

weighting scheme $w^{-1} = \sigma^2(F) + 0.001F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while bond lengths and angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using the *SHELXTL-Plus* package (Sheldrick, 1990). Fig. 2 shows the molecular packing and was also produced with the *SHELXTL-Plus* package.

Related literature. Constable (Constable & Lewis, 1982; Constable, Lewis, Liptrot, Raithby & Schröder, 1983; Constable, Lewis, Liptrot & Raithby, 1984; Constable, Khan, Lewis, Liptrot & Raithby, 1985; Constable, Holmes & McQueen, 1987; Constable & Holmes, 1988) has reported the preparation of macrocyclic ligands and complexes derived from this and related 2,2':6',2''-terpyridines. Interest in the structures of oligopyridines arises from the conformation of the pyridyl rings, which need to change from *trans* to *cis* orientations about the interannular C—C bonds upon coordination to a metal. Although a number of structure determinations of 2,2'-

bipyridine have been reported [see Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff (1981) for the most recent of these], few of the higher oligopyridines have been studied. To date, the only structure determinations reported are of 4'-phenyl-2,2':6',2''-terpyridine (Constable, Lewis, Liptrot & Raithby, 1990) and 2,2':6',2''-6'',2'''-quaterpyridine (Constable, Elder, Healy & Tocher, 1990).

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Structure of Octabenzone

BY I. BRITO,* F. CATALDO AND L. ASTUDILLO

Centro de Productos Naturales Orgánicos Antonio González, Instituto Universitario de Bio-Orgánica, Universidad de La Laguna, Carretera de la Esperanza, 2 La Laguna, Tenerife, Spain

AND P. RIVERA

Departamento de Química, Facultad de Ciencias, Universidad de Chile, Casilla 563, Santiago, Chile

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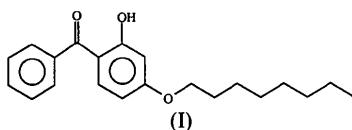
Abstract. [2-Hydroxy-4-(octyloxy)phenyl]phenyl-methanone, $C_{21}H_{26}O_3$, $M_r = 326.4$, monoclinic, $P2_1/n$, $a = 10.186$ (1), $b = 5.546$ (1), $c = 33.232$ (2) Å, $\beta = 98.48$ (4)°, $V = 1856.8$ (2) Å³, $Z = 4$, $D_x = 1.17$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 5.74$ cm⁻¹,

$F(000) = 704$, room temperature, $R = 0.056$, $wR = 0.069$ for 1820 reflections with $I \geq 3\sigma(I)$. Bond lengths and angles are in the usual range. The molecule is in an extended configuration, the benzene and the phenol planes forming a dihedral angle of 53.8 (1)°. Intramolecular hydrogen bonding was observed between O(2) and O(3) of the carbonyl and

* To whom correspondence should be addressed.

hydroxyl groups, O3(H)···O(2), with distance 1.855 (2) Å and angle 122.6 (3)°, forming a sterically favoured six-membered ring.

Experimental. The compound under study (I) has been isolated from the brown alga *Desmarestia menziesii* extract collected in the Antarctic (Rivera, Astudillo & Cataldo, 1990). This is the first report of the isolation of this solar-light screen compound from a natural source.



A single crystal with dimensions $0.75 \times 0.15 \times 0.20$ mm was used for measurements on a Siemens AED diffractometer, with graphite-monochromated $\text{Cu K}\alpha$ radiation, ω - θ scan mode. The lattice constants were obtained from least-squares refinement of 17 reflections in the range $20 \leq 2\theta \leq 40^\circ$, and two standard reflections monitored every hour showed no significant intensity decay. Of the 2642 data measured with $3 \leq 2\theta \leq 121^\circ$, 1985 were independent and 1820 having $I \geq 3\sigma(I)$ (index range $h = 0 \rightarrow 10$, $k = 0 \rightarrow 5$, $l = -32 \rightarrow 32$) were considered observed and used in the structure determination and refinement; $R_{\text{int}} = 0.144$ before and 0.125 after absorption correction; data were corrected for Lorentz-polarization and absorption (Walker & Stuart, 1983) effects; maximum and minimum values of the applied absorption correction were 1.514 and 0.773, respectively.

