Références

ALLINGER, N. L. & CHANG, S. H. M. (1977). *Tetrahedron*, **33**, 1561–1567.
AYCARD, J. P. & BODOT, H. (1973). *Can. J. Chem.* **51**, 741–747.
BEAGLEY, B. (1973). *Mol. Struct. Diffr. Methods*, **1**, 93–95.
BEAGLEY, B. (1975). *Mol. Struct. Diffr. Methods*, **3**, 66–67.
BRISSE, F., BEAUCHAMP, A., RICHER, J. C., BELLUCCI, G. & INGROSSO, G. (1976). *Acta Cryst.* B**32**, 2128–2132.
BUCOURT, R. (1974). *Top. Stereochem.* **8**, 159–224.
BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Rapport ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
COSSU, M., VIANI, R. & LAPASSET, J. (1981). *Acta Cryst.* B**37**, 481–483.
DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* A**24**, 390–397.
FABER, D. H. & ALTONA, C. (1974). *Acta Cryst.* B**30**, 446–458.
GEDOUX, B. (1978). Thèse de Spécialité (Chimie Organique). Univ. de Provence, Marseille, France.
HUGHES, E. W. (1941). *J. Am. Chem. Soc.* **63**, 1737–1752.
MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J.-P. (1974). *MULTAN*74. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. de York, Angleterre, et Louvain, Belgique.
MONNIER, M. (1977). Thèse de Spécialité (Chimie Organique). Univ. de Provence, Marseille, France.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
VIANI, R., COSSU, M. & LAPASSET, J. (1981). *Acta Cryst.* B**37**, 484–486.
VIANI, R. & LAPASSET, J. (1978). *Acta Cryst.* B**34**, 1195–1198.
VIANI, R. & LAPASSET, J. (1981). *Acta Cryst.* B**37**, 477–480.
VIANI, R., LAPASSET, J. & AYCARD, J. P. (1984). *Acta Cryst.* C**40**, 2074–2076.
VIANI, R., LAPASSET, J. & AYCARD, J. P. (1985). *Acta Cryst.* C**41**, 791–793.
VIANI, R., LAPASSET, J. AYCARD, J. P. & BODOT, H. (1979). *J. Org. Chem.* **44**, 899–902.

Acta Cryst. (1985). C**41**, 1270–1271

Structure of 3,3′-Dimethoxyisodiospyrin,* $C_{24}H_{18}O_8$

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(*Received* 2 *February* 1985; *accepted* 27 *March* 1985)

Abstract. $M_r = 434$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}1422\ (5)$, $b = 10{\cdot}370\ (3)$, $c = 21{\cdot}494\ (3)$ Å, $V = 2037{\cdot}7$ Å^3, $Z = 4$, $D_m = 1{\cdot}34$, $D_x = 1{\cdot}42$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7093$ Å, $\mu = 1{\cdot}0$ cm^{-1}, $F(000) = 904$, $T = 298$ K, final $R = 0{\cdot}057$ for 1587 observed reflections. The molecule could be considered as two juglone molecules connected through a C—C single bond. The dihedral angle between the two planes is 79·7 (5)°. There is an intramolecular hydrogen bond in each juglone moiety forming a pseudo-six-membered ring. No intermolecular hydrogen bonding was found.

Introduction. The isolation and identification of four compounds: lupeol, bisisodiospyrin, isodiospyrin and betulinic acid, from the root of the Morris persimmon, *Diospyros morrisiana* (Ebenaceae), were reported (Yoshihira, Tezuka & Natori, 1971). A very strong antibacterial activity was found in the acetone extract of the heart wood of this plant (Wu, Yang, Hsu & Chen, 1972). In order to understand the constituents of the heart wood, the isolation and identification of the extracts were carried out. Four compounds were isolated; two of them are the same as those (last two above) found in the root, the other two are new compounds: 3′-methoxyisodiospyrin and 3,3′-dimethoxyisodiospyrin. These were characterized by mass spectrometry, IR and NMR spectroscopy. The golden-yellow crystal of the title compound was studied by X-ray diffraction.

