The structure of compound (II) is isomorphous to that of [3^2]paracyclophane–TCNE (Bernstein & Trueblood, 1971). The charge-transfer direction is along the [111] direction in a sandwich arrangement. The shortest TCNE–TCNE distance is 9·374 (5) Å (9·63 Å in [3^2]paracyclophane–TCNE). The relative arrangement of TCNE and the benzene rings is the same in both compounds: the value of 52° defined above is equal to 45° in [3^2]paracyclophane–TCNE. However, the TCNE disorder is less important in the present compound than in the [3^2] complex where a 75–25% orientation disorder was found.

Both compounds show normal intermolecular distances involving van der Waals interactions.

Energy calculations are in progress on these complexes.

The authors are indebted to Professor J. Lajzérowicz for having suggested this study and for fruitful discussions. Thanks are due to Professor P. Baret for contribution to the synthesis of compound (I) and G. d'Assenza for technical assistance.

The data collection was performed within the 'Groupe Grenoblois de Diffractométrie'.

References

André, A., Fourme, R. & Renaud, M. (1971). *Acta Cryst.* B**27**, 2371–2380.

Bernstein, J. & Trueblood, K. N. (1971). *Acta Cryst.* B**27**, 2078–2088.

Busing, W. R. & Levy, H. A. (1962). *ORXFLS*-3. Report ORNL-TM-271. Oak Ridge National Laboratory, Tennessee.

Cohen-Addad, C., Baret, P., Chautemps, P. & Pierre, J. L. (1983). *Acta Cryst.* C**39**, 1346–1349.

Cohen-Addad, C., Lebars, M., Renault, A. & Baret, P. (1984). *Acta Cryst.* C**40**, 1927–1931.

Cram, D. J. & Dewhirst, K. C. (1959). *J. Am. Chem. Soc.* **81**, 5963–5971.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Hope, H., Bernstein, J. & Trueblood, K. N. (1972). *Acta Cryst.* B**28**, 1733–1743.

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

Keehn, P. M. & Rosenfeld, S. M. (1983). In *Cyclophanes in Organic Chemistry*, 45-I. New York: Academic Press.

Little, R. G., Pautler, D. & Coppens, P. (1971). *Acta Cryst.* B**27**, 1493–1499.

Pierre, G., Baret, P., Chautemps, P. & Pierre, J. L. (1983). *Electrochim. Acta*, **28**(9), 1269–1271.

Pierre, J. L., Baret, P., Chautemps, P. & Armand, M. (1981). *J. Am. Chem. Soc.* **103**, 2986–2988.

Trueblood, K., Mirsky, K., Maverick, E., Knobler, C. & Grossenbacher, L. (1982). *Acta Cryst.* B**38**, 2328–2435.

Acta Cryst. (1986). C**42**, 1533–1535

Structure of 2,5-Dihydroxy-3,8-dimethyl-1,4-naphthalenedione (Aristolindiquinone)*

By Heather J. Cowe and Philip J. Cox†

School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, Scotland

Geoffrey A. Cordell, Chun-Tao Che and Harry H. S. Fong

Program for Collaborative Research in the Pharmaceutical Sciences, College of Pharmacy, University of Illinois at Chicago, Chicago, Illinois 60612, USA

and R. Alan Howie

Chemistry Department, University of Aberdeen, Aberdeen AB9 2UE, Scotland

(*Received* 9 *April* 1986; *accepted* 3 *June* 1986)

Abstract. $C_{12}H_{10}O_4$, $M_r = 218·21$, monoclinic, $P2_1/n$, $a = 4·894$ (2), $b = 11·595$ (6), $c = 18·039$ (6) Å, $\beta =$ 106·27 (3)°, $V = 982·6$ Å^3, $Z = 4$, $D_x = 1·47$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·068$ mm^{-1}, $F(000) =$ 456, $T = 293$ K, $R = 0·072$ for 866 reflexions. The aristolindiquinone molecule is essentially planar and exhibits intramolecular hydrogen bonding involving the 5-hydroxy group and the 4-keto O atom, O···O 2·568 (7) Å, angle O—H···O 140 (8)°. Short O···O contacts, C(2)—OH···O=C(1), intramolecularly of 2·643 (7) Å and intermolecularly of 2·731 (8) Å also occur.

Introduction. Aristolindiquinone (1) was extracted from the roots of the Indian medicinal plant *Aristolochia indica* L. (Aristolochiaceae). A complete description of phytochemical and biological studies of the compound

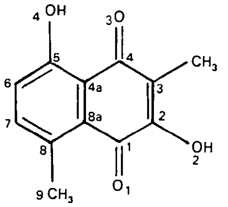
---

* Studies on *Aristolochia*. IV. Part III: Che *et al.* (1984).

† To whom correspondence should be addressed.

0108-2701/86/111533-03$01.50

has been given by Che, Cordell, Fong & Evans (1983) and an initial molecular structure, based primarily on ^{13}C NMR data, was proposed (Che *et al.*, 1984). Confirmation of the structure was sought through crystallographic analysis.



