results in significant deviations of the S atoms from the mean planes of the adjacent Ph rings. These deviations are clearly seen in Fig. 2.

The S—C bond lengths of 1·776–1·792 (3) Å (ave. 1·784 Å) agree well with the values observed in the molecules of (II) [1·771–1·784 (4), ave. 1·778 Å], (III) [1·771–1·788 (4), ave. 1·779 Å] and (IV) [1·773–1·791 (8), ave. 1·781 Å]. Owing to the ring closure the C—S—C bond angles of 98·4–99·0 (1) (ave. 98·7°) in the smallest of the *p*-phenylenesulfide rings in (I) are naturally somewhat smaller than in (II)–(IV), where these values are 101·9–103·0 (3), 100·1–104·0 (2) and 101·7–105·6 (4)° with average values of 102·5, 102·5 and 103·6° respectively. The S—C bond lengths in (I)–(IV) are almost the same as in acyclic diphenylene-sulfide (1·771 Å; Rozsondai, Moore, Gregory & Hargittai, 1977) and 1,4-bis(phenylthiobenzene) (ave. 1·780 Å; Andreetti, Garbarczyk & Krolikowska, 1981). The C—S—C bond angles in these acyclic analogues are nearly the same [103·7 and 105·4 (2)°] as in the macrocycles (I)–(IV).

The molecular packing in the crystal (I) is shown in Fig. 2. There is only one intermolecular contact S(1)···S(3) (1+*x*, *y*, 1+*z*) of 3·497 (11) Å shorter than twice the van der Waals radius of S (3·60 Å; Bondi, 1964). These contacts form infinite molecular chains along [101].

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Structure of 1,4-Dihydroxy-2-(1-hydroxy-2,3:4,5-di-*O*-isopropylidene-D-arabinityl)-9,10-anthraquinone

BY O. JOHNSON AND D. W. JONES

Department of Chemistry and Chemical Technology, University of Bradford, Bradford BD7 1DP, England

IN PART R. G. HAZELL

Department of Inorganic Chemistry, Aarhus University, DK8000, Århus C, Denmark

AND IN PART B. SHELDRICK

Astbury Department of Biophysics, University of Leeds, Leeds LS2 9TJ, England
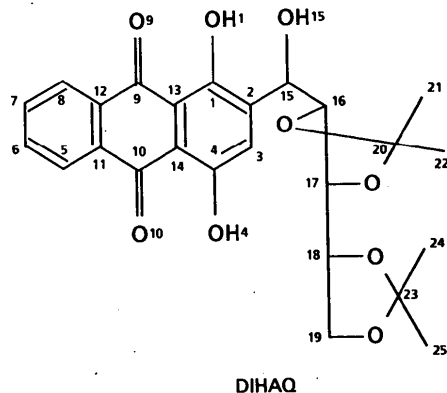
Abstract. $C_{25}H_{26}O_9$, $M_r = 470$, monoclinic, $P2_1$, $a = 18·739$ (8), $b = 8·263$ (5), $c = 7·264$ (3) Å, $\beta = 95·44$ (3)°, $U = 1120$ (2) Å^3, $Z = 2$, $D_m = 1·39$ (1) (carbon tetrachloride/*n*-pentane), $D_x = 1·39$ Mg m^{-3}, $\lambda(Cu K\alpha) = 1·54178$ Å, $\mu = 0·797$ mm^{-1}, $F(000) = 496$, $T = 298$ K, final $R = 0·027$ for 1604 observed diffractometer data. The hydroxyls at O(1) and O(4) of the planar quinizarin moiety in this hydroxyglycityl-quinizarin have close intermolecular approaches to oxygens O(4) and O(9) of neighbouring molecules and are also engaged in intramolecular hydrogen bonds O(1)···O(9) = 2·55 (1) and O(4)···O(10) = 2·59 (1) Å; at the bridge, hydroxyl O(15) has a 2·95 (1) Å intermolecular approach to O(19) of the open-chain arabinose. At the new C(1')-type benzylic chiral carbon labelled C(15), C(15)—OH makes a torsion angle C(3)—C(2)—C(15)—O(15) = 11·5 (1)° with the quinizarin C(3)—(2) bond.

