C42	0.8071 (2)	0.16091 (15)	0.81263 (14)	0.0238 (5)
C43	0.7877 (2)	0.24113 (15)	0.82536 (13)	0.0208 (4)
C44	0.6626 (2)	0.28606 (13)	0.83242 (12)	0.0170 (4)
C45	0.7560 (2)	0.41272 (14)	0.85072 (13)	0.0185 (4)
C46	0.3216 (2)	0.2510 (2)	0.81408 (13)	0.0202 (4)
C47	0.7606 (2)	0.2994 (2)	1.00306 (14)	0.0264 (5)
C48	0.8165 (2)	0.2615 (2)	1.07887 (15)	0.0352 (6)
C49	0.9276 (2)	0.2936 (2)	1.0837 (2)	0.0348 (6)
C50	0.9824 (2)	0.3611 (2)	1.0118 (2)	0.0331 (6)
C51	0.9257 (2)	0.3967 (2)	0.9371 (2)	0.0262 (5)
C52	0.1325 (2)	0.1711 (2)	0.88846 (15)	0.0259 (5)
C53	0.0562 (2)	0.1219 (2)	0.9596 (2)	0.0326 (6)
C54	0.0934 (2)	0.0977 (2)	1.03984 (15)	0.0316 (5)
C55	0.2088 (2)	0.1233 (2)	1.0460 (2)	0.0339 (6)
C56	0.2828 (2)	0.1724 (2)	0.97339 (14)	0.0269 (5)

Table 2. *Selected geometric parameters (Å)*

N1—C21	1.344 (3)	N3—C47	1.339 (3)
N1—C17	1.349 (3)	N3—C51	1.353 (3)
N1—C15	1.504 (3)	N3—C45	1.506 (3)
N2—C22	1.336 (3)	N4—C52	1.344 (3)
N2—C26	1.352 (3)	N4—C56	1.347 (3)
N2—C16	1.507 (3)	N4—C46	1.508 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	0.92 (3)	2.23 (3)	3.145 (2)	173 (3)
O2—H2···O6	0.85 (3)	1.99 (3)	2.842 (3)	173 (3)
O3—H3A···Cl4	0.89 (3)	2.17 (3)	3.051 (2)	172 (3)
O4—H4A···Cl3^{i}	0.83 (2)	2.24 (2)	3.058 (2)	170 (3)
O5—H5A···Cl1	0.84 (4)	2.39 (4)	3.210 (3)	168 (3)
O5—H5B···Cl2^{ii}	0.87 (3)	2.38 (3)	3.204 (3)	159 (3)
O6—H6A···Cl2^{i}	0.83 (3)	2.32 (3)	3.140 (3)	174 (3)
O6—H6B···Cl1^{iii}	0.85 (3)	2.36 (3)	3.195 (3)	167 (3)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $-x, -y, 1-z$; (iii) $x, 1+y, z$.

All H atoms were located in the difference maps and were refined isotropically. The C—H distances ranged from 0.87 (2) to 1.03 (3) Å and the O—H distances ranged from 0.83 (2) to 0.92 (3) Å. H-atom U_{iso} values ranged from 0.012 (5) to 0.124 (19) Å^2. Anisotropic displacement parameters were used for all non-H atoms.

Data collection and cell refinement were performed using *CAD-4/PC* (Enraf–Nonius, 1993) and data reduction was carried out with *XCAD*4 (Harms, 1995). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined using *SHELXL*93 (Sheldrick, 1993). *SHELXTL* (Siemens, 1995) was used for producing the molecular graphics and *PLATON* (Spek, 1990) was used for both the preparation of the CIF and the geometric analysis.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1271). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9,10-Bis(7-fluoro-2,5-dioxaheptyl)triptycene

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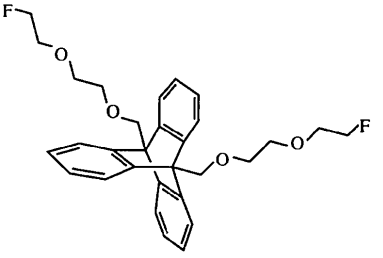
Abstract

The structure of the title compound, $C_{30}H_{32}F_2O_4$, exhibits C—H···π-arene hydrogen bonding.

