Fig. 2. Packing diagram viewed down **b**. Double lines indicate hydrogen bonds.

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H⋯H contact is 2·185 (6) Å. The packing is different from other lynestrenol derivatives (Griffin, Duax & Weeks, 1984) and can be described as $M\,a_{36}b_{19}c_{54}\,211$ (Duax & Norton, 1975), indicating that the molecules are packed two thick, one wide and one long, with the steroid length parallel to the ac diagonal.

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Structure of Bis(4,5,6,7,8,9-hexafluoronaphtho[2,1-*b*]fur-2-yl)methane

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Abstract. $C_{25}H_4F_{12}O_2$, $M_r = 564\cdot29$, monoclinic, $C2$, $a = 31\cdot86$ (3), $b = 4\cdot600$ (5), $c = 6\cdot93$ (1) Å, $\beta = 101\cdot2$ (1)°, $V = 995$ (6) Å^3, $Z = 2$, $D_m = 1\cdot90$, $D_x = 1\cdot88$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot139$ mm^{-1}, $F(000) = 556$, room temperature, $R = 0\cdot054$ for 643 observed diffractometer-measured intensities. The two halves of the molecule are related by a crystallographic twofold axis passing through the central C atom. Within each half of the molecule, the furan ring is tilted very slightly out of the plane of the naphthalene skeleton. The molecule is probably only partly delocalized, as some bonds are distinctly shorter than others.

Introduction. The compound was prepared as part of a continuing investigation into the preparation of partially fluorinated furan derivatives (Brooke & Wallis, 1982).

Its structure was determined in order to confirm the unexpected reaction product obtained when 1,3,4,-5,6,7,8-heptafluoro-2-naphthyl 2-propynyl ether was heated with 1,1,2-trichloro-1,2,2-trifluoroethane.

Experimental. Accurate cell dimensions obtained from least-squares refinement of positions of 12 strong reflections measured on a two-circle diffractometer (Mo $K\alpha$, θ_{min} 5, θ_{max} 15°, μ_{min} 0, μ_{max} 18°). Density measured by flotation and indicated $Z = 2$. 953 reflections measured on a Stoe Stadi-2 two-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation; crystal 0·04 × 0·54 × 0·19 mm, layers $k = 0$–5, $h = 0$–36, $l \pm 8$, max. $\sin\theta/\lambda = 0\cdot595$ Å^{-1}, separate standard for each layer measured every 10 reflections (intensity variation < 1%). Of the 933 unique reflections ($R_{int} = 0\cdot030$) 643 with $F > 4\sigma(F)$ used for

Table 1. *Final atomic coordinates* ($\times 10^4$) *and isotropic temperature factors* ($\text{Å}^2 \times 10^3$)

	x	y	z	$U_{eq}*$ or U
C(1)†	5000	4362 (45)	0000	57 (8)*
C(2)	4625 (3)	6162 (37)	246 (13)	51 (5)*
C(3)	4274 (3)	7000 (35)	9022 (13)	33 (5)*
C(4)	4062 (3)	9022 (34)	103 (13)	41 (5)*
C(5)	4318 (3)	9251 (38)	1968 (14)	50 (5)*
C(6)	4222 (3)	1035 (39)	3443 (13)	49 (6)*
C(7)	3867 (3)	2632 (37)	3087 (14)	60 (6)*
C(8)	3578 (3)	2668 (fixed)	1212 (12)	46 (5)*
C(9)	3683 (3)	803 (32)	9707 (14)	53 (6)*
C(10)	3401 (3)	810 (38)	7857 (12)	45 (5)*
C(11)	3051 (3)	2574 (37)	7458 (15)	56 (7)*
C(12)	2959 (3)	4339 (41)	8915 (17)	60 (6)*
C(13)	3210 (3)	4372 (33)	748 (15)	62 (6)*
O(1)	4668 (2)	7441 (31)	2094 (9)	49 (4)*
F(1)	4488 (2)	1082 (34)	5213 (7)	73 (4)*
F(2)	3782 (2)	4403 (33)	4519 (9)	80 (4)*
F(3)	3105 (2)	6163 (33)	2121 (9)	83 (4)*
F(4)	2612 (2)	6077 (32)	8532 (10)	83 (4)*
F(5)	2979 (2)	2549 (32)	5680 (9)	73 (4)*
F(6)	3493 (2)	9067 (32)	6422 (8)	62 (3)*
H(1)	4942 (28)	2883 (243)	9043 (109)	37 (27)
H(2)	4143 (21)	6784 (193)	7612 (192)	20 (22)

$$* \ U_{eq} = \tfrac{1}{3}(\textstyle\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j).$$
† Special position.

refinement. No absorption correction applied. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined using *SHELX*76 (Sheldrick, 1976) full-matrix least squares on F magnitudes. The y coordinate of C(8) was fixed to define the origin, this atom being chosen due to its position near the centre of the unique half molecule. H atoms located from difference map. All atoms except H given anisotropic U_{ij}. Individual scale factors for data-collection layers introduced in final cycles of refinement. Unit weights used throughout as a weighting scheme did not improve R. Max. Δ/σ 0·09 for heavy atoms [0·11 for U of H(1) and 0·23 for U of H(2)], $R = 0\cdot054$ and largest features on final difference map $\pm 0\cdot3$ e Å^{-3}. Atomic scattering factors from *SHELX*76. Final atomic positions, U, and U_{eq} values are given in Table 1, bond distances and angles in Table 2.*

Discussion. The structure determination shows that the two halves of the molecule are related by a crystallographic twofold axis passing through the central C atom C(1) (Fig. 1). Each half of the molecule is approximately planar, with the maximum deviation from the best least-squares plane through the C, O and F atoms being 0·152 (2) Å [C(1)]. However, the best least-squares plane through the furan ring is tilted away from the best least-squares plane through the naphthalene skeleton by 1·77 (2)°. The F atoms are

coplanar with the naphthalene skeleton, the largest deviation from the least-squares plane being only 0·039 (2) Å [F(4)].

