

N(1)	0.9703 (2)	0.085 (1)	0.0503 (3)	3.9 (1)
C(1)	1	-0.064 (1)	0	3.6 (2)
C(2)	0.9383 (2)	-0.050 (1)	0.1089 (3)	3.5 (1)
C(3)	0.8799 (2)	0.136 (1)	0.1111 (3)	3.5 (1)
C(4)	1.0044 (2)	-0.157 (1)	0.2324 (3)	3.8 (1)
C(5)	1.0548 (2)	0.063 (1)	0.3196 (3)	3.6 (1)
C(6)	1.1200 (2)	0.178 (1)	0.3248 (3)	4.4 (2)
C(7)	1.1664 (2)	0.377 (1)	0.4065 (4)	5.1 (2)
C(8)	1.1490 (2)	0.465 (1)	0.4835 (3)	5.2 (2)
C(9)	1.0853 (2)	0.355 (1)	0.4801 (3)	5.3 (2)
C(10)	1.0381 (2)	0.157 (1)	0.3983 (3)	4.3 (2)
C(11)	0.7894 (2)	0.185 (1)	0.1669 (4)	5.6 (2)
C(12)	0.7567 (3)	0.023 (2)	0.2218 (5)	7.9 (3)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(1)	1.234 (6)	C(4)—C(5)	1.508 (5)
O(2)—C(3)	1.195 (5)	C(5)—C(6)	1.393 (5)
O(3)—C(3)	1.324 (4)	C(5)—C(10)	1.383 (5)
O(3)—C(11)	1.437 (5)	C(6)—C(7)	1.381 (6)
N(1)—C(1)	1.354 (4)	C(7)—C(8)	1.362 (6)
N(1)—C(2)	1.443 (4)	C(8)—C(9)	1.363 (5)
C(2)—C(3)	1.498 (5)	C(9)—C(10)	1.386 (6)
C(2)—C(4)	1.553 (5)	C(11)—C(12)	1.481 (7)
C(3)—O(3)—C(11)	116.7 (3)	C(2)—C(4)—C(5)	114.4 (3)
C(1)—N(1)—C(2)	120.3 (3)	C(4)—C(5)—C(6)	121.6 (3)
O(1)—C(1)—N(1)	122.5 (2)	C(4)—C(5)—C(10)	121.0 (3)
N(1)—C(1)—N(1)	115.1 (5)	C(6)—C(5)—C(10)	117.4 (4)
N(1)—C(2)—C(3)	109.5 (3)	C(5)—C(6)—C(7)	120.8 (4)
N(1)—C(2)—C(4)	113.3 (3)	C(6)—C(7)—C(8)	120.6 (4)
C(3)—C(2)—C(4)	112.4 (3)	C(7)—C(8)—C(9)	119.6 (4)
O(2)—C(3)—O(3)	122.9 (4)	C(8)—C(9)—C(10)	120.4 (4)
O(2)—C(3)—C(2)	124.3 (3)	C(5)—C(10)—C(9)	121.0 (4)
O(3)—C(3)—C(2)	112.8 (3)	O(3)—C(11)—C(12)	109.1 (5)

The data were corrected for Lp effects. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984). The origin was defined by fixing the y-axis position of O(1). H atoms were generated using optimum bonding geometry and included in  $F_c$ , but their displacement parameters and positions were not refined; non-H atoms were refined anisotropically. The function minimized was  $\sum w(|F_o| - |F_c|)^2$ . Anomalous-dispersion effects were included in  $F_c$  (Ibers & Hamilton, 1964). This compound has two chiral centres, therefore we collected all Friedel pairs and switched the signs of the coordinates in an attempt to determine the absolute configuration. We were unable to distinguish between the two enantiomers with our data; we expect that the chirality of the starting compound was maintained under the reaction conditions. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1985).

This research was supported by the National Cancer Institute of Canada (DSCY) and by the Medical Research Council of Canada (WWC).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BR1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 5-Ethoxy-6-[1-(4-methoxyphenyl)ethyl]-1,3-benzodioxole

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(Received 31 August 1993; accepted 4 January 1994)

## Abstract

The title compound, C<sub>18</sub>H<sub>20</sub>O<sub>4</sub>, is structurally similar to podophyllotoxin. It contains a fused dioxole and phenyl ring system and an unfused phenyl ring. However, this compound lacks the cyclohexyl and lactone rings which complete the four-membered ring system of podophyllotoxin. In addition, the unfused phenyl ring in podophyllotoxin contains three methoxy groups in the *para* and *meta* positions, whereas the title compound contains only a methoxy group in the *para* position.

