motion but no resolvable disorder was found at the C(4)-atom position, resulting in an observed short bond distance of C(3)—C(4). Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion factors are those given by Cromer & Liberman (1970). All calculations carried out on the CDC-Cyber 170/760 computer of the University of Groningen with the program packages *XTAL* (Hall & Stewart, 1987), *EUCLID* (Spek, 1982; calculations of geometric data) and a locally modified version of the program *PLUTO* (Meetsma, 1986; Motherwell & Clegg, 1978; preparation of illustrations). Final fractional atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are presented in Table 1.* Intramolecular bond distances and angles are summarized in Table 2. Fig. 1 shows the molecular struture with the adopted numbering scheme; Fig. 2 shows the arrangement of the compound in the unit cell viewed along **a**.

The observed conformation of the inorganic PNP ring approaches that of a crown with torsion angles ranging from −17·3 (7) to 15·5 (7)° and lowest asymmetry parameter value ΔC_2 [N(1)—P(2), P(1)—N(3)] = 2·2 (7)° (Duax, Weeks & Rohrer, 1976). Puckering parameters are $Q = 0{\cdot}138$ (6) Å, $\theta = 151$ (2)°, $\varphi = 164$ (5)° [Cremer & Pople (1975); e.s.d.'s following Norrestam (1981)].

Related literature. The structural parameters of the inorganic ring skeleton can be compared with those found in related structures: $[(NSOCl)_2NPClO]^-$ (van Bolhuis, de Ruiter & van de Grampel, 1981), $[(NSOClNHPO_2NPCl_2]^-$ (Winter, 1986) and $[NPHBEt_3(NPF)_2CpFeCp]^-$ (Manners, Coggio, Mang, Parvez & Allcock, 1989).

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* Lists of structure factors, anisotropic thermal parameters of the non-H atoms, H-atom parameters, bond lengths and angles excluding H and an *ORTEP* plot (Johnson, 1965) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52518 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Room-Temperature Structure of Cordiaquinone B

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Abstract. $C_{21}H_{24}O_3$, $M_r = 324{\cdot}42$, monoclinic, $P2_1$, $a = 7{\cdot}729$ (1), $b = 7{\cdot}180$ (1), $c = 16{\cdot}206$ (3) Å, $\beta = 100{\cdot}24$ (1)°, $V = 885{\cdot}1$ (5) Å^3, $Z = 2$, $D_x = 1{\cdot}21$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}05$ mm^{-1}, $F(000) = 346$, $T = 293$ K, $R = 0{\cdot}041$ for 1155 unique observed reflections. The naphtho-

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Table 1. *Coordinates and equivalent isotropic temperature factors* (Å^2) *in* $C_{21}H_{24}O_3$ (*e.s.d.'s in parentheses*)

$B_{\rm eq} = (4/3)\sum_i\sum_j b_{ij}u(\mathbf{a}_i.\mathbf{a}_j)$ (Hamilton, 1959).

	x	y	z	$B_{\rm eq}$
O(1)	−0·4333 (3)	0·2733 (3)	0·5427 (2)	5·91 (4)
O(2)	−0·1072 (3)	0·5711 (4)	−0·0343 (2)	8·37 (4)
O(3)	0·5161 (3)	0·7973 (4)	0·1262 (2)	6·92 (4)
C(1)	−0·4616 (3)	0·3179	0·4686 (2)	4·17 (4)
C(2)	−0·3138 (3)	0·3867 (4)	0·4257 (2)	3·54 (4)
C(3)	−0·3211 (3)	0·2882 (4)	0·3385 (2)	3·66 (4)
C(4)	−0·5064 (3)	0·3236 (4)	0·2867 (2)	4·13 (4)
C(5)	−0·6498 (3)	0·2408 (4)	0·3296 (2)	5·08 (4)
C(6)	−0·6426 (3)	0·3195 (4)	0·4174 (2)	5·02 (4)
C(7)	−0·1766 (3)	0·3711 (4)	0·2944 (2)	4·13 (4)
C(8)	−0·1847 (3)	0·5818 (4)	0·2768 (2)	4·66 (4)
C(9)	−0·0441 (3)	0·6365 (4)	0·2284 (2)	4·16 (4)
C(10)	0·1153 (3)	0·7092 (4)	0·2679 (2)	4·40 (4)
C(11)	0·2510 (3)	0·7436 (4)	0·2237 (2)	4·31 (4)
C(12)	0·2271 (3)	0·7048 (4)	0·1392 (2)	4·08 (4)
C(13)	0·0652 (3)	0·6380 (4)	0·0977 (2)	4·31 (4)
C(14)	−0·0696 (3)	0·6061 (4)	0·1420 (2)	4·39 (4)
C(15)	0·0379 (4)	0·6048 (4)	0·0057 (2)	6·06 (4)
C(16)	0·1959 (4)	0·6099 (4)	−0·0349 (2)	7·49 (4)
C(17)	0·3482 (4)	0·6682 (4)	0·0049 (2)	7·25 (4)
C(18)	0·3751 (4)	0·7297 (4)	0·0921 (2)	5·45 (4)
C(19)	−0·1357 (3)	0·3750 (4)	0·4843 (2)	5·23 (4)
C(20)	−0·2825 (3)	0·0791 (4)	0·3505 (2)	4·49 (4)
C(21)	−0·5311 (4)	0·2582 (4)	0·1948 (2)	6·02 (4)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *in* $C_{21}H_{24}O_3$ (*e.s.d.'s in parentheses*)