Experimental. Crystal 0·3 × 0·5 × 0·5 mm. CAD-4 diffractometer. Unit cell: six reflections, 2θ range 17·68 to 22·78°. D_m by flotation (*n*-hexane/CCl_4). $2\theta_{max} = 60°$. Ranges of h, k, l: 0 to 12, 0 to 14, 0 to 30, respectively. Three standard reflections $2\bar{2}5$, $0\bar{1}3$, 105 monitored every half hour: variation $<\pm 3\%$. 3374 unique reflections, 1587 observed with $I \geq 2\sigma(I)$. $R = 0{\cdot}0568$, $wR = 0{\cdot}0275$, $S = 1{\cdot}84$. Weighting scheme from counting statistics. Structure solved by direct methods using *MULTAN* with 228 highest E's, 50 smallest E's and 2119 $\sum_2$ relationships. H atoms found in difference Fourier map after isotropic refinement and then refined. Least-squares refinement based on F. $(\Delta/\sigma)_{max} = 0{\cdot}75$. Peak in final map $\pm 0{\cdot}23$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: NRC PDP-11 *SDP* (Gabe & Lee, 1981), *MULTAN* and *ORTEP* from Enraf–Nonius (1979) *SDP*.

* 1′,4-Dihydroxy-6,7′-dimethoxy-2,3′-dimethyl-[1,2′-binaphthalene]-5,5′,8,8′-tetrone.

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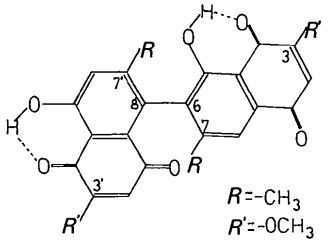
0108-2701/85/081270-02$01.50

Discussion. The molecular structure of the title compound is shown in Fig. 1. The atomic coordinates and equivalent isotropic temperature factors of non-H atoms are listed in Table 1.* Bond lengths are also given in Fig. 1. Bond angles are roughly in the range expected for carbon sp^2 arrangement. The molecule can be thought of as two planar juglone-derivative (5-hydroxy-3-methoxy-7-methyl-1,4-naphthalenedione) parts connected by a C—C (6,8′-) single bond. The dihedral angle between these two planes is 79·7 (5)°. The hydroxyl group at the 5-position and the quinone group at the 4-position form a pseudo-six-membered ring through intramolecular hydrogen bonds:



The bond distances of the molecule are as expected for a juglone molecule with localized double bonds [C(8)=C(9) 1·339 (6); C(18)=C(19) 1·325 (6) Å] at the quinone ring; the other benzene ring is more or less totally delocalized with C—C distance 1·40 Å. Intramolecular hydrogen bonding occurs in each juglone moiety with O(5)—H(1), O(7)—H(14), O(1)···O(5), and O(4)···O(7) distances 0·832, 0·732, 2·561, and 2·583 Å, respectively. The dihedral angle between the two benzene rings in biphenyl compounds varies from 0 (Charbonneau & Delugeard, 1976) to 90° (Singh & McKinney, 1979; Hamor & Hamor, 1978) depending on the substituents at the ring. An interesting intra- and intermolecular hydrogen-bonding network [between O(2) and O(7)] was found in the magnolol structure where the dihedral angle is 45° (Wang, Cheng, Lee & Chen, 1983). The title compound could form a similar hydrogen-bond network through O(2)···O(7) if the dihedral angle were favorable. However, because of the steric hindrance caused by the two methyl groups at C(3) and C(12), the dihedral angle is 79·7 (5)° with the two planes nearly perpendicular to each other. No intermolecular hydrogen bond could possibly be formed.

* Lists of anisotropic thermal parameters, structure factors, H-atom coordinates and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42162 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

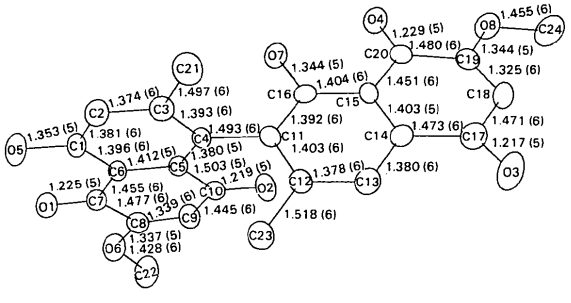


Fig. 1. The molecular structure and bond lengths (Å) of $C_{24}H_{18}O_8$.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors of* $C_{24}H_{18}O_8$