## Comment

The title compound (I) belongs to a series of 6-benzyl-1,3-benzodioxole derivatives which have podophyllotoxin-like antimetabolic activity (Batra, Jurd & Hamel 1985). These derivatives are structurally similar to podophyllotoxin, which is characterized by a four-membered fused ring system and an unfused phenyl ring, and may be used to probe the structure–activity relationships of podophyllotoxin. A recent crystal structure determination for a 6-benzyl-1,3-benzodioxole derivative has shown that an intact fused ring system may not be necessary for antimetabolic activity (Sicheri, Derry, Gupta & Yang,

1992). The results of our structure determination may be used to investigate the influence of the fused-ring environment on activity.

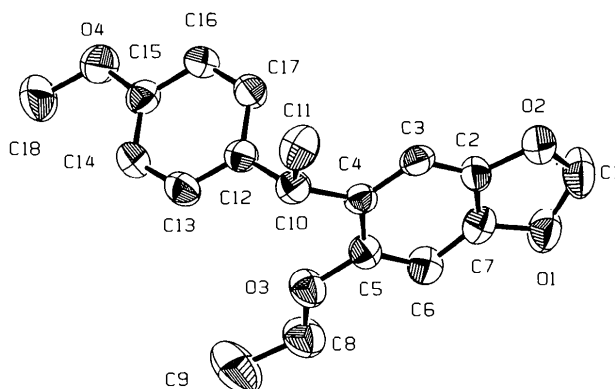
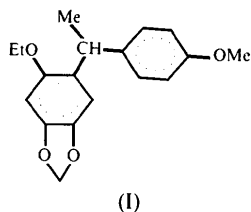


Fig. 1. ORTEP (Johnson, 1976) plot of the molecular structure of the title benzylbenzodioxole derivative showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

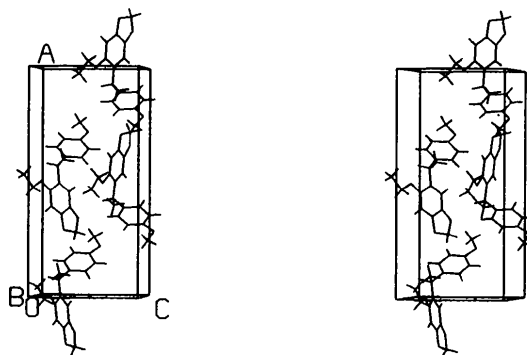


Fig. 2. PLUTO (Motherwell & Clegg, 1978) stereoview of the crystal packing.

## Experimental

The title compound was obtained in crystalline form from the National Cancer Institute (NSC#321567).

### Crystal data

$C_{18}H_{20}O_4$   
 $M_r = 300.35$   
 Orthorhombic  
 $Pca_21$

Cu  $K\alpha$  radiation  
 $\lambda = 1.5418 \text{ \AA}$   
 Cell parameters from 20 reflections

$a = 19.863 (2) \text{ \AA}$   
 $b = 8.644 (2) \text{ \AA}$   
 $c = 9.417 (2) \text{ \AA}$   
 $V = 1616.8 (7) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.234 \text{ Mg m}^{-3}$

$\theta = 22.8\text{--}30.15^\circ$   
 $\mu = 0.668 \text{ mm}^{-1}$   
 $T = 296 \text{ K}$   
 Rectangular  
 $0.25 \times 0.07 \times 0.04 \text{ mm}$   
 Colorless

### Data collection

AFC-6R diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction:  
 empirical (North, Phillips  
 & Mathews, 1968)  
 $T_{\min} = 0.93$ ,  $T_{\max} = 1.00$   
 1434 measured reflections  
 1434 independent reflections  
 1024 observed reflections  
 $[I > 3.0\sigma(I)]$

$\theta_{\max} = 60.05^\circ$   
 $h = 0 \rightarrow 22$   
 $k = 0 \rightarrow 9$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 monitored every 150  
 reflections  
 intensity variation: none