Introduction. 1,4-Dihydroxy-2-(1-hydroxy-2,3:4,5-di-*O*-isopropylidene-D-arabinityl)-9,10-anthraquinone or 1,4-dihydroxy-2-(1-hydroxy-2,3:4,5-di-*O*-isopropyl-idene-D-arabinityl)quinizarin (DIHAQ) is a precursor in the preparation (Mincher, Shaw & Declercq, 1983) of tetrasubstituted anthracyclinones, a class of promising antibiotics for cancer treatment (Arcamone,

$$C_{25}H_{26}O_9$$

1980). In a modification of the Marschalk (1939) reaction, Mincher & Shaw (1981) condensed a D-arabinose derivative with *leuco*-quinizarin to produce stereospecifically at the new chiral centre the hydroxy-glycitylquinizarin DIHAQ. Since the straight-chain D-sugar can be assumed to persist, a crystal structure analysis confirms that the stereochemistry at the benzylic C(1') [here designated C(15)] chiral carbon atom is in the *S* configuration (Johnson, Jones, Mincher & Shaw, 1983) and provides evidence of the hydrogen-bonding system.

DIHAQ

Experimental. Dark-red needles, recrystallized from ethanol from sample supplied by Dr D. J. Mincher (Mincher, Shaw & Declercq, 1983). Single crystal, $0.2 \times 0.2 \times 0.6$ mm (*b* elongation), used to confirm photographically measured cell dimensions (25 reflections, $24 \leq 2\theta \leq 44°$) and for intensity-data collection on Enraf–Nonius CAD-4 diffractometer. Collection details: $\omega/2\theta$ scan, maximum time 60 s, 2θ range 1–70°, index ranges $h -22/22$, $k -10/0$, $l -8/8$; intensity variation of standard reflection $90\bar{2}$ within 10% for hourly checks, orientation checked by $\bar{9}03$ and $60\bar{3}$ and reorientated every 100 reflections. 4649 measurements (including 90 standards and 450 weak) gave 4109 from half-sphere which merged ($R_{int} = 0.011$) to yield 2159 unique data.

Attempts at direct-methods solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) with input of 18-atom quinizarin and 14-atom O-isopropylidene blocking-group rings produced four fused rings in the *E* map, but F_o synthesis and origin shift failed to reveal side chain. Input of 20-atom fragment from *MULTAN* with all unique data into *DIRDIF* (Van den Hark, Prick & Beurskens, 1976) on VAX 11/780 gave some further atoms; inclusion of the highest five non-quinizarin peaks in subsequent *DIRDIF* then revealed the complete molecule. Isotropic full-matrix, followed by aniso-tropic, least-squares refinement on F to $R = 0.061$ enabled all 26 H atoms to be located by difference Fourier syntheses. Final cycles of full-matrix least-squares refinement, anisotropic for C, O, isotropic for