C(1)—O(1)	1·223 (3)	C(15)—O(2)	1·217 (3)
C(18)—O(3)	1·231 (3)	C(2)—C(1)	1·521 (4)
C(6)—C(1)	1·496 (4)	C(3)—C(2)	1·572 (4)
C(19)—C(2)	1·531 (3)	C(4)—C(3)	1·548 (3)
C(7)—C(3)	1·547 (4)	C(20)—C(3)	1·536 (4)
C(5)—C(4)	1·528 (4)	C(21)—C(4)	1·540 (4)
C(6)—C(5)	1·523 (4)	C(8)—C(7)	1·538 (4)
C(9)—C(8)	1·501 (4)	C(10)—C(9)	1·386 (3)
C(14)—C(9)	1·396 (4)	C(11)—C(10)	1·392 (4)
C(12)—C(11)	1·378 (4)	C(13)—C(12)	1·397 (4)
C(18)—C(12)	1·494 (4)	C(14)—C(13)	1·384 (4)
C(15)—C(13)	1·488 (5)	C(16)—C(15)	1·485 (4)
C(17)—C(16)	1·307 (4)	C(18)—C(17)	1·460 (5)

C(2)—C(1)—O(1)	121·0 (3)	C(6)—C(1)—O(1)	122·3 (3)
C(6)—C(1)—C(2)	116·6 (3)	C(3)—C(2)—C(1)	110·8 (2)
C(19)—C(2)—C(1)	111·3 (2)	C(19)—C(2)—C(3)	114·8 (2)
C(4)—C(3)—C(2)	107·2 (2)	C(7)—C(3)—C(2)	109·1 (2)
C(7)—C(3)—C(4)	111·2 (2)	C(20)—C(3)—C(2)	110·5 (2)
C(20)—C(3)—C(4)	111·7 (2)	C(20)—C(3)—C(7)	107·2 (2)
C(5)—C(4)—C(3)	111·4 (2)	C(21)—C(4)—C(3)	114·7 (2)
C(21)—C(4)—C(5)	110·2 (2)	C(6)—C(5)—C(4)	111·8 (2)
C(5)—C(6)—C(1)	113·1 (2)	C(8)—C(7)—C(3)	117·1 (2)
C(9)—C(8)—C(7)	110·2 (2)	C(10)—C(9)—C(8)	121·5 (3)
C(14)—C(9)—C(8)	119·8 (2)	C(14)—C(9)—C(10)	118·6 (3)
C(11)—C(10)—C(9)	121·2 (3)	C(12)—C(11)—C(10)	119·6 (3)
C(13)—C(12)—C(11)	120·0 (3)	C(18)—C(12)—C(11)	120·2 (3)
C(18)—C(12)—C(13)	119·8 (3)	C(14)—C(13)—C(12)	120·0 (3)
C(15)—C(13)—C(12)	119·6 (3)	C(15)—C(13)—C(14)	120·4 (3)
C(13)—C(14)—C(9)	120·5 (3)	C(13)—C(15)—O(2)	121·3 (3)
C(16)—C(15)—O(2)	121·5 (3)	C(16)—C(15)—C(13)	117·2 (3)
C(17)—C(16)—C(15)	121·9 (3)	C(18)—C(17)—C(16)	122·5 (3)
C(18)—C(17)—H(17)	118·8 (2)	C(12)—C(18)—O(3)	121·0 (3)
C(17)—C(18)—O(3)	121·2 (3)	C(17)—C(18)—C(12)	117·9 (3)

quinone part of the molecule is nearly flat with non-H atoms deviating ≤ 0·2 Å from the mean plane. The relative configuration deduced from biogenetic pathways is confirmed.

Experimental. 100 mg of pure cordiaquinone B (m.p. 405–407 K) were dissolved in 20 ml of hot cyclohexane containing 5% of acetone, cooled to room temperature and left to stand overnight; 38 mg of crystals deposited on the glass wall. Crystals dried *in vacuo* after decantation of the solvent. Light-yellow platelet, 0·046 × 0·40 × 0·40 mm. Data collected on a Siemens AED2 four-circle diffractometer. ω–θ step-scan mode in N steps of 0·03°, $37 \leq N \leq 43$, time per step: min. 1 s, max. 4 s. Aperture = 3·0 mm. Lattice constants based on 30 reflections measured in double-step-scan mode at $\pm(2\theta \approx 30°)$; no correction for absorption. Intensity measurement to $2\theta \leq 55°$ within range $-10 \leq h \leq 10$, $0 \leq k \leq 9$, $0 \leq l \leq 21$. Standard reflections $\bar{3}05$, 017, 031, intensity variation 1·5%, 2199 independent reflections measured, 1155 reflections used for refinements [$|F| > 6\sigma(|F|)$].

