

Table 1. Atomic parameters  $x$ ,  $y$ ,  $z$  and  $B_{\text{iso}}$  values

E.s.d.'s refer to the last digit printed.				
	$x$	$y$	$z$	$B_{\text{iso}}^*(\text{\AA}^2)$
O(1)	0.6623 (3)	0.4171	0.6727 (2)	4.92 (9)
O(2)	0.4723 (3)	0.2218 (4)	0.7017 (2)	4.74 (8)
O(3)	0.5325 (2)	0.3685 (4)	1.0185 (2)	3.96 (8)
O(4)	1.0236 (3)	0.0825 (4)	1.3094 (2)	4.92 (9)
C(1)	0.7289 (3)	0.2359 (4)	0.8916 (3)	3.10 (8)
C(2)	0.6787 (3)	0.2708 (4)	1.0283 (3)	3.11 (9)
C(3)	0.7780 (3)	0.2234 (4)	1.1701 (3)	3.37 (9)
C(4)	0.9281 (3)	0.1314 (5)	1.1701 (3)	3.46 (9)
C(5)	0.9786 (3)	0.0949 (5)	1.0348 (3)	3.45 (9)
C(6)	0.8819 (3)	0.1485 (4)	0.8931 (3)	3.30 (9)
C(7)	0.6204 (3)	0.3034 (5)	0.7447 (3)	3.33 (9)
C(8)	0.3530 (5)	0.2827 (7)	0.5661 (4)	5.63 (16)
C(9)	0.9394 (5)	0.1055 (6)	0.7479 (4)	5.16 (14)
C(10)	0.7305 (5)	0.2734 (6)	1.3182 (4)	4.83 (14)
C(11)	0.3825 (4)	0.2798 (6)	1.0400 (5)	5.13 (15)

\*  $B_{\text{iso}}$  is the mean of the principal axes of the thermal ellipsoid.

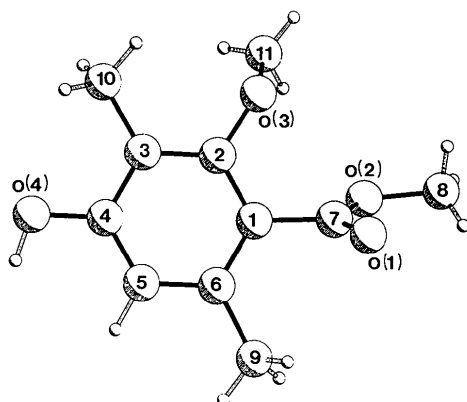


Fig. 1. PLUTO (Motherwell & Clegg, 1978) plot of (2) showing the numbering scheme.

ing the 4-hydroxy substituent and the carboxylate carbonyl group (see Table 2) is also similar to that found in the above-mentioned compound (Nicolaou *et al.*, 1988), and to that found in acetylcholine  $\beta$ -resorcyate (Jensen, 1975) and (1) (Brehm, Stoeckli-Evans, Tabacchi & Bürgi, 1983).

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## Structure of 6,7,8,9,10,11-Hexafluoro-2,4-diphenyl-3H-naphtho[2,1-*b*]azepine

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**Abstract.**  $\text{C}_{26}\text{H}_{13}\text{F}_6\text{N}$ ,  $M_r = 453.39$ , triclinic,  $P\bar{1}$ ,  $a = 13.59$  (1),  $b = 10.62$  (1),  $c = 7.59$  (1)  $\text{\AA}$ ,  $\alpha = 110.0$  (1),  $\beta = 106.2$  (1),  $\gamma = 91.4$  (1) $^\circ$ ,  $V = 979.74$   $\text{\AA}^3$ ,  $Z = 2$ ,