Table 1. *Fractional positional coordinates for the non-hydrogen atoms of DIHAQ* $(\times 10^4)$ *and isotropic equivalent thermal parameters* $(\times 10^4$ Å$^2)$, *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	−1038 (2)	−2551	8554 (4)	395 (17)
C(2)	−1531 (2)	−1405 (5)	9110 (4)	390 (17)
C(3)	−1380 (2)	213 (6)	8979 (4)	404 (18)
C(4)	−744 (2)	746 (5)	8286 (4)	397 (19)
C(5)	1590 (2)	−557 (6)	5993 (5)	543 (22)
C(6)	2086 (2)	−1721 (7)	5572 (5)	619 (24)
C(7)	1940 (2)	−3353 (7)	5716 (5)	585 (23)
C(8)	1290 (2)	−3858 (6)	6270 (5)	507 (21)
C(9)	103 (2)	−3258 (5)	7339 (4)	403 (18)
C(10)	414 (2)	175 (6)	7060 (4)	424 (18)
C(11)	938 (2)	−1061 (6)	6575 (4)	422 (18)
C(12)	790 (2)	−2696 (6)	6710 (4)	406 (18)
C(13)	−406 (2)	−2045 (6)	7872 (4)	373 (17)
C(14)	−254 (2)	−372 (5)	7725 (4)	386 (17)
O(9)	−25 (1)	−4720 (5)	7418 (3)	536 (15)
O(10)	548 (1)	1649 (5)	6903 (3)	543 (15)
O(1)	−1205 (1)	−4126 (5)	8771 (3)	482 (14)
O(4)	−645 (1)	2368 (5)	8226 (4)	509 (15)
C(15)	−2212 (2)	−2001 (6)	9880 (4)	397 (17)
O(15)	−2579 (1)	−753 (5)	10748 (3)	531 (14)
C(16)	−2701 (2)	−2852 (6)	8394 (4)	407 (16)
C(17)	−3371 (2)	−3605 (5)	9061 (4)	398 (17)
C(18)	−3258 (2)	−5273 (6)	9854 (5)	481 (20)
C(19)	−3156 (2)	−6629 (6)	8483 (6)	628 (24)
O(16)	−2996 (1)	−1679 (5)	7091 (3)	528 (13)
O(17)	−3857 (1)	−3601 (5)	7433 (3)	544 (14)
O(18)	−3874 (1)	−5775 (5)	10714 (3)	616 (15)
O(19)	−3460 (1)	−7992 (5)	9304 (4)	590 (15)
C(20)	−3654 (2)	−2327 (6)	6240 (4)	478 (19)
C(21)	−3547 (3)	−3029 (8)	4369 (6)	717 (29)
C(22)	−4200 (3)	−1000 (7)	6176 (7)	696 (29)
C(23)	−4040 (2)	−7409 (6)	10269 (5)	485 (19)
C(24)	−4045 (3)	−8391 (8)	12004 (8)	802 (33)
C(25)	−4735 (3)	−7457 (9)	9083 (9)	864 (36)

all 26 H, with $w^{-1} = [(1 + 0.1)F_o^2 + \sigma(F_o^2)] - |F_o|$ for 410 parameters and 1604 data $[F_o > 6\sigma(F_o)]$ gave $R = 0.027$ ($wR = 0.028$). Only the 100 reflection showed evidence of appreciable extinction; no absorption or extinction corrections. Scattering factors from Cromer & Mann (1968) for C and O atoms and from Stewart, Davidson & Simpson (1965) for H atoms. Dimensions by *ORFFE* (Busing, Martin & Levy, 1962). Final difference Fourier heights within ±0.2 e Å^{-3}.

Discussion. Final fractional coordinates of non-hydrogen atoms are given in Table 1, with bond lengths and angles in Table 2.* The molecules pack (Figs. 1 and 2) in chains parallel to the short *b* axis, linked by intermolecular OH···O contacts between the bridge hydroxyl O(15) [or O(1') as a sugar] and an O(19) [or sugar O(5')] of 2.95 (1) Å and between the quinizarin hydroxyls and acceptor oxygens on the quinizarin, $O(4)\cdots O(9) = 2.76$ (1) and $O(1)\cdots O(4) = 3.12$ (1) Å.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51363 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *for DIHAQ, with e.s.d.'s in parentheses*

C(1)–O(1)	1·351 (4)	C(2)–C(15)	1·523 (5)
C(1)–C(2)	1·410 (5)	C(15)–O(15)	1·419 (5)
C(1)–C(13)	1·390 (5)	C(15)–C(16)	1·520 (5)
C(2)–C(3)	1·371 (5)	C(16)–O(16)	1·432 (4)
C(3)–C(4)	1·408 (5)	C(16)–C(17)	1·522 (5)
C(4)–O(4)	1·356 (4)	C(17)–O(17)	1·423 (4)
C(4)–C(14)	1·390 (5)	C(17)–C(18)	1·501 (5)
C(5)–C(6)	1·390 (7)	C(18)–O(18)	1·425 (4)
C(5)–C(11)	1·396 (5)	C(18)–C(19)	1·525 (6)
C(6)–C(7)	1·388 (7)	C(19)–O(19)	1·418 (5)
C(7)–C(8)	1·381 (6)	C(20)–O(16)	1·430 (4)
C(8)–C(12)	1·402 (5)	C(20)–O(17)	1·439 (4)
C(9)–O(9)	1·235 (4)	C(20)–C(21)	1·511 (6)
C(9)–C(12)	1·482 (5)	C(20)–C(22)	1·497 (6)
C(9)–C(13)	1·460 (5)	C(23)–O(18)	1·420 (5)
C(10)–O(10)	1·251 (5)	C(23)–O(19)	1·435 (4)
C(10)–C(11)	1·481 (5)	C(23)–C(24)	1·498 (7)
C(10)–C(14)	1·456 (5)	C(23)–C(25)	1·490 (7)
C(11)–C(12)	1·386 (5)	C(13)–C(14)	1·417 (5)