(1)

Experimental. Orange crystal, dimensions 1·0 × 0·1 × 0·5 mm, Nicolet *P*3 diffractometer, graphite monochromator, Mo $K\alpha$ radiation. Cell dimensions from setting angles of 12 independent reflexions with $2\theta \approx 20°$. 1424 intensities measured, with $2\theta < 50°$, as θ–2θ scans. 866 independent observed reflections with $I > 3\sigma(I)$; index range h ±5, k 13, l 21; two reference reflexions monitored periodically showed no significant variation in intensity. No absorption correction. Approximate coordinates of the C and O atoms obtained by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located in difference maps calculated at intermediate stages of refinement. Full-matrix least-squares calculations on *F* with anisotropic thermal parameters for C and O atoms and isotropic for H atoms converged at *R* 0·072, unit weights, $\Delta/\sigma \leq 0{\cdot}03$. *R* influenced by crystal quality. Calculations used the *SHELX* (Sheldrick, 1976) suite of programs. Final $(\Delta\rho)_{\max}$ 0·13, $(\Delta\rho)_{\min}$ −0·12 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates are listed in Table 1* and bond angles in Table 2. The molecular structure with bond lengths is shown in Fig. 1, and the packing of the molecules in Fig. 2.

The X-ray study confirms that the aromatic methyl group is attached at C(8) rather than C(6) and establishes the presence of an intramolecular hydrogen bond, O(4)—H···O(3) with O(4)—H 1·00 (7), H···O(3) 1·72 (7), O(4)···O(3) 2·568 (7) Å, and angle at H 140 (8)°. The O(2)···O(1) contact distance of 2·643 (7) Å probably does not involve a hydrogen bond as the angle subtended at the H atom is only 88 (5)°.

The molecule (excluding H atoms) is essentially planar with the largest torsion-angle deviation from expected planar values less than 4 (1)°.

The structure is in complete agreement with the recently reported synthesis of aristolindiquinone (Achari, Bandyopadhyay, Basu & Pakrashi, 1985).

Table 1. *Fractional atomic coordinates* (×10^4) *and equivalent isotropic temperature factors* (×10^3) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	y	z	U_{eq}(Å^2)
O(1)	7058 (12)	322 (5)	4411 (3)	49 (3)
O(2)	9596 (11)	1540 (5)	5651 (3)	42 (3)
O(3)	2007 (12)	4127 (5)	5012 (3)	50 (3)
O(4)	−1986 (12)	4000 (5)	3740 (3)	52 (4)
C(1)	5835 (16)	1206 (6)	4488 (4)	36 (4)
C(2)	7091 (15)	1924 (6)	5187 (4)	33 (4)
C(3)	5887 (16)	2877 (6)	5359 (4)	35 (4)
C(4)	3172 (15)	3273 (6)	4845 (4)	35 (4)
C(4a)	1899 (16)	2628 (6)	4123 (4)	33 (4)
C(5)	−615 (16)	3043 (6)	3619 (4)	35 (4)
C(6)	−1834 (18)	2436 (8)	2924 (4)	40 (5)
C(7)	−584 (17)	1462 (8)	2769 (4)	46 (4)
C(8)	1917 (16)	990 (6)	3250 (4)	46 (4)
C(8a)	3147 (15)	1604 (6)	3937 (4)	36 (5)
C(9)	3105 (19)	−94 (7)	3006 (4)	43 (5)
C(10)	7166 (20)	3540 (7)	6090 (4)	46 (5)

Table 2. *Bond angles* (°) *with e.s.d.'s in parentheses*

C(2)—C(1)—O(1)	117·9 (7)	C(8a)—C(1)—O(1)	124·0 (7)
C(1)—C(2)—O(2)	115·3 (6)	C(3)—C(2)—O(2)	120·9 (6)
C(3)—C(4)—O(3)	120·1 (6)	C(4a)—C(4)—O(3)	121·1 (7)
C(4a)—C(5)—O(4)	124·6 (7)	C(6)—C(5)—O(4)	116·6 (7)
C(8a)—C(1)—C(2)	118·1 (6)	C(3)—C(2)—C(1)	123·8 (7)
C(4a)—C(8a)—C(1)	117·5 (6)	C(8)—C(8a)—C(1)	120·8 (6)
C(4)—C(3)—C(2)	119·7 (6)	C(10)—C(3)—C(2)	121·8 (7)
C(10)—C(3)—C(4)	118·4 (7)	C(4a)—C(4)—C(3)	118·8 (6)
C(5)—C(4a)—C(4)	118·5 (7)	C(8a)—C(4a)—C(4)	121·9 (6)
C(8a)—C(4a)—C(5)	119·6 (7)	C(6)—C(5)—C(4a)	118·8 (7)
C(8)—C(8a)—C(4a)	121·7 (7)	C(7)—C(6)—C(5)	119·7 (8)
C(8)—C(7)—C(6)	124·6 (7)	C(8a)—C(8)—C(7)	115·6 (7)
C(9)—C(8)—C(7)	119·3 (7)	C(9)—C(8)—C(8a)	125·1 (7)