Table 2. Final distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

O(1)—C(7)	1.197 (4)	C(1)—C(7)	1.500 (4)
O(2)—C(7)	1.322 (3)	C(2)—C(3)	1.387 (4)
O(2)—C(8)	1.445 (4)	C(3)—C(4)	1.397 (4)
O(3)—C(2)	1.382 (3)	C(3)—C(10)	1.507 (4)
O(3)—C(11)	1.432 (4)	C(4)—C(5)	1.385 (4)
O(4)—C(4)	1.361 (3)	C(5)—C(6)	1.393 (4)
C(1)—C(2)	1.393 (4)	C(6)—C(9)	1.506 (4)
C(1)—C(6)	1.396 (4)	O(4)—H(O4)	0.91 (6)
O(4)···O(1')	2.790 (3)	O(1')···H(O4)	1.92 (6)
C(7)—O(2)—C(8)	117.0 (3)	O(4)—C(4)—C(3)	116.8 (3)
C(2)—O(3)—C(11)	115.2 (3)	O(4)—C(4)—C(5)	121.8 (3)
C(2)—C(1)—C(6)	120.5 (2)	C(3)—C(4)—C(5)	121.4 (2)
C(2)—C(1)—C(7)	118.3 (2)	C(4)—C(5)—C(6)	121.4 (3)
C(6)—C(1)—C(7)	121.0 (2)	C(1)—C(6)—C(5)	117.6 (2)
O(3)—C(2)—C(1)	117.1 (2)	C(1)—C(6)—C(9)	122.3 (2)
O(3)—C(2)—C(3)	120.6 (2)	C(5)—C(6)—C(9)	120.1 (3)
C(1)—C(2)—C(3)	122.0 (2)	O(1)—C(7)—O(2)	123.3 (3)
C(2)—C(3)—C(4)	117.0 (2)	O(1)—C(7)—C(1)	124.5 (2)
C(2)—C(3)—C(10)	121.9 (3)	O(2)—C(7)—C(1)	112.3 (3)
C(4)—C(3)—C(10)	121.1 (3)	O(4)—H(O4)···O(1')	159 (5)

Symmetry operation: (i)  $2 - x, -0.5 + y, 2 - z$ .

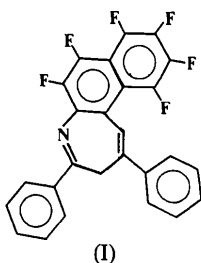
We wish to thank the Swiss National Science Foundation for financial support.

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within the fused 3*H*-azepine ring are similar to those reported in compounds with unfused 3*H*-azepine rings except for a lengthening of the common C—C bond (0.05–0.07 Å longer) and the adjacent C—N bond (0.02–0.05 Å longer). The hexafluoronaphtho group is planar to within 0.083 (5) Å.

**Experimental.** The compound (I) (Brooke & Matthews, 1988) was in the form of colourless platy crystals, tabular on (100). Cell dimensions from



Weissenberg photographs, refined from setting angles of 20 reflexions in the range  $10 < 2\theta < 40^\circ$  on a STADI-2 two-circle diffractometer (graphite-monochromated Mo  $K\alpha$ ). This was also used for measurement of 3615 intensities from a crystal  $0.6 \times 0.4 \times 0.01$  mm for the layers  $hk0$  to  $hk8$ , standard measured every 20 reflexions, intensity variation  $< 5\%$ , corrected by linear interpolation.  $h - 16$  to 15,  $k - 12$  to 11, maximum  $\sin\theta/\lambda = 0.60 \text{ \AA}^{-1}$ ; variable  $\omega$  scan,  $2\theta'$  fixed, stationary background count. Lp correction applied but absorption correction considered unnecessary. The measured intensities were merged to give 3370 unique values ( $R_{\text{int}} = 0.016$ ), 2047 with  $I > 3\sigma(I)$  were considered as

