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Structures of (±)-*cis*-2-Hydroxy-4-(2-phenylethyl)- (I) and *cis*-4-Phenyl-2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one (II)

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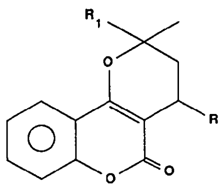
Abstract. Derivatives of 2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one. (I) Racemic *cis*-2-hydroxy-4-(2-phenylethyl), $C_{21}H_{20}O_4$, $M_r = 336{\cdot}39$, triclinic, $P\bar{1}$, $a = 8{\cdot}660\,(7)$, $b = 14{\cdot}299\,(4)$, $c = 7{\cdot}320\,(4)$ Å, $\alpha = 96{\cdot}68\,(3)$, $\beta = 99{\cdot}07\,(5)$, $\gamma = 87{\cdot}554\,(4)°$, $V = 888{\cdot}8\,(16)$ Å^3, $Z = 2$, $D_x = 1{\cdot}257$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71078$ Å, $\mu = 0{\cdot}807$ cm^{-1}, $F(000) = 354$, $T = 294$ K, final $R = 0{\cdot}045$ for 2844 observations [$I \geq 3\sigma(I)$]. Molecules are hydrogen bonded between hydroxyls and coumarin carbonyl groups in chains along **c**, with O···O separation of 2·772 (2) Å. The dihydropyran ring is a half-chair, $\Delta C_2 = 0{\cdot}0057\,(8)$. (II) Spontaneously resolved *cis*-4-phenyl, $C_{19}H_{16}O_3$, $M_r = 292{\cdot}34$, monoclinic, $P2_1$, $a = 8{\cdot}267\,(6)$, $b = 10{\cdot}699\,(7)$, $c = 9{\cdot}039\,(6)$ Å, $\beta = 113{\cdot}67\,(6)°$, $V = 732{\cdot}2\,(18)$ Å^3, $Z = 2$, $D_x = 1{\cdot}326$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71078$ Å, $\mu = 0{\cdot}831$ cm^{-1}, $F(000) = 308$, $T = 294$ K, final $R = 0{\cdot}061$ for 2149 observations [$I \geq 2\sigma(I)$]. The dihydropyran ring is in the *d,e*-diplanar conformation, $\Delta C_2 = 0{\cdot}0569\,(13)$, and retains the relatively long C—O bond, 1·474 (2) Å, compared to its 2-hydroxy derivatives.

Introduction. Dihydropyran-ring shape near the low-energy conformation can be studied through the molecular structures of a series of crystalline warfarin derivatives containing the embedded heterocycle. Warfarin (R_1 = OH, R_2 = phenyl) crystallizes as the hemiketal while in solution it exists as a mixture of diastereomeric hemiketals and an intermediate keto form. This contribution describes the structures of two analogs: one in which the phenyl has a short intervening alkyl chain (I), and another which lacks the hemiketal oxygen (II).



(I) R_1 = OH $R_2 = CH_2CH_2C_6H_5$
(II) R_1 = H $R_2 = C_6H_5$

The latter compound is examined for comparison with the many 4-substituted 2-hydroxy-2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-ones already studied. This molecule does not undergo solution equilibrium between configurational isomers, lacks the operative anomeric effect, and does not form intermolecular hydrogen bonds as do warfarin hemiketals. Consequently the embedded dihydropyran ring in (II) has fewer influences upon it.

Experimental. The Michael-type addition of *α,β*-unsaturated ketones to 4-hydroxycoumarin is a useful path to a variety of warfarin analogs (Ikawa, Stahmann & Link, 1944). Thus 6-phenyl-3-buten-2-one produces (I) on formation of the cyclic

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hemiketal. Crystals were obtained as plates by evaporation of solutions of (I) in propanone, m.p. 423–425 K. Following synthesis of warfarin with 4-phenyl-3-buten-2-one, reduction and dehydration (Chan, Lewis & Trager, 1972) produces deoxywarfarin (II), m.p. 495–496 K. Recrystallization from propanone produced a conglomerate; the compound spontaneously resolves. The data crystal (see below) was later dissolved in acetonitrile and the circular dichroism spectrum showed positive, negative and positive Cotton effects of increasing intensity at 310, 260 and 225 nm, indicative of the (2*R*,4*R*) absolute configuration (Valente & Trager, 1978).

Crystallographic specimens: (I) 0·5 × 0·5 × 0·25 mm; (II) 0·3 × 0·4 × 0·5 mm were chosen for data collection on a CAD-4 diffractometer. Unit-cell constants were derived from a least-squares fit of 25 reflections with $30 < 2\theta < 35°$. Intensities were recorded with variable speed θ–2θ scans to $2\theta = 56°$ (h: −11 to 11, k: −18 to 18, l: 0 to 9) for (I), to 60° (h: 0 to 11, k: 0 to 15, l: −12 to 11) for (II). Three standard intensities monitored every 3 h of exposure time showed departures of −1·0 (4·3) and +0·3 (2·3)%, respectively, over the course of data collection; no correction for deterioration was made. The unique data [4609 (I), 2369 (II)] were corrected for coincidence and polarization, and equivalent observations were averaged. An extinction coefficient of the form proposed by Zachariasen (1963) was later applied and refined for (I): $g = 1{\cdot}4\ (3) \times 10^{-6}$.