The bis(4,5,6,7,8,9-hexafluoronaphtho[2,1-*b*]fur-2-yl)methane molecule shows little thermal disorder: the two H atoms were easily located from a difference map and their positions and isotropic temperature factors were refined without difficulty. There is no evidence for libration of F atoms such as occurs in 1,8-difluoro-naphthalene (Meresse, Courseille, Leroy & Chanh, 1975). Presumably the addition of an extra furan ring to the naphthalene skeleton increases the rigidity of the molecule and reduces the level of molecular vibrations.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)–C(2)	1·49 (1)	C(10)–C(11)	1·36 (2)
C(2)–C(3)	1·32 (1)	C(11)–C(12)	1·37 (2)
C(2)–O(1)	1·39 (1)	C(12)–C(13)	1·36 (1)
C(3)–C(4)	1·44 (1)	C(13)–C(8)	1·39 (1)
C(4)–C(5)	1·39 (1)	C(6)–F(1)	1·35 (1)
C(5)–C(6)	1·39 (2)	C(13)–F(3)	1·35 (1)
C(6)–C(7)	1·33 (2)	C(12)–F(4)	1·35 (1)
C(7)–C(8)	1·44 (1)	C(11)–F(5)	1·34 (1)
C(8)–C(9)	1·44 (1)	C(7)–F(2)	1·35 (1)
C(9)–C(4)	1·44 (1)	C(10)–F(6)	1·35 (1)
C(5)–O(1)	1·38 (1)	C(1)–H(1)	0·94 (9)
C(9)–C(10)	1·41 (1)	C(3)–H(2)	0·99 (7)
		F(4)···F(3′)	3·18 (2)

C(1)–C(2)–C(3)	133·3 (8)	C(13)–C(8)–C(9)	118·1 (8)
C(2)–O(1)–C(5)	105·3 (8)	C(9)–C(10)–F(6)	118·4 (9)
C(2)–C(3)–C(4)	106·1 (8)	C(11)–C(10)–F(6)	119·2 (9)
C(3)–C(4)–C(5)	106·4 (9)	C(10)–C(11)–F(5)	120·5 (11)
C(4)–C(5)–O(1)	109·5 (10)	C(12)–C(11)–F(5)	120·4 (11)
C(4)–C(5)–C(6)	123·6 (11)	C(11)–C(12)–F(4)	119·4 (10)
C(5)–C(6)–C(7)	119·4 (9)	C(13)–C(12)–F(4)	119·2 (10)
C(6)–C(7)–C(8)	123·1 (10)	C(12)–C(13)–F(3)	118·7 (10)
C(7)–C(8)–C(9)	116·7 (8)	C(8)–C(13)–F(3)	119·6 (9)
C(8)–C(9)–C(4)	120·4 (8)	C(7)–C(8)–C(13)	125·2 (9)
C(9)–C(4)–C(5)	116·9 (10)	C(4)–C(9)–C(10)	122·3 (10)
C(8)–C(9)–C(10)	117·3 (9)	C(8)–C(7)–F(2)	118·1 (10)
C(9)–C(10)–C(11)	122·3 (10)	C(6)–C(7)–F(2)	118·8 (10)
C(10)–C(11)–C(12)	119·1 (10)	C(5)–C(6)–F(1)	119·1 (11)
C(11)–C(12)–C(13)	121·4 (11)	C(7)–C(6)–F(1)	121·4 (11)
C(12)–C(13)–C(8)	121·6 (9)	C(6)–C(5)–O(1)	126·9 (9)
O(1)–C(2)–C(3)	112·7 (9)	C(1)–C(2)–O(1)	113·7 (7)

$$(') = \tfrac{1}{2} - x, \tfrac{1}{2} + y, 1 - z.$$
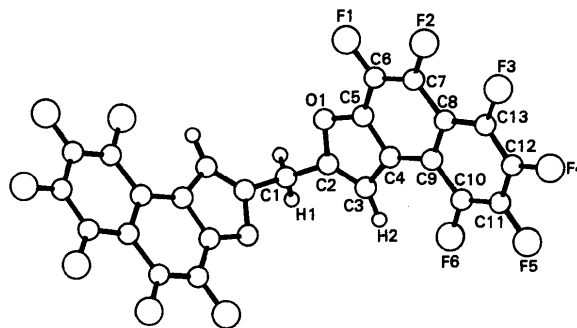
Fig. 1. Atom numbering scheme.

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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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) Å^3, $Z = 4$, $D_x = 1.239$ 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) Å^3, $Z = 2$, $D_x = 1.332$ 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 synthetized (Ghelardoni, Pestellini, Pisanti & Volterra, 1973). In these molecules the influence of the