	x	y	z	B_{eq}(Å²)
C(1)	0·8120 (5)	0·0797 (4)	0·1497 (2)	3·64 (2)
C(2)	0·9007 (5)	0·0661 (4)	0·0982 (2)	3·97 (2)
C(3)	1·0080 (5)	0·1551 (4)	0·0855 (2)	3·59 (2)
C(4)	1·0278 (4)	0·2599 (4)	0·1251 (2)	2·90 (2)
C(5)	0·9435 (4)	0·2715 (4)	0·1781 (2)	2·72 (2)
C(6)	0·8313 (4)	0·1820 (4)	0·1912 (2)	2·58 (2)
C(7)	0·7349 (5)	0·1956 (4)	0·2447 (2)	3·32 (2)
C(8)	0·7599 (5)	0·3074 (4)	0·2860 (2)	3·66 (2)
C(9)	0·8703 (5)	0·3901 (4)	0·2770 (2)	3·99 (2)
C(10)	0·9653 (4)	0·3801 (4)	0·2234 (2)	3·12 (2)
C(11)	1·1373 (4)	0·3598 (4)	0·1067 (2)	2·73 (2)
C(12)	1·1019 (4)	0·4547 (4)	0·0627 (2)	3·32 (2)
C(13)	1·2021 (5)	0·5483 (4)	0·0467 (2)	3·69 (2)
C(14)	1·3399 (5)	0·5501 (4)	0·0728 (2)	3·10 (2)
C(15)	1·3791 (4)	0·4560 (4)	0·1167 (2)	2·56 (2)
C(16)	1·2766 (4)	0·3610 (4)	0·1329 (2)	2·96 (2)
C(17)	1·4453 (5)	0·6500 (4)	0·0537 (2)	4·02 (2)
C(18)	1·5836 (5)	0·6591 (4)	0·0884 (2)	3·92 (2)
C(19)	1·6158 (4)	0·5734 (4)	0·1320 (2)	3·73 (2)
C(20)	1·5196 (4)	0·4634 (4)	0·1479 (2)	3·34 (2)
C(21)	1·1017 (5)	0·1380 (5)	0·0290 (2)	5·00 (3)
C(22)	0·6658 (7)	0·4249 (5)	0·3705 (2)	7·00 (3)
C(23)	0·9504 (5)	0·4564 (5)	0·0339 (2)	4·90 (3)
C(24)	1·8357 (6)	0·6807 (5)	0·1620 (2)	6·30 (3)
O(1)	0·6356 (3)	0·1194 (3)	0·2552 (1)	4·56 (2)
O(2)	1·0612 (3)	0·4603 (3)	0·2159 (1)	4·71 (2)
O(3)	1·4204 (4)	0·7234 (3)	0·0107 (1)	5·95 (2)
O(4)	1·5574 (3)	0·3810 (3)	0·1857 (1)	5·06 (2)
O(5)	0·7065 (3)	−0·0100 (3)	0·1583 (1)	5·39 (2)
O(6)	0·6599 (3)	0·3140 (3)	0·3313 (1)	5·34 (2)
O(7)	1·3078 (3)	0·2669 (3)	0·1738 (1)	4·42 (2)
O(8)	1·7370 (3)	0·5712 (3)	0·1674 (2)	5·34 (2)

References

CHARBONNEAU, G. P. & DELUGEARD, Y. (1976). *Acta Cryst.* B**32**, 1420–1423.

Enraf–Nonius (1979). *Structure Determination Package.* Enraf–Nonius, Delft.

GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* A**37**, C339.

HAMOR, M. J. & HAMOR, T. A. (1978). *Acta Cryst.* B**34**, 2994–2997.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

SINGH, P. & MCKINNEY, J. D. (1979). *Acta Cryst.* B**35**, 259–262.

WANG, Y., CHENG, M. C., LEE, J. S. & CHEN, F. C. (1983). *J. Chin. Chem. Soc. (Taipei)*, **30**, 215–221.

WU, S. C., YANG, Y. H., HSU, K. K. & CHEN, F. C. (1972). *Bulletin of Experimental Forestry,* National Taiwan Univ. Rep. 97.

YOSHIHIRA, K., TEZUKA, M. & NATORI, S. (1971). *Chem. Pharm. Bull.* **19**, 2308–2313.