Structures were discovered with *MULTAN* (Germain, Main & Woolfson, 1971). Non-H-atom positions were refined with their $U_{\rm iso}$'s by full-matrix least squares on F, then with their U_{ij}'s. The function minimized in full-matrix least squares was $\sum w(|F_o - F_c|)^2$. H-atom positions were calculated and placed 1·0 Å from their attached atom and assigned B's approximately 1·3 times the $B_{\rm eq}$ for the attached carbon; except for the hydroxyl H [in (I)] they were not refined. Scattering factors were from *International Tables for X-ray Crystallography* (1974) except for H (Stewart, Davidson & Simpson, 1965). Final agreement factors for (I): $R = 0{\cdot}045$, $wR = 0{\cdot}066$, GOF = 1·79, and for (II): $R = 0{\cdot}061$, $wR = 0{\cdot}088$, GOF = 2·74 for the 2844 and 2149 intensities greater than $3\sigma(I)$ and $2\sigma(I)$, respectively. Weights corresponding to $1/\sigma(F^2)$ where $\sigma(F^2) = [\sigma(I)^2 + (0{\cdot}05 \times F^2)^2]^{1/2}$ were used in refinements on the 231 (I) and 198 (II) variables; scale factors 0·108 (I) and 0·033 (II); final maximum Δ/σ less than 0·01 for each structure. Maximum $|\Delta\rho|$ excursions for (I) are less than 0·19 e Å^{-3}; some residual density (+0·516 e Å^{-3}) remains near C12 in (II) but appears to have no chemical significance; otherwise the maximum $|\Delta\rho|$ excursions are less than 0·30 e Å^{-3}. All other programs used were from the locally modified Enraf–Nonius (1979) *Structure Determination Package*, version 3·0. Final atom positions and equivalent isotropic vibrational factors for the non-H atoms are given in Tables 1 and 2.* Bond distances and angles are in Tables 3 and 4.

Table 1. *Positions and B_{eq}'s for* (I), *with e.s.d.'s in parentheses*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	$B_{\rm eq}$(Å^2)
O1	0·1569 (1)	0·46652 (8)	0·1866 (1)	4·07 (2)
O2	0·2244 (2)	0·33379 (9)	0·0348 (2)	5·15 (3)
O3	0·4087 (1)	0·39887 (7)	0·6793 (1)	3·83 (2)
O4	0·3404 (1)	0·24945 (8)	0·7221 (2)	4·03 (2)
C2	0·2361 (2)	0·3814 (1)	0·1847 (2)	3·63 (3)
C3	0·3257 (2)	0·3548 (1)	0·3549 (2)	3·10 (3)
C4	0·3288 (2)	0·4149 (1)	0·5132 (2)	3·10 (3)
C5	0·2296 (2)	0·5639 (1)	0·6773 (2)	4·09 (3)
C6	0·1358 (2)	0·6445 (1)	0·6686 (3)	4·85 (4)
C7	0·0514 (2)	0·6649 (1)	0·4988 (3)	4·87 (4)
C8	0·0598 (2)	0·6056 (1)	0·3378 (3)	4·36 (4)
C9	0·1543 (2)	0·5252 (1)	0·3484 (2)	3·52 (3)
C10	0·2400 (2)	0·5030 (1)	0·5155 (2)	3·27 (3)
C11	0·4143 (2)	0·2615 (1)	0·3497 (2)	3·48 (3)
C12	0·5326 (2)	0·2612 (1)	0·5289 (2)	3·68 (3)
C13	0·4667 (2)	0·3022 (1)	0·7013 (2)	3·44 (3)
C14	0·5875 (2)	0·3127 (1)	0·8756 (2)	4·86 (4)
C15	0·3034 (2)	0·1778 (1)	0·3096 (2)	3·92 (3)
C16	0·3870 (3)	0·0832 (1)	0·2740 (4)	7·57 (7)
C17	0·2735 (2)	0·0037 (1)	0·2239 (4)	6·53 (6)
C18	0·2343 (3)	−0·0352 (2)	0·0405 (4)	8·22 (7)
C19	0·1249 (4)	−0·1065 (2)	−0·0053 (5)	10·07 (8)
C20	0·0579 (4)	−0·1389 (2)	0·1290 (6)	10·8 (1)
C21	0·0944 (4)	−0·1011 (2)	0·3095 (5)	10·14 (9)
C22	0·2020 (3)	−0·0292 (2)	0·3578 (4)	8·06 (7)
H4	0·311 (2)	0·275 (1)	0·828 (3)	3·5 (4)*

* Refined isotropically.