Comment

In the course of our work on crown ether molecules incorporating the triptycene group (Gakh, Sachleben, Bryan & Moyer, 1995), we isolated the title compound. The interplanar angles between the arene rings (C3–C8 = *A*1, C9–C14 = *A*2 and C15–C20 = *A*3) on the triptycene are *A*1^*A*2 = 116.25 (7), *A*1^*A*3 = 121.74 (8) and *A*2^*A*3 = 121.68 (8)°.



Hydrogen-bond interactions involve the triptycene arene rings (Bakshi *et al.*, 1994; Gakh *et al.*, 1995). The space between rings *A*1 and *A*2 is occupied by

$A1^i$, which π stacks (interplanar distance $\simeq$3.53 Å) with $A1$. This stacking allows H7 to hydrogen bond with $A2^i$, and $H7^i$ to hydrogen bond with $A2$. The distance from H7 to the centroid of $A2^i$ is 2.64 Å. The space between $A1$ and $A3$ is occupied by $C30^{ii}$ and its substituents. $H30B^{ii}$ is oriented towards the centroid of $A3$ ($H30B^{ii}\cdots A3$-centroid = 2.49 Å) and while $H30A^{ii}$ is oriented towards $A1$, the long $H30A^{ii}\cdots A1$-centroid distance (3.19 Å) probably precludes significant interaction. The space between $A2$ and $A3$ is occupied by a symmetry equivalent to the segment C23 to C25. While most of the H atoms on this segment are oriented towards $A2$ and $A3$, none is oriented towards the centroids and all are further than 2.9 Å from the C atoms making up $A2$ and $A3$.

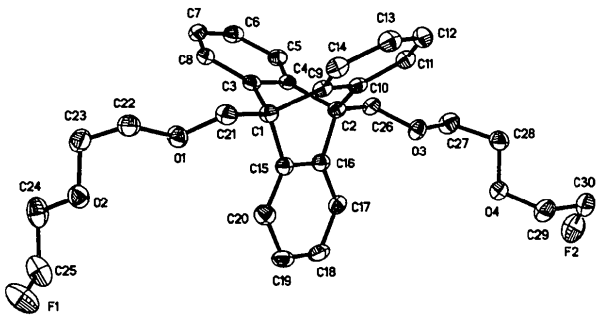


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

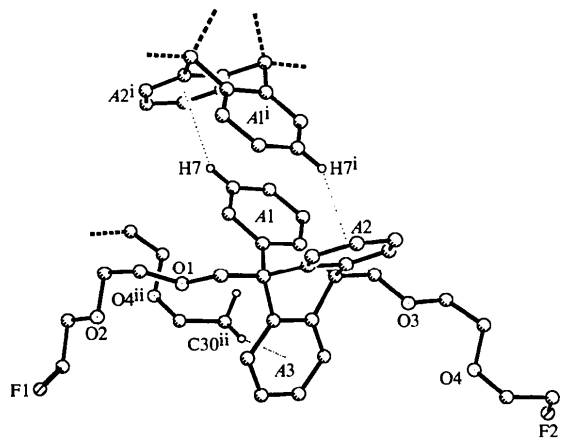


Fig. 2. Hydrogen-bond interactions illustrated using fragments of symmetry-equivalent molecules. For clarity, all atoms are represented as circles and only H atoms involved in hydrogen bonding are drawn. Symmetry codes: (i) $-x, 1-y, -z$; (ii) $x-1, y, z$.