The structure was solved by direct methods (Sheldrick, 1986) and refined on F by full-matrix least squares (Stewart, Kundell & Baldwin, 1980). Some H

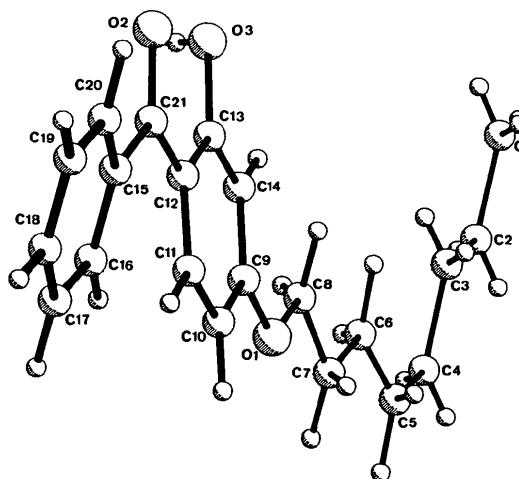


Fig. 1. Plot of a molecule of octabenzone with atomic numbering scheme.

Table 1. Positional parameters and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$) for the non-H atoms with e.s.d.'s in parentheses

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	0.6439 (2)	-0.2528 (4)	0.51497 (5)	60 (1)
O2	1.0496 (2)	0.1061 (4)	0.40072 (6)	76 (1)
O3	1.0337 (2)	-0.2302 (4)	0.45230 (6)	72 (1)
C1	0.9184 (4)	-1.1890 (8)	0.72821 (12)	101 (1)
C2	0.7795 (4)	-1.1855 (7)	0.70632 (11)	91 (2)
C3	0.7448 (3)	-0.9694 (6)	0.67935 (9)	67 (1)
C4	0.6032 (4)	-0.9682 (6)	0.65797 (9)	73 (1)
C5	0.5641 (3)	-0.7509 (6)	0.63112 (9)	67 (1)
C6	0.6488 (3)	-0.7114 (5)	0.59758 (9)	62 (1)
C7	0.5987 (3)	-0.5081 (5)	0.56885 (8)	59 (1)
C8	0.6967 (3)	-0.4497 (5)	0.54071 (8)	57 (1)
C9	0.7190 (3)	-0.1649 (5)	0.48791 (8)	50 (1)
C10	0.6630 (3)	0.0311 (5)	0.46463 (8)	55 (1)
C11	0.7303 (3)	0.1325 (5)	0.43633 (8)	54 (1)
C12	0.8562 (3)	0.0483 (5)	0.43019 (8)	51 (1)
C13	0.9109 (3)	-0.1445 (5)	0.45503 (8)	54 (1)
C14	0.8423 (3)	-0.2534 (5)	0.48310 (8)	55 (1)
C15	0.8640 (3)	0.3107 (5)	0.36704 (8)	56 (1)
C16	0.7422 (3)	0.2516 (6)	0.34445 (9)	65 (1)
C17	0.6890 (4)	0.3935 (7)	0.31172 (10)	83 (1)
C18	0.7551 (5)	0.5937 (8)	0.30184 (12)	90 (2)
C19	0.8754 (5)	0.6566 (7)	0.32438 (13)	90 (2)
C20	0.9304 (4)	0.5134 (6)	0.35643 (11)	74 (1)
C21	0.9294 (3)	0.1506 (5)	0.40029 (9)	58 (1)

Table 2. Interatomic distances (Å) and bond angles (°) with e.s.d.'s in parentheses

O1—C8	1.441 (3)	C9—C14	1.379 (4)
O1—C9	1.355 (4)	C10—C11	1.364 (4)
O2—C21	1.247 (4)	C11—C12	1.408 (4)
O3—C13	1.354 (4)	C12—C13	1.414 (4)
C1—C2	1.493 (6)	C12—C21	1.443 (4)
C2—C3	1.507 (5)	C13—C14	1.383 (4)
C3—C4	1.511 (5)	C15—C16	1.391 (4)
C4—C5	1.517 (5)	C15—C20	1.385 (5)
C5—C6	1.523 (5)	C15—C21	1.495 (4)
C6—C7	1.516 (4)	C16—C17	1.387 (5)
C7—C8	1.500 (4)	C17—C18	1.364 (6)
C9—C10	1.406 (4)	C18—C19	1.383 (6)
		C19—C20	1.380 (5)
C8—O1—C9	117.9 (3)	O3—C13—C12	120.1 (3)
C1—C2—C3	115.0 (4)	C12—C13—C14	122.0 (3)
C2—C3—C4	114.0 (3)	O3—C13—C14	117.1 (3)
C3—C4—C5	115.5 (3)	C9—C14—C13	118.8 (3)
C4—C5—C6	114.6 (3)	C20—C15—C21	119.3 (3)
C5—C6—C7	113.2 (3)	C16—C15—C21	121.6 (3)
C6—C7—C8	111.1 (3)	C16—C15—C20	119.0 (3)
O1—C8—C7	108.1 (3)	C15—C16—C17	120.3 (4)
O1—C9—C10	124.2 (3)	C16—C17—C18	120.1 (4)
C10—C9—C14	114.9 (3)	C17—C18—C19	120.3 (4)
C10—C9—C14	120.9 (3)	C18—C19—C20	120.0 (4)
C9—C10—C11	119.6 (3)	C15—C20—C19	120.4 (4)
C10—C11—C12	121.6 (3)	C12—C21—C15	121.5 (3)
C11—C12—C21	122.9 (3)	O2—C21—C12	117.1 (3)
C11—C12—C13	117.1 (3)	O2—C21—C12	121.3 (3)
C13—C12—C21	120.0 (3)		