### Refinement

Refinement on  $F$   
 $R = 0.040$   
 $wR = 0.047$   
 $S = 1.43$   
 1024 reflections  
 198 parameters  
 H-atom parameters not  
 refined  
 $w = 4F_o^2/\sigma^2(F_o^2)$   
 $(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.16 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$   
 Extinction correction:  
 secondary (MITHRIL;  
 Gilmore, 1984)  
 Extinction coefficient:  
 $0.26382 \times 10^{-5}$   
 Atomic scattering factors  
 from *International Tables*  
 for X-ray Crystallography  
 (1974, Vol. IV)

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

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}$
O(1)	-0.2100 (1)	0.4004 (4)	0.2727	5.4 (2)
O(2)	-0.1579 (1)	0.6104 (4)	0.3776 (6)	5.4 (2)
O(3)	0.0037 (2)	0.2107 (4)	0.0850 (6)	4.7 (2)
O(4)	0.2149 (2)	0.0092 (3)	0.5399 (6)	5.3 (2)
C(1)	-0.2184 (2)	0.5254 (6)	0.3705 (9)	6.4 (3)
C(2)	-0.1100 (2)	0.5165 (5)	0.3138 (7)	4.0 (2)
C(3)	-0.0418 (2)	0.5384 (5)	0.3022 (6)	3.8 (2)
C(4)	-0.0044 (2)	0.4322 (5)	0.2250 (6)	3.5 (2)
C(5)	-0.0370 (2)	0.3072 (5)	0.1598 (7)	3.6 (2)
C(6)	-0.1065 (2)	0.2844 (5)	0.1722 (7)	4.3 (2)
C(7)	-0.1411 (2)	0.3927 (6)	0.2502 (7)	4.1 (2)
C(8)	-0.0223 (3)	0.0709 (6)	0.0301 (8)	5.8 (3)
C(9)	0.0321 (4)	-0.0151 (8)	-0.0426 (9)	8.4 (4)
C(10)	0.0720 (2)	0.4480 (5)	0.2087 (6)	3.8 (2)
C(11)	0.0983 (2)	0.6122 (6)	0.2359 (7)	5.3 (3)
C(12)	0.1099 (2)	0.3290 (5)	0.2940 (7)	3.5 (2)
C(13)	0.1502 (2)	0.2207 (6)	0.2301 (6)	4.3 (2)
C(14)	0.1862 (2)	0.1113 (6)	0.3089 (7)	4.4 (2)
C(15)	0.1829 (2)	0.1127 (5)	0.4534 (7)	3.8 (2)
C(16)	0.1435 (2)	0.2218 (5)	0.5214 (6)	3.9 (2)
C(17)	0.1074 (2)	0.3291 (5)	0.4429 (7)	3.9 (2)
C(18)	0.2570 (4)	-0.1006 (7)	0.4743 (8)	7.4 (3)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O(1)—C(1)	1.436 (6)	C(4)—C(10)	1.532 (6)
O(1)—C(7)	1.386 (5)	C(5)—C(6)	1.400 (6)
O(2)—C(1)	1.409 (6)	C(6)—C(7)	1.373 (7)
O(2)—C(2)	1.387 (5)	C(8)—C(9)	1.476 (8)