Experimental

The title compound was prepared by addition of benzyne (*o*-anthranylic acid, *i*-amylnitrite, CH_2Cl_2, reflux) to 9,10-bis(7-fluoro-2,5-dioxaheptyl)anthracene. The latter fluoroanthracene compound was prepared from the corresponding ditosylate using $[Bu_4N][F].2H_2O$ as a nucleophilic fluorinating agent (acetone, 2 h reflux). X-ray quality crystals were grown by slow evaporation of a methanol solution.

Crystal data

$C_{30}H_{32}F_2O_4$
M_r = 494.58
Monoclinic
$P2_1/c$
a = 10.413 (2) Å
b = 15.7825 (13) Å
c = 15.503 (2) Å
β = 99.513 (7)°
V = 2512.8 (6) Å^3
Z = 4
D_x = 1.307 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10.2–13.3°
μ = 0.10 mm^{-1}
T = 163 (2) K
Prism
0.70 × 0.41 × 0.33 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-scans
Absorption correction: empirical *via* ψ scans (*SHELXTL*: Siemens, 1990)
T_{min} = 0.932, T_{max} = 0.955
7156 measured reflections
4420 independent reflections

3409 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.025
θ_{max} = 25°
$h = -6 \rightarrow 12$
$k = -22 \rightarrow 18$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: 5.7%

Refinement

Refinement on F^2
$R(F)$ = 0.035
$wR(F^2)$ = 0.095
S = 1.017
4420 reflections
325 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.7586P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
F1	−0.35666 (13)	0.25088 (7)	0.46672 (8)	0.0573 (5)
F2	1.01372 (10)	0.63631 (7)	0.43623 (8)	0.0497 (4)
O1	−0.07460 (10)	0.33991 (7)	0.21520 (7)	0.0292 (3)
O2	−0.27803 (11)	0.31484 (7)	0.31468 (8)	0.0320 (4)
O3	0.51904 (10)	0.56440 (7)	0.24019 (7)	0.0261 (3)
O4	0.76964 (10)	0.56811 (8)	0.35077 (7)	0.0324 (4)
C1	0.12855 (14)	0.39220 (9)	0.18336 (9)	0.0222 (5)
C2	0.29777 (14)	0.51799 (9)	0.19844 (10)	0.0216 (4)
C3	0.07015 (14)	0.47709 (9)	0.14693 (9)	0.0210 (4)
C4	0.15912 (14)	0.54378 (9)	0.15625 (9)	0.0204 (4)
C5	0.1204 (2)	0.62433 (10)	0.12634 (10)	0.0244 (5)
C6	−0.0082 (2)	0.63744 (10)	0.08707 (10)	0.0275 (5)
C7	−0.0958 (2)	0.57096 (10)	0.07651 (10)	0.0270 (5)
C8	−0.05685 (14)	0.49048 (10)	0.10613 (10)	0.0238 (5)
C9	0.24434 (14)	0.37673 (9)	0.13529 (9)	0.0215 (4)
C10	0.33159 (14)	0.44483 (9)	0.14059 (9)	0.0212 (4)