C(2)–C(1)–C(13)	120·3 (3)	C(4)–C(14)–C(13)	118·8 (3)
C(2)–C(1)–O(1)	116·5 (3)	C(10)–C(14)–C(13)	120·9 (3)
C(13)–C(1)–O(1)	123·2 (3)	C(2)–C(15)–O(15)	112·8 (3)
C(1)–C(2)–C(3)	119·4 (4)	C(2)–C(15)–C(16)	111·0 (3)
C(1)–C(2)–C(15)	118·9 (3)	O(15)–C(15)–C(16)	111·7 (3)
C(3)–C(2)–C(15)	121·7 (3)	C(15)–C(16)–C(17)	115·0 (3)
C(2)–C(3)–C(4)	121·0 (4)	C(15)–C(16)–O(16)	108·9 (3)
C(3)–C(4)–C(14)	120·2 (3)	C(17)–C(16)–O(16)	102·0 (3)
C(3)–C(4)–O(4)	116·3 (3)	C(16)–C(17)–C(18)	114·0 (3)
C(14)–C(4)–O(4)	123·4 (3)	C(16)–C(17)–O(17)	102·6 (3)
C(6)–C(5)–C(11)	119·1 (4)	C(18)–C(17)–O(17)	112·2 (3)
C(5)–C(6)–C(7)	121·0 (4)	C(17)–C(18)–C(19)	116·4 (3)
C(6)–C(7)–C(8)	120·1 (4)	C(17)–C(18)–O(18)	110·1 (3)
C(7)–C(8)–C(12)	119·3 (4)	C(19)–C(18)–O(18)	103·3 (3)
C(12)–C(9)–C(13)	118·3 (3)	C(18)–C(19)–O(19)	103·2 (3)
C(12)–C(9)–O(9)	120·0 (3)	C(16)–O(16)–C(20)	107·1 (3)
C(13)–C(9)–O(9)	121·7 (3)	C(17)–O(17)–C(20)	108·4 (3)
C(11)–C(10)–C(14)	118·4 (3)	C(18)–O(18)–C(23)	110·3 (3)
C(11)–C(10)–O(10)	120·4 (3)	C(19)–O(19)–C(23)	107·0 (3)
C(14)–C(10)–O(10)	121·2 (4)	O(16)–C(20)–O(17)	106·4 (3)
C(5)–C(11)–C(10)	119·2 (4)	O(16)–C(20)–C(21)	110·6 (4)
C(5)–C(11)–C(12)	120·0 (4)	O(16)–C(20)–C(22)	107·5 (4)
C(10)–C(11)–C(12)	120·9 (3)	O(17)–C(20)–C(21)	109·0 (4)
C(8)–C(12)–C(9)	118·5 (3)	O(17)–C(20)–C(22)	109·3 (4)
C(8)–C(12)–C(11)	120·6 (4)	C(21)–C(20)–C(22)	113·7 (4)
C(9)–C(12)–C(11)	120·9 (3)	O(18)–C(23)–O(19)	105·6 (3)
C(1)–C(13)–C(9)	119·1 (3)	O(18)–C(23)–C(24)	110·1 (4)
C(1)–C(13)–C(14)	120·3 (3)	O(18)–C(23)–C(25)	108·6 (4)
C(9)–C(13)–C(14)	120·6 (3)	O(19)–C(23)–C(24)	107·3 (4)
C(4)–C(14)–C(10)	120·3 (3)	O(19)–C(23)–C(25)	112·0 (4)
		C(24)–C(23)–C(25)	113·0 (5)