atoms could be localized in the difference Fourier maps, and others were calculated (Fayos & Martínez-Ripoll), but all were included with a fixed isotropic contribution ($U = 0.06 \text{ \AA}^2$) and not refined. Refinement converged to $R = 0.056$, $wR = 0.069$, $S = 1.44$ for 217 refined parameters. A weighting

scheme to obtain flat dependence of $\Delta^2 F$ vs F_o and $\sin\theta/\lambda$ was used (Martínez-Ripoll & Cano, 1975); overdetermination ratio was 8.4; maximum shift/e.s.d. = 0.37; minimum and maximum heights in final ΔF map were -0.14 and 0.32 e \AA^{-3} , respectively. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Geometrical calculations were made with *PARST* (Nardelli, 1983).

The molecule and numbering scheme are shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* and interatomic distances and bond angles in Table 2.

Related literature. Spectral studies were described by Rivera, Astudillo & Cataldo (1990). For related

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54699 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0492]

compounds see Rivera, Norte, Cataldo, Podestá & González (1990), Faulkner (1987) and Carrick & Paisley (1974).

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Methyl 8-[(2,6-Dimethoxyphenyl)ethynyl]-7-methoxy-2-naphthoate

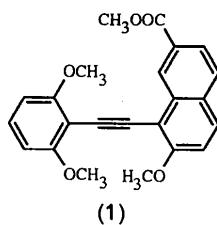
BY PHILIPPE PRINCE, KEVIN L. EVANS, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

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Abstract. $C_{23}H_{20}O_5$, $M_r = 376.4$, orthorhombic, $P2_12_12_1$, $a = 7.0784(8)$, $b = 12.1106(8)$, $c = 22.721(2)$ \AA , $V = 1947.7(5)$ \AA^3 , $Z = 4$, $D_x = 1.284$ g cm^{-3} , $\lambda(\text{Cu } K\alpha) = 1.54184$ \AA , $\mu = 7.0$ cm^{-1} , $F(000) = 792$, $T = 296$ K, $R = 0.036$ for 3727 observations (of 3902 unique data). The average deviations from planarity are 0.003(2) \AA with a maximum of 0.006(2) \AA for the phenyl ring, and 0.005(2) \AA with a maximum of 0.012(2) \AA for the naphthyl ring. The dihedral angle between the two rings is 14.7(1) $^\circ$. The two methoxy groups on the phenyl ring are nearly coplanar with the ring, with C-C-O-C torsion angles of 9.2(2) $^\circ$ for the methoxy group opposite the ester, and -2.7(3) $^\circ$ for the other one. The third methoxy group is nearly coplanar with the naphthyl ring, with a C-C-O-C torsion angle of -8.0(2) $^\circ$. The triple-bond distance is 1.184(2) \AA , and bond angles at the two ethynyl C atoms are 175.9(1) and 176.9(1) $^\circ$.

Experimental. The title compound (1), was prepared by the palladium-catalyzed coupling of 1-ethynyl-2,6-dimethoxybenzene and methyl 8-iodo-7-



methoxy-2-naphthoate in diethylamine at room temperature (Sonogashira, Tohda & Hagiwara, 1975). Pale yellow plates of (1) were isolated by slow evaporation of dichloromethane. Crystal size 0.28 \times 0.40 \times 0.45 mm, mounted on a glass fiber in random orientation on an Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator, $\lambda(\text{Cu } K\alpha) = 1.54184$ \AA . Cell dimensions from setting angles of 25 reflections having $25 < \theta < 29^\circ$. Space

* To whom correspondence should be addressed.