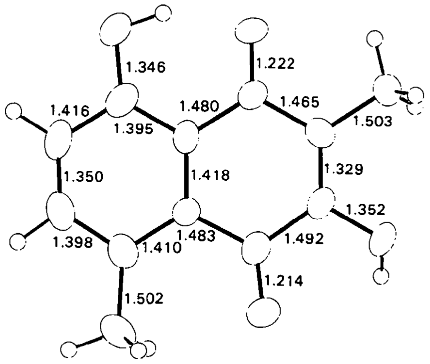


Fig. 1. Atomic arrangement of the molecule showing bond lengths (e.s.d.'s 0·008–0·013 Å).

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and non-bonded contact distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43137 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

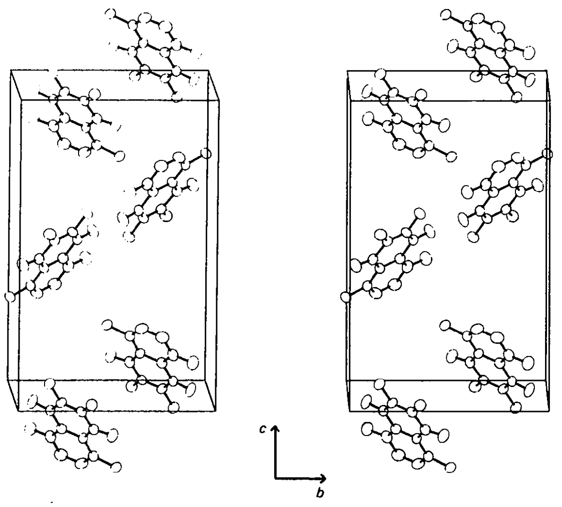


Fig. 2. Stereoscopic view of the molecular packing along **a**.

The investigation at the University of Illinois was supported, in part, by the Special Programme of Research, Development and Reproduction, World Health Organization (Project WHO 77918C). One of us (C-TC) wishes to thank the Graduate College of the University of Illinois at the Medical Center for the award of a fellowship. We also thank the SERC for a studentship (to HJC).

References

ACHARI, B., BANDYOPADHYAY, S., BASU, K. & PAKRASHI, S. C. (1985). *Tetrahedron*, **41**, 107–110.

CHE, C.-T., AHMED, M. S., KANG, S. S., WALLER, D. P., BINGEL, A. S., MARTIN, A., RAJAMAHENDRAN, P., BUNYAPRAPHATSARA, N., LANKIN, D. C., CORDELL, G. A., SOEJARTO, D. D., WIJESEKARA, R. O. B. & FONG, H. H. S. (1984). *J. Nat. Prod.* **47**, 331–341.

CHE, C.-T., CORDELL, G. A., FONG, H. H. S. & EVANS, C. A. (1983). *Tetrahedron Lett.* **24**, 1333–1336.

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

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1986). C**42**, 1535–1537

4,4-Dimethyl-2-oxomorpholinium Bromide. A Novel Six-membered Lactone with a Quaternary Nitrogen

BY GABRIEL GARCIA-GUAJARDO, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

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

(*Received* 2 *October* 1985; *accepted* 8 *May* 1986)

Abstract. $C_6H_{12}NO_2^+.Br^-$, $M_r = 210{\cdot}1$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}166\,(2)$, $b = 11{\cdot}204\,(2)$, $c = 6{\cdot}772\,(1)$ Å, $V = 847{\cdot}2\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}647$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 47{\cdot}5$ cm^{-1}, $F(000) = 424$, $T = 297$ K, $R = 0{\cdot}026$ for 1029 observations (of 1258 unique data). The molecule has a flatter ring than found in a 2-morpholone analogue or six-membered lactones. The ring is in the half-chair conformation. The distance between two tetrahedral carbons, C(3) and C(4), is unusually short, 1·475 (6) Å.

Introduction. As part of a larger study of structural changes in reactions involving tetrahedral intermediates, we are investigating substituent effects in structures of 2-substituted 2-hydroxy-4,4-dimethylmorpholinium salts (Gandour, Stelly, Colucci & Fronczek, 1987). The title compound is the product structure for the reaction under study.

The title compound also has some biological significance. The chloride salt has been used as an algicide and bactericide (Rucka, Oswiecimska, Pawlaczyk-Szpilowa & Witek, 1980) as well as a growth regulator (Borkowski, 1977; Dolnicki, 1980). Because the structure is thought to be a cyclic analogue of acetylcholine, the bromide (Schueler, 1956) and iodide (Nungester & Ames, 1952; Capetola, Gero & Zarro, 1975) salts have been tested for pharmacological activity.

Experimental. The title compound, prepared as described previously (Vieles & Galsomias, 1970) recrystallized from methanol, m.p. 520–521 K decomposition, crystal size 0·16 × 0·36 × 0·48 mm. Space

---

* To whom correspondence should be addressed.