Taken with an O(9)···O(10) approach of 3·22 (1) Å, this implies an analogous arrangement in the quinizarin to the bifurcated hydrogen bonding in 1,4-dihydroxy-anthraquinone (1,4-DHA) (Nigam & Deppisch, 1980) and quite different from the crystal packing of methyl-2,3-*O*-isopropylidene-5-(2-quinizaryl)-α-D-lyxofuranoside (MIQL) (Johnson & Jones, 1988). However, the bond dimensions of the quinizarin structures are closely similar in 1,4-DHA and MIQL to those in DIHAQ, which has chelate hydrogen bonds, O(1)···O(9) = 2·55 (1) and O(4)···O(10) = 2·59 (1) Å, from hydroxyls to oxygens of slightly lengthened [1·235 (4), 1·251 (5) Å] carbonyls at C(9) and C(10), respectively. Carbon atoms of the quinizarin are planar within 0·08 Å in both MIQL and DIHAQ; the outer rings are mutually inclined at 1·3° about the C(9)—C(10) axis. The single intermolecular hydrogen bond involving the straight-chain sugar contrasts with the extensive hydrogen bonding possible in the un-blocked β-DL-arabinopyranose (Takagi, Nordenson &

Jeffrey, 1979) and in arabinofuranosyl nucleosides (*e.g.* Birnbaum, Darzynkiewicz & Shugar, 1975; Birnbaum & Gentry, 1983).

Since, in the synthesis of DIHAQ, the arabinose may be taken to retain the D form, there is evidently an *S* arrangement of anticlockwise hydroxyl, sugar and quinizarin (C—H bond away from viewer) at the new chiral carbon C(15) [or C(1′) in sugar nomenclature], as in MIQL (Johnson *et al.*, 1983). The analogy between the conformations of the two structures at the bridge is emphasized by the similarity between pairs of corresponding torsion angles: C(3)—C(2)—C(15)—O(15) = 11·5 (1) in DIHAQ, C(3)—C(2)—C(19)—O(19) = 14·0 (1) in MIQL, and O(15)—C(15)—C(16)—C(17) = 57·8 (1) in DIHAQ, O(19)—C(19)—C(20)—C(21) = 64·1 (1)° in MIQL. The oxygens bearing the isopropylidene groups in DIHAQ are not eclipsed and have torsion angles O(16)—C(16)—C(17)—O(17) = −35·8 (1) and O(18)—C(18)—C(19)—O(19) = 27·3 (1)°. These blocking groups reduce flexibility so that there is a diverse sequence of torsion angles along
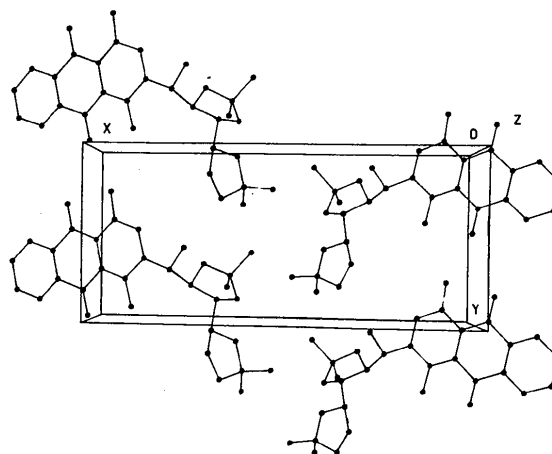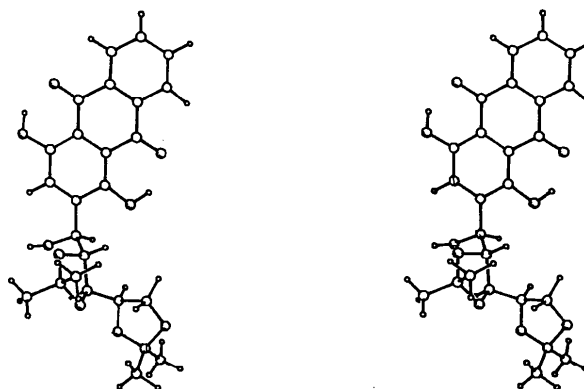

Fig. 1. Projection down c showing molecular packing.