Structure solved from direct methods with *TANG* option of *SHELX76* (Sheldrick, 1976). F magnitudes used in least-squares refinements, 259 parameters refined; mean $\Delta/\sigma = 0{\cdot}039$, max. $\Delta/\sigma = 0{\cdot}19$; atomic scattering factors for C, O, H from *International Tables for X-ray Crystallography* (1974); calculations with *SHELX76* (Sheldrick, 1976). Multisolution tangent direct method of *SHELX76* gives a solution with all non-H atoms. Successive refinements and Fourier maps distinguished O from C atoms. H atoms were then found from difference Fourier maps and geometric constraints were applied to CH_3, CH_2 and CH groups ($d_{\rm C—H} = 1{\cdot}08$ Å). A unique isotropic thermal parameter was refined for all H atoms. The residuals decrease to $R = 0{\cdot}041$, $wR = 0{\cdot}038$, $w = 2{\cdot}2/\sigma^2(F)$. Max. and min. heights in final difference Fourier map: $+0{\cdot}12$ and $-0{\cdot}20$ e Å^{-3}.

The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* Selected bond lengths and angles are listed in Table 2.

The *ORTEP* plot (Johnson, 1965) of the molecule is represented in Fig. 1. Mean-plane calculations show that the naphthoquinone part of the molecule is nearly flat; the carbon and oxygen distances to the mean plane do not exceed 0·2 Å.

Related literature. The sesquiterpenoid quinone cordiaquinone B (1) has been isolated as one the antimicrobial principles of the roots of *Cordia corymbosa* (Bieber, Messana, Lins, Da Silva Filho, Chiappeta & De Mello, 1989). Its relative structure

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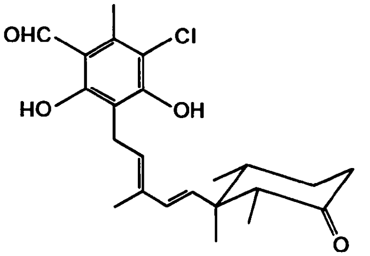
* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52356 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

was established mainly on the basis of the two-dimensional ^{1}H NMR spectrum. The singular rearranged skeleton of the terpenoid part of the molecule shows only a remote similarity with the diterpene alkaloid thelepogine (Fridrichsons & Mathieson, 1960).

The structure determination confirms the general configuration deduced by Bieber *et al.* (1989) from biogenetic pathways. The tetrasubstituted cyclohexanone part of (1) (Fig. 1) has a relative configuration identical with that found in ascochlorin (2), an antibiotic isolated from *Ascochyta viciae* (Nawata, Ando, Tamura, Arima & Iitaka, 1969). Interestingly, cordiaquinone B is dextrorotatory, whereas ascochlorin and all its synthetic precursors containing the same cyclohexanone moiety are levorotatory (Mori & Takechi, 1985).



(2)

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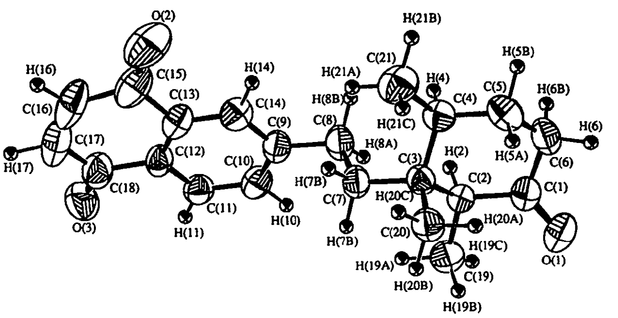

Fig. 1. *ORTEP* plot of the molecule of $C_{21}H_{24}O_3$. For the sake of clarity, the thermal parameters of the H atoms have been divided by ten.

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Structure of 3-(*p*-Chlorophenyl)-1-(3-pyridyl)-2,5-pyrrolidinedione

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Abstract. $C_{15}H_{11}ClN_2O_2$, $M_r = 286{\cdot}72$, monoclinic, $P2_1/c$, $a = 12{\cdot}586$ (2), $b = 6{\cdot}423$ (1), $c = 17{\cdot}186$ (3) Å, $\beta = 92{\cdot}73$ (2)°, $V = 1387{\cdot}7$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}372$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}7$ cm^{-1}, $F(000) = 592$, room temperature, final $R = 0{\cdot}044$ for 1198 observed reflections (of 2393 unique data). There are no unusual distances or angles. The angles between the plane of the five-membered ring and those of the phenyl and pyridyl rings are 89·1 (1) and 53·9 (1)° respectively.

Experimental. The title compound was recrystallized from ethanol to give colourless block-like crystals. Crystal dimensions 0·15 × 0·25 × 0·2 mm. Preliminary data obtained from a CAD-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections ($4 < \theta < 12°$); no absorption correction applied; $\theta < 25°$ (h 0/14, k 0/7, l −20/20); ω–2θ scan technique; Mo $K\alpha$ radiation at room temperature; intensity of three standard reflections monitored every 70 reflections showed no significant fluctuations; 2393 unique reflections measured; 1198 observed reflections with $I > 2\sigma(I)$.

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