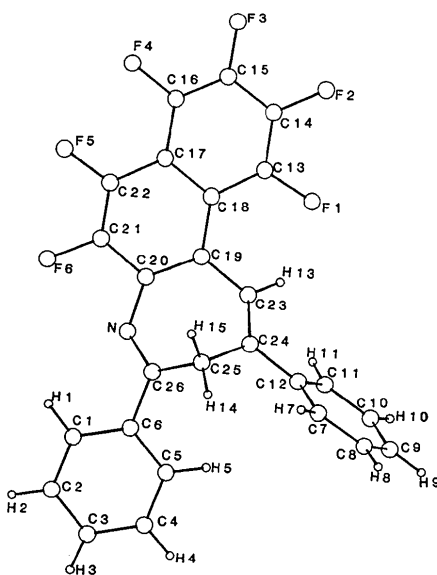


Fig. 1. Molecule of the title compound, with atom labelling.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
C(1)	0.2138 (3)	0.8159 (3)	0.4822 (5)	0.048 (3)
C(2)	0.2747 (3)	0.9422 (4)	0.5702 (6)	0.063 (4)
C(3)	0.3750 (3)	0.9551 (4)	0.6894 (6)	0.064 (5)
C(4)	0.4138 (3)	0.8420 (4)	0.7209 (6)	0.058 (4)
C(5)	0.3544 (3)	0.7149 (4)	0.6295 (5)	0.047 (3)
C(6)	0.2539 (2)	0.7001 (3)	0.5072 (4)	0.038 (3)
C(7)	0.4180 (3)	0.3310 (4)	0.5766 (5)	0.048 (4)
C(8)	0.5034 (3)	0.3153 (4)	0.7125 (5)	0.052 (4)
C(9)	0.4988 (3)	0.3201 (3)	0.8940 (5)	0.047 (3)
C(10)	0.4080 (3)	0.3367 (3)	0.9385 (5)	0.048 (3)
C(11)	0.3206 (3)	0.3499 (3)	0.8005 (5)	0.044 (3)
C(12)	0.3239 (2)	0.3472 (3)	0.6169 (4)	0.037 (3)
C(13)	-0.0347 (3)	0.0665 (3)	0.2214 (5)	0.047 (3)
C(14)	-0.1125 (3)	-0.0400 (3)	0.1267 (5)	0.051 (4)
C(15)	-0.2061 (3)	-0.0224 (4)	0.0107 (5)	0.051 (3)
C(16)	-0.2191 (3)	0.1005 (3)	-0.0073 (5)	0.045 (3)
C(17)	-0.1398 (2)	0.2127 (3)	0.0879 (4)	0.039 (3)
C(18)	-0.0429 (2)	0.1975 (3)	0.2104 (4)	0.037 (3)
C(19)	0.0409 (2)	0.3108 (3)	0.3063 (4)	0.036 (3)
C(20)	0.0258 (2)	0.4336 (3)	0.2716 (4)	0.037 (3)
C(21)	-0.0716 (2)	0.4440 (3)	0.1487 (4)	0.038 (3)
C(22)	-0.1499 (2)	0.3409 (3)	0.0641 (4)	0.040 (3)
C(23)	0.1362 (3)	0.3002 (3)	0.4451 (5)	0.039 (3)
C(24)	0.2305 (2)	0.3578 (3)	0.4660 (4)	0.038 (3)
C(25)	0.2401 (3)	0.4369 (3)	0.3360 (5)	0.039 (3)
C(26)	0.1900 (3)	0.5641 (3)	0.4016 (4)	0.038 (3)
N	0.0915 (2)	0.5576 (3)	0.3651 (4)	0.040 (3)
F(1)	0.0540 (2)	0.0368 (2)	0.3257 (3)	0.072 (2)
F(2)	-0.0999 (2)	-0.1632 (2)	0.1450 (3)	0.075 (2)
F(3)	-0.2828 (2)	-0.1289 (2)	-0.0798 (3)	0.072 (2)
F(4)	-0.3110 (1)	0.1137 (2)	-0.1210 (3)	0.064 (1)
F(5)	-0.2400 (1)	0.3618 (2)	-0.0473 (3)	0.057 (1)
F(6)	-0.0849 (1)	0.5634 (2)	0.1173 (3)	0.052 (1)

