113 (2)
C(6)—N(1)—C(4)	111 (1)	C(21)—C(16)—C(15)	119 (2)
C(15)—N(2)—C(12)	131 (2)	C(18)—C(17)—C(16)	118 (2)
C(3)—C(2)—N(1)	114 (2)	C(19)—C(18)—C(17)	113 (2)
C(5)—C(4)—N(1)	107 (2)	C(20)—C(19)—C(18)	131 (3)
C(7)—C(6)—N(1)	114 (1)	C(21)—C(20)—C(19)	117 (2)
C(6)—C(7)—O(1)	108 (1)	C(20)—C(21)—C(16)	113 (2)
O(2)—C(8)—O(1)	122 (2)	C(16)—C(21)—O(4)	128 (2)
C(9)—C(8)—O(1)	116 (2)	C(20)—C(21)—O(4)	118 (2)
C(9)—C(8)—O(2)	122 (2)	C(23)—C(22)—O(4)	109 (2)
C(10)—C(9)—C(8)	118 (2)	C(24)—C(23)—C(22)	114 (2)
C(14)—C(9)—C(8)	122 (2)	C(25)—C(24)—C(23)	102 (3)
C(11)—C(10)—C(9)	116 (2)	C(26)—C(25)—C(24)	111 (5)
C(12)—C(11)—C(10)	124 (2)	C(27)—C(26)—C(25)	106 (5)
C(11)—C(12)—N(2)	119 (2)	C(28)—C(27)—C(26)	88 (4)
C(13)—C(12)—N(2)	121 (2)	C(29)—C(28)—C(27)	80 (4)

Table 6. Bond angles (°) for compound (II)

C(7)—O(1)—C(8)	117.3 (7)	C(11)—C(12)—C(13)	120.1 (10)
C(21)—O(4)—C(22)	119.5 (9)	C(11)—C(12)—N(2)	116.1 (10)
C(1)—N(1)—C(3)	108.4 (9)	C(13)—C(12)—N(2)	123.8 (10)
C(1)—N(1)—C(5)	110.1 (8)	C(12)—C(13)—C(14)	120.0 (10)
C(1)—N(1)—C(6)	109.3 (8)	C(9)—C(14)—C(13)	120.5 (10)
C(3)—N(1)—C(5)	110.2 (9)	N(2)—C(15)—O(3)	122.9 (12)
C(3)—N(1)—C(6)	111.9 (9)	N(2)—C(15)—C(16)	116.9 (12)
C(5)—N(1)—C(6)	106.9 (7)	O(3)—C(15)—C(16)	120.1 (13)
C(12)—N(2)—C(15)	128.4 (10)	C(15)—C(16)—C(21)	127.6 (13)
C(2)—C(3)—N(1)	112.0 (13)	C(15)—C(16)—C(17)	115.4 (12)
C(4)—C(5)—N(1)	117.2 (9)	C(17)—C(16)—C(21)	117.0 (12)
C(7)—C(6)—N(1)	117.1 (8)	C(16)—C(17)—C(18)	119.9 (13)
C(6)—C(7)—O(1)	109.1 (8)	C(17)—C(18)—C(19)	121.1 (14)
O(1)—C(8)—O(2)	123.0 (10)	C(18)—C(19)—C(20)	121.1 (15)
C(9)—C(8)—O(1)	113.1 (9)	C(19)—C(20)—C(21)	117.4 (14)
C(9)—C(8)—O(2)	123.9 (11)	C(20)—C(21)—C(16)	123.5 (13)
C(8)—C(9)—C(10)	122.0 (10)	C(20)—C(21)—O(4)	119.7 (12)
C(8)—C(9)—C(14)	118.7 (9)	C(16)—C(21)—O(4)	116.8 (12)
C(10)—C(9)—C(14)	119.3 (9)	C(23)—C(22)—O(4)	108.5 (10)
C(9)—C(10)—C(11)	120.2 (10)	C(24)—C(23)—C(22)	111.5 (11)
C(10)—C(11)—C(12)	119.9 (10)		

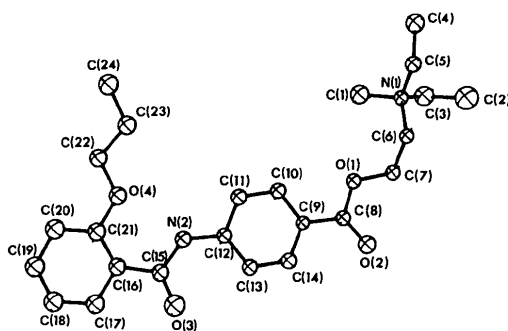


Fig. 2. View of the propyloxy derivative (II) with the atomic labelling system.

-0.013 (11) Å for rings *A* and *B*, respectively. The conformation of the benzanilide moiety is close to planarity in both structures: in (I) the angles between rings *A* and *B* and the amide plane are 6 (1) and 5 (1)°, respectively; in (II) the same angles are 3 (1) and 6 (1)°, respectively. An intramolecular hydrogen bond is present between O(4) and the amide nitrogen in both molecular structures: (I) O(4)...H = 1.78 (14) Å; (II) O(4)...H = 1.86 (11) Å.