O(3)—C(5)	1.359 (5)	C(10)—C(11)	1.533 (7)
O(3)—C(8)	1.412 (6)	C(10)—C(12)	1.507 (6)
O(4)—C(15)	1.374 (5)	C(12)—C(13)	1.372 (7)
O(4)—C(18)	1.412 (6)	C(12)—C(17)	1.403 (6)
C(2)—C(3)	1.371 (6)	C(13)—C(14)	1.400 (7)
C(2)—C(7)	1.374 (7)	C(14)—C(15)	1.366 (7)
C(3)—C(4)	1.388 (6)	C(15)—C(16)	1.381 (6)
C(4)—C(5)	1.400 (6)	C(16)—C(17)	1.386 (6)
C(1)—O(1)—C(7)	104.5 (4)	O(1)—C(7)—C(6)	127.5 (4)
C(1)—O(2)—C(2)	105.0 (4)	C(2)—C(7)—C(6)	122.6 (4)
C(5)—O(3)—C(8)	119.9 (4)	O(3)—C(8)—C(9)	109.4 (4)
C(15)—O(4)—C(18)	117.1 (4)	C(4)—C(10)—C(11)	113.8 (4)
O(1)—C(1)—O(2)	109.0 (4)	C(4)—C(10)—C(12)	112.4 (4)
O(2)—C(2)—C(3)	129.1 (5)	C(11)—C(10)—C(12)	111.8 (4)
O(2)—C(2)—C(7)	109.6 (4)	C(10)—C(12)—C(13)	121.6 (4)
C(3)—C(2)—C(7)	121.1 (4)	C(10)—C(12)—C(17)	121.0 (4)
C(2)—C(3)—C(4)	118.6 (4)	C(13)—C(12)—C(17)	117.3 (5)
C(3)—C(4)—C(5)	119.6 (4)	C(12)—C(13)—C(14)	121.9 (5)
C(3)—C(4)—C(10)	121.6 (4)	C(13)—C(14)—C(15)	120.0 (5)
C(5)—C(4)—C(10)	118.8 (4)	O(4)—C(15)—C(16)	124.6 (5)
O(3)—C(5)—C(4)	115.3 (4)	O(4)—C(15)—C(16)	115.7 (4)
O(3)—C(5)—C(6)	122.9 (4)	C(14)—C(15)—C(16)	119.7 (5)
C(4)—C(5)—C(6)	121.8 (4)	C(15)—C(16)—C(17)	120.2 (4)
C(5)—C(6)—C(7)	116.2 (4)	C(12)—C(17)—C(16)	121.0 (5)
O(1)—C(7)—C(2)	109.9 (4)		

The title compound has one chiral centre but crystallized in an achiral space group as both enantiomers crystallized together. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984). H atoms were placed using optimum bonding geometry and were included in  $F_c$ . The H-atom displacement parameters and positions were not refined. Non-H atoms were refined anisotropically. The function minimized was  $\sum w(|F_o| - |F_c|)^2$ . Anomalous-dispersion effects were included in  $F_c$  (Ibers & Hamilton, 1964). All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1985).

This research was supported by the National Cancer Institute of Canada (DSCY).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: BR1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1994). **C50**, 1834–1837

## (Ozaphyrin)(TCNQ)<sub>1/2</sub>

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(Received 23 November 1993; accepted 15 March 1994)

## Abstract

Slow diffusion of an acetonitrile solution of tetracyanoquinodimethane (TCNQ) into a chloroform solution of the macrocycle ozaphyrin affords the title compound, 4,9,14,19-tetrapropyl-25-oxa-26,27,28,29-tetraaza-hexacyclo[20.2.1.1<sup>2,5</sup>.1<sup>8,11</sup>.1<sup>12,15</sup>.1<sup>18,21</sup>]nonacos-2(26),3,5,7,9,11,13,15(28),16,18,20,22,24-tridecaene tetracyanoquinodimethane (2/1), C<sub>36</sub>H<sub>40</sub>N<sub>4</sub>O·0.5C<sub>12</sub>H<sub>4</sub>N<sub>4</sub>. The compound was characterized by a single-crystal X-ray structure determination. The packing consists of interspersed planar ozaphyrin and TCNQ molecules [mean deviations from least-squares planes, 0.041 (4) and 0.022 (4) Å, respectively]. The TCNQ molecule, which lies on an inversion center, is above one side of the ozaphyrin ring at a distance of about 3.3 Å.

## Comment

Porphyryns and related macrocycles have been widely used as donors in molecular conductors (Hoffman & Ibers, 1983; Marks, 1985) and some charge-transfer complexes (Kirner, Reed & Scheidt, 1977; Pace, Ullman & Ibers, 1982; Sidorov, 1975). The tetracyanoquinodimethane (TCNQ) molecule has been used in many organic conductors (Underhill, 1974). Here we report the structure of the 1:0.5 complex formed between the expanded macrocycle ozaphyrin and TCNQ. Ozaphyrin was synthesized as described previously (Miller, Johnson, Becker & Ibers, 1993). Crystals of the title compound (I) were obtained from slow diffusion of an acetonitrile solution of TCNQ into a chloroform solution of ozaphyrin.