observed and used in the refinement. Structure solved by direct methods using *MULTAN87* (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and refined using *SHELX76* (Sheldrick, 1976). H-atom positions from  $\Delta F$  map. Positions and  $U_{ij}$  of C, F and N atoms, and positions and isotropic  $U$  of H atoms were refined by least squares, using  $F$ , in two approximately equal blocks. Interlayer scale factors were refined at an early stage. Final  $\Delta/\sigma < 0.07$ , variations in  $F$  map  $0.20$  to  $-0.23 \text{ e \AA}^{-3}$ ,  $R = 0.049$ ,  $wR = 0.042$ ,  $S = 2.24$ , 367 parameters refined,  $Q$  (reflexions/parameters) = 5.58,  $w = 4.412/[\sigma^2(F) + 0.000063F^2]$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters are given in Table 1,\* bond distances and angles in Table 2. The molecule with atomic labelling is shown in Fig. 1.

**Related literature.** One other structure with a naphtho ring fused to an azepine ring has been reported, 6,6a,7,8,9,13b-hexahydro-7-methyl-5*H*-benzo[*d*]-naphtho[2,1-*b*]azepine (Berger, Chang, Clader, Hou,

\* Lists of structure factors, H-atom coordinates, bond distances involving H atoms, anisotropic vibrational factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54777 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0494]

Table 2. Bond distances (Å) and angles (°)

C(1)—C(2)	1.398 (5)	C(18)—C(13)	1.428 (5)
C(2)—C(3)	1.383 (6)	C(18)—C(19)	1.465 (4)
C(3)—C(4)	1.391 (7)	C(19)—C(20)	1.424 (5)
C(4)—C(5)	1.396 (5)	C(20)—C(21)	1.422 (4)
C(5)—C(6)	1.390 (4)	C(21)—C(22)	1.351 (4)
C(6)—C(1)	1.407 (5)	C(22)—C(17)	1.439 (5)
C(6)—C(26)	1.498 (4)	C(19)—C(23)	1.458 (4)
C(7)—C(8)	1.378 (5)	C(23)—C(24)	1.351 (5)
C(8)—C(9)	1.380 (6)	C(24)—C(25)	1.526 (6)
C(9)—C(10)	1.368 (6)	C(25)—C(26)	1.519 (5)
C(10)—C(11)	1.393 (5)	C(26)—N	1.284 (4)
C(11)—C(12)	1.398 (5)	N—C(20)	1.410 (4)
C(12)—C(7)	1.396 (5)	C(13)—F(1)	1.355 (4)
C(12)—C(24)	1.490 (4)	C(14)—F(2)	1.371 (5)
C(13)—C(14)	1.369 (4)	C(15)—F(3)	1.360 (4)
C(14)—C(15)	1.389 (5)	C(16)—F(4)	1.347 (4)
C(15)—C(16)	1.368 (6)	C(22)—F(5)	1.352 (4)
C(16)—C(17)	1.420 (4)	C(21)—F(6)	1.375 (4)
C(17)—C(18)	1.433 (4)		
C(6)—C(1)—C(2)	121.1 (3)	C(16)—C(17)—C(18)	118.8 (3)
C(1)—C(2)—C(3)	119.7 (4)	C(16)—C(17)—C(22)	123.6 (3)
C(2)—C(3)—C(4)	119.4 (4)	C(18)—C(17)—C(22)	117.5 (3)
C(3)—C(4)—C(5)	121.1 (3)	C(17)—C(22)—C(21)	122.5 (3)
C(4)—C(5)—C(6)	120.0 (4)	C(17)—C(22)—F(5)	120.2 (3)
C(5)—C(6)—C(1)	118.5 (3)	F(5)—C(22)—C(21)	117.3 (3)
C(5)—C(6)—C(26)	121.0 (3)	C(22)—C(21)—C(20)	121.9 (3)
C(1)—C(6)—C(26)	120.5 (3)	C(22)—C(21)—F(6)	120.0 (3)
C(12)—C(7)—C(8)	120.7 (4)	F(6)—C(21)—C(20)	118.2 (3)
C(7)—C(8)—C(9)	120.7 (4)	C(21)—C(20)—C(19)	119.0 (3)
C(8)—C(9)—C(10)	120.0 (3)	C(21)—C(20)—N	112.6 (3)
C(9)—C(10)—C(11)	119.7 (4)	N—C(20)—C(19)	127.9 (3)
C(10)—C(11)—C(12)	121.4 (4)	C(20)—C(19)—C(18)	119.3 (3)
C(11)—C(12)—C(7)	117.7 (3)	C(20)—C(19)—C(23)	120.6 (3)
C(11)—C(12)—C(24)	121.8 (3)	C(23)—C(19)—C(18)	120.0 (3)
C(7)—C(12)—C(24)	120.5 (3)	C(19)—C(18)—C(13)	124.6 (3)
C(18)—C(13)—C(14)	124.3 (3)	C(19)—C(18)—C(17)	119.8 (3)
C(18)—C(13)—F(1)	121.6 (3)	C(17)—C(18)—C(13)	115.5 (3)
F(1)—C(13)—C(14)	114.1 (4)	C(19)—C(23)—C(24)	123.7 (4)
C(13)—C(14)—C(15)	119.1 (4)	C(23)—C(24)—C(25)	119.1 (3)
C(13)—C(14)—F(2)	121.4 (3)	C(23)—C(24)—C(12)	120.6 (3)
F(2)—C(14)—C(15)	119.5 (3)	C(12)—C(24)—C(25)	120.2 (3)
C(14)—C(15)—C(16)	119.7 (3)	C(24)—C(25)—C(26)	106.8 (3)
C(14)—C(15)—F(3)	118.2 (3)	C(25)—C(26)—N	120.7 (3)
F(3)—C(15)—C(16)	122.2 (3)	C(25)—C(26)—C(6)	120.9 (3)
C(15)—C(16)—C(17)	122.6 (3)	C(6)—C(26)—N	118.3 (3)
C(15)—C(16)—F(4)	117.9 (3)	C(26)—N—C(20)	122.1 (3)
F(4)—C(16)—C(17)	119.5 (3)		