C11	0.43392 (15)	0.44285 (10)	0.09394 (10)	0.0245 (5)
C12	0.4525 (2)	0.37152 (10)	0.04472 (10)	0.0282 (5)
C13	0.3702 (2)	0.30256 (10)	0.04282 (10)	0.0286 (5)
C14	0.2649 (2)	0.30498 (10)	0.08777 (10)	0.0257 (5)
C15	0.19232 (14)	0.41037 (9)	0.27802 (10)	0.0221 (5)
C16	0.28290 (14)	0.47666 (9)	0.28591 (10)	0.0218 (5)
C17	0.34900 (15)	0.49961 (10)	0.36757 (10)	0.0248 (5)
C18	0.3235 (2)	0.45674 (10)	0.44180 (10)	0.0287 (5)
C19	0.2334 (2)	0.39171 (10)	0.43397 (10)	0.0288 (5)
C20	0.1674 (2)	0.36805 (10)	0.35188 (10)	0.0271 (5)
C21	0.03189 (15)	0.31977 (10)	0.17223 (10)	0.0259 (5)
C22	−0.1849 (2)	0.28788 (11)	0.18757 (11)	0.0320 (5)
C23	−0.2986 (2)	0.32389 (11)	0.22254 (11)	0.0344 (6)
C24	−0.3783 (2)	0.35232 (12)	0.35365 (13)	0.0396 (6)
C25	−0.3484 (2)	0.33713 (12)	0.44923 (13)	0.0447 (7)
C26	0.39102 (14)	0.59181 (9)	0.20536 (10)	0.0240 (5)
C27	0.6112 (2)	0.63049 (11)	0.24087 (11)	0.0326 (5)
C28	0.7454 (2)	0.59421 (11)	0.26214 (11)	0.0316 (5)
C29	0.8870 (2)	0.52072 (11)	0.37232 (12)	0.0352 (5)
C30	1.0073 (2)	0.57196 (11)	0.37320 (12)	0.0368 (6)

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

F1—C25	1.393 (2)	O2—C24	1.419 (2)
F2—C30	1.403 (2)	O3—C26	1.421 (2)
O1—C21	1.421 (2)	O3—C27	1.416 (2)
O1—C22	1.419 (2)	O4—C28	1.417 (2)
O2—C23	1.416 (2)	O4—C29	1.425 (2)
C21—O1—C22	112.6 (1)	O2—C24—C25	108.1 (2)
C23—O2—C24	112.8 (1)	F1—C25—C24	110.1 (2)
C26—O3—C27	111.8 (1)	O3—C26—C2	110.1 (1)
C28—O4—C29	112.9 (1)	O3—C27—C28	109.1 (1)
O1—C21—C1	109.5 (1)	O4—C28—C27	109.6 (2)
O1—C22—C23	108.7 (1)	O4—C29—C30	113.9 (1)
O2—C23—C22	109.3 (2)	F2—C30—C29	110.0 (2)
O1—C22—C23—O2	−68.2 (2)	O3—C27—C28—O4	−70.0 (2)
O2—C24—C25—F1	−66.4 (2)	O4—C29—C30—F2	61.1 (2)

Anisotropic displacement parameters were used for all non-H atoms. All H atoms were placed in calculated positions, refined using a riding model and given isotropic displacement parameters equal to 1.2 times those of the atoms to which they are attached.

Data collection and cell refinement were performed using *CAD-4/PC* (Enraf–Nonius, 1993), and data reduction by *XCAD*4 (Harms, 1995). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990), and refined using *SHELXL*93 (Sheldrick, 1993). *SHELXTL* (Siemens, 1990) was used for molecular graphics, and *PLATON* (Spek, 1990) for preparation of the CIF and geometry analysis.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1273). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,4,5,6,9,10-Hexahydro-14,16-dihydroxy-3-methyl-1*H*-2-benzoxacyclotetradecin-1,7(8*H*)-dione (Zearalenone)†

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Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, $C_{18}H_{22}O_5$. The molecule consists of a 14-membered lactone ring fused to a benzene ring. The crystal structure is stabilized by O—H···O and C—H···O hydrogen bonds.

Comment

The compound zearalenone is a micotoxin produced by *Giberella zeare* when the fungus has the proper moisture and temperature conditions. This molecule is responsible for an estrogenic syndrome which attacks the genital system of male and female laboratory animals (Taylor & Watson, 1976). Related molecules have been used to produced curvularin macrolites (Ellestad, Lowell, Perkinson, Hargreaves & McGahren, 1978) and recently it has been recognized that related compounds have a potent activity against P388 leukemia (Agatsuma, Takahashi, Kabuto & Nozoe, 1993). The crystal and molecular structure of the title compound, (I), was first reported by Griffin, Duax, Strong & Mirocha (1981)

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