The quaternary ammonium side chain strongly resembles that of procaine hydrochloride and related compounds (Kashino, Ikeda & Haisa, 1982); the sequence of torsional angles: τ_1 [C(9)—C(8)—O(1)—C(7)], τ_2 [C(8)—O(1)—C(7)—C(6)], τ_3 [O(1)—C(7)—C(6)—N(1)], which are -179 (1) (I) and 175 (1)° (II), 157 (1) (I) and -132 (1)° (II), 84 (1) (I) and -89 (1)° (II), respectively, shows the same *trans-trans-gauche* 'pharmacophoric pattern' present in these molecules.

The octyloxy side chain of (I) has very high thermal motion as can be seen by the exceptionally high isotropic temperature factors for C(22) to C(29). Owing to this situation the localization, even in assigned positions, of the corresponding hydrogen atoms is unreliable. In contrast, in (II) the favourable reflections/parameters ratio allowed anisotropic temperature factors to be used for all non-hydrogen atoms. The conformations of the two alkyloxy chains are different as can be seen from the first two torsional angles: τ_1 [C(21)—O(4)—C(22)—C(23)] and τ_2 [O(4)—C(22)—C(23)—C(24)], which are -173 (2) (I) and -174 (1)° (II), -72 (3) (I) and -178 (1)° (II), respectively; so the conformation of the propyloxy chain is *trans-trans* while the corresponding atoms in the octyloxy chain are *trans-gauche*.

The conformation found for (I) corresponds to a 'loop' structure with a clear bending of the ends of the two side chains toward each other [C(29)—C(5) = 4.33 (6) Å]. Chains shorter or longer than that of the octyloxy derivative (I) cannot adopt this particular

conformation. This result might support the surprising findings, from the proton two-dimensional nuclear Overhauser effect, of a dipolar connection between the protons of C(29) and those of C(5) [or C(3)] (Sega, Gaggelli & Valensin, 1985).

This stresses the importance of the effect of the length of the alkyloxy side chain on the pharmacological activity.

We wish to thank the Istituto per lo Studio della Stereochimica ed Energetica dei Composti di Coordinazione of Florence for collection of the intensity data and for computer time.

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Structure of 1,8-Bis(1'-H-1',2',3'-triazolyl)naphthalene*

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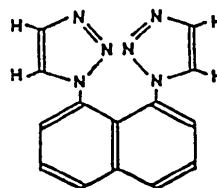
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(Received 20 September 1985; accepted 10 December 1985)

Abstract. C₁₄H₁₀N₆, *M_r* = 262.27, orthorhombic, *Fdd2*, *a* = 5.489 (1), *b* = 25.241 (1), *c* = 17.675 (1) Å, *U* = 2448.6 (2) Å³, *Z* = 8, *D_x* = 1.423 g cm⁻³, *Cu Kα*, *λ* = 1.5418 Å, *μ* = 7.65 cm⁻¹, *F*(000) = 1088, *T* = 295 K, *R* = 0.069 for 411 observed independent reflections. The title compound has twofold symmetry in the molecule. The naphthalene framework is distorted. The fusion angle of 129.0° is large compared with those of other *peri*-substituted naphthalenes. Severe steric repulsion occurs between the two triazole rings which are in a *trans* conformation to each other.

Introduction. Naphthalenes substituted by bulky groups at the 1 and 8 positions (*peri* substituents) exhibit several unique properties in both reactivity and structure. Recently, we have succeeded in synthesizing novel 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazoles (Honda, Nakanishi, Nagawa & Yabe, 1984). These compounds show very interesting rotational isomerism in solution (Nakanishi, Honda, Yabe, Nagawa & Yamamoto, 1986). In the present paper, we describe the crystal and molecular structure of (I), with the intention

of confirming the conformation of the two triazole rings and the distortion of the naphthalene framework.



(I)

Experimental. Title compound synthesized by method previously reported (Honda *et al.*, 1984); needle-shaped colorless crystals obtained by sublimation. Crystal 0.85 × 0.30 × 0.08 mm. Data collection: Rigaku four-circle diffractometer, graphite-monochromated *Cu Kα* radiation; cell parameters determined by least squares from 2θ values for 25 reflections (84 < 2θ < 110°); systematic absences of *hkl* for *h + k* and *k + l* odd, *Ok_l* for *k + l* ≠ 4*n* and *h0l* for *l + h* ≠ 4*n*; intensity data 4 < 2θ < 120°, ω-2θ scan, 0 ≤ *h* ≤ 7, 0 ≤ *k* ≤ 35, 0 ≤ *l* ≤ 24, scan speed 2° min⁻¹; three standard reflections (040, 020 and 220) showed only statistical

* IUPAC name: 1,1'-(1,8-naphthylene)di-1H-1,2,3-triazole.