Fig. 2. Stereographic projection of the molecule, showing all atoms.

the arabinose chain C(2)—C(15)—C(16)—C(17)—C(18)—C(19) of, successively, −175·6 (1), 84·9 (1), 72·3 (1)°.

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Structures of Two N-Arylpyridones: 1-(o-Chlorophenyl)-3-methoxy-2-methyl-4-pyridone (1) and 3-Methoxy-2-methyl-1-(1-naphthyl)-4-pyridone (2)

By B. KOJIĆ-PRODIĆ, Ž. RUŽIĆ-TOROŠ AND D. HORVATIĆ

Ruder Bošković Institute, PO Box 1016, 41001 Zagreb, Yugoslavia

Abstract. Compound (1), $C_{13}H_{12}ClNO_2$, $M_r = 249·7$, orthorhombic, *Pbca*, $a = 10·889$ (5), $b = 12·064$ (6), $c = 18·663$ (8) Å, $V = 2451·7$ (2) Å³, $Z = 8$, $D_x = 1·35$ g cm⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 2·96$ cm⁻¹, $F(000) = 1040$, $T = 293$ K, $R = 0·044$ for 1296 unique observed reflections [$I \geq 3\sigma(I)$]. Compound (2), $C_{17}H_{15}NO_2$, $M_r = 265·3$, orthorhombic, $P2_12_12_1$, $a = 15·207$ (4), $b = 11·360$ (4), $c = 7·897$ (3) Å, $V = 1364·2$ (8) Å³, $Z = 4$, $D_x = 1·292$ g cm⁻³, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, $\mu = 6·45$ cm⁻¹, $F(000) = 560$, $T = 293$ K, $R = 0·067$ for 752 unique observed reflections [$I \geq 3\sigma(I)$]. The aryl and pyridone rings are twisted towards each other with dihedral angles between the two ring planes of 91·31 (9) (compound 1) and 85·5 (3)° (compound 2). Molecular packing is realized *via* C—H···O contacts through the pyridone oxygen with C···O distances of 3·247 (4), 3·269 (4), 3·359 (4) Å (1) and 3·26 (1), 3·32 (1) Å (2).

Introduction. Many biphenyls with bulky substituents in *ortho* positions exhibit an axial chirality due to the restricted rotation along the C—C bond; even their enantiomeric pairs can be resolved (Stoddart, 1982).

N-Aryl-4-pyridones have been synthesized and barriers to partial rotation about the C—N bond studied (Mintas, Orhanović, Jakopčić, Koller, Stühler & Mannschreck, 1985). X-ray structure determination of two representatives of the series was performed in order to determine the angles of rotation along the C—N bond (Fig. 1). Perpendicular orientation of the pyridone and aryl rings (Fig. 4) stabilizes the particular enantiomer conformation and permits the enantiomer separation.

Experimental. Weissenberg photographs (Cu $K\alpha$ radiation) determined the space groups *Pbca* for (1) and $P2_12_12_1$ for (2). Both compounds exhibit axial chirality; the centrosymmetrical space group of (1) implies the presence of two enantiomers in a 1:1 ratio (Fig. 1). However, the quality of the crystals of (2) prevented the determination of the absolute configuration by X-ray structure analysis. Preliminary cell dimensions were from oscillation photographs; final ones from diffractometer measurements; 20 reflections ($6 < \theta < 13°$) for (1) and 16 ($7 < \theta < 12°$) for (2). Crystals used: 0·10 × 0·16 × 0·35 (1) and 0·12 × 0·15 × 0·25 mm (2); Philips PW 1100 computer-controlled four-circle diffractometer, ω-scan mode [scan width =