Chipkin & McPhail, 1989); the bond distances are not strictly comparable with the present compound as that azepine ring is fully reduced. It is of interest to compare the dimensions in the present compound with those involving an unfused 3*H*-azepine ring. A few such structures have been reported: 2-(diethylamino)-3-isopropyl-5-phenyl-3*H*-azepine (Streef, van der Plas, van Veldhuizen & Goubits, 1984); 2,4,6,7-tetraphenyl-3*H*-azepine (Katritzky, Aurecochea, Quian, Koziol & Palenik, 1987). Although there are many reported structures with a benz- group fused to other bonds in the azepine ring, none have the common bond in the position comparable with the present structure. The hexa-fluoronaphtho group occurs in one other reported structure, bis(4,5,6,7,8,9-hexafluoronaphtho[2,1-*b*]-fur-2-yl)methane (Halfpenny, 1986).

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## Structure Cristalline du [1]Benzopyrano[6,5,4-*def*][1]benzopyrane (1,6-Dioxapyrène)

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(Reçu le 4 juillet 1991, accepté le 21 octobre 1991)

**Abstract.** C<sub>14</sub>H<sub>8</sub>O<sub>2</sub> (I), *M<sub>r</sub>* = 208.2, monoclinic, *P*2<sub>1</sub>/*n*, *Z* = 2, *a* = 9.027 (4), *b* = 3.922 (1), *c* = 13.396 (6) Å, β = 93.127 (3)°, *V* = 473.6 Å<sup>3</sup>, *D<sub>x</sub>* = 1.461 Mg m<sup>-3</sup>, λ(Cu Kα) = 1.54178 Å, graphite monochromator, μ = 0.747 mm<sup>-1</sup>, *F*(000) = 216, *T* = 293 K, *R* = 0.061 for 449 observed reflections. The

dioxapyrene nuclei lie on centres of symmetry; one of the C—C distances has double-bond character (1.31 Å); the dioxapyrene geometry is rather different from that of pyrene, in agreement with NMR experiments. Molecules are stacked with a distance between their parallel mean planes of 3.50 Å.