Discussion. The crystal structure of (I) consists of molecules hydrogen bonded along **c** with donor hydroxyls and receptor coumarin carbonyls. The O4···O2 separation is 2·772 (2) Å, the H4···O2 separation is 1·88 (2) Å and the angle at H is 173 (2)°. Coumarin and dihydropyran ring atoms have nominal r.m.s. amplitudes of vibration amounting to less than 0·3 Å. Side-chain phenyl rings pack parallel along the c axis but loosely with considerable in-plane vibration amounting to at most 0·4–0·5 Å (r.m.s.) along **c** for the phenyl-ring atoms. The hydroxy group is axial and the 2′-phenylethyl group is pseudoaxial (*cis*) on the embedded dihydropyran ring. The dihydropyran ring is a nearly undistorted half-chair; the ring displacement asymmetry parameter $\Delta C_2 = 0{\cdot}0057\ (8)$ (Nardelli, 1983). Dihydropyran

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* Lists of H-atom positions, anisotropic vibrational amplitudes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51863 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

intraring torsion angles are given in Table 5; an *ORTEP* drawing (Johnson, 1976) of (I) is given in Fig. 1.

Table 2. *Positions and B_{eq}'s for* (II), *with e.s.d.'s in parentheses*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}(Å^2)
O1	0·8991 (2)	0·250	0·8611 (2)	4·03 (4)
O2	0·8955 (3)	0·1475 (3)	1·0715 (2)	5·31 (5)
O3	0·7368 (2)	−0·0619 (2)	0·5744 (2)	3·87 (4)
C2	0·8597 (3)	0·1448 (3)	0·9275 (3)	3·78 (5)
C3	0·7836 (3)	0·0390 (3)	0·8239 (2)	3·15 (4)
C4	0·7783 (3)	0·0390 (2)	0·6708 (2)	3·03 (4)
C5	0·7986 (3)	0·1603 (3)	0·4415 (3)	3·71 (5)
C6	0·8327 (3)	0·2725 (3)	0·3839 (3)	4·31 (5)
C7	0·8863 (4)	0·3762 (3)	0·4845 (3)	4·51 (5)
C8	0·9097 (4)	0·3684 (3)	0·6457 (3)	4·19 (5)
C9	0·8745 (3)	0·2551 (3)	0·7019 (3)	3·38 (4)
C10	0·8186 (3)	0·1509 (2)	0·6028 (2)	3·10 (4)
C11	0·7245 (3)	−0·0710 (3)	0·8924 (3)	3·70 (4)
C12	0·6483 (4)	−0·1723 (3)	0·7600 (3)	4·82 (6)
C13	0·7510 (4)	−0·1824 (3)	0·6575 (3)	4·52 (6)
C14	0·6843 (5)	−0·2793 (3)	0·5283 (4)	5·40 (8)
C15	0·5816 (3)	−0·0376 (2)	0·9517 (3)	3·25 (4)
C16	0·5802 (4)	−0·0906 (3)	1·0912 (3)	4·58 (6)
C17	0·4429 (4)	−0·0685 (4)	1·1381 (4)	5·74 (7)
C18	0·3035 (4)	0·0041 (4)	1·0463 (4)	4·92 (6)
C19	0·3037 (4)	0·0594 (3)	0·9088 (4)	4·83 (6)
C20	0·4424 (4)	0·0391 (3)	0·8628 (3)	4·48 (5)

Table 3. *Bond distances* (Å) *and angles* (°) *for* (I)

O1—C2	1·371 (1)	C8—C9	1·386 (2)
O1—C9	1·372 (1)	C9—C10	1·389 (2)
O2—C2	1·215 (1)	C11—C12	1·532 (2)
O3—C4	1·340 (1)	C11—C15	1·540 (2)
O3—C13	1·468 (1)	C12—C13	1·513 (2)
O4—C13	1·392 (1)	C13—C14	1·514 (2)
C2—C3	1·441 (1)	C15—C16	1·520 (2)
C3—C4	1·357 (1)	C16—C17	1·510 (2)
C3—C11	1·510 (1)	C17—C18	1·388 (3)
C4—C10	1·448 (1)	C17—C22	1·375 (3)
C5—C6	1·383 (2)	C18—C19	1·397 (3)
C5—C10	1·399 (2)	C19—C20	1·351 (4)
C6—C7	1·393 (2)	C20—C21	1·362 (4)
C7—C8	1·379 (2)	C21—C22	1·396 (3)

C2—O1—C9	121·58 (8)	C3—C11—C12	108·48 (9)
C4—O3—C13	117·18 (8)	C3—C11—C15	111·85 (9)
O1—C2—O2	115·6 (1)	C12—C11—C15	114·77 (9)
O1—C2—C3	119·38 (9)	C11—C12—C13	113·40 (9)
O2—C2—C3	125·0 (1)	O3—C13—O4	107·69 (9)
C2—C3—C4	118·9 (1)	O3—C13—C12	109·45 (9)
C2—C3—C11	118·67 (9)	O3—C13—C14	104·02 (9)
C4—C3—C11	122·42 (9)	O4—C13—C12	109·33 (9)
O3—C4—C3	124·7 (1)	O4—C13—C14	112·4 (1)
O3—C4—C10	113·90 (9)	C12—C13—C14	113·7 (1)
C3—C4—C10	121·4 (1)	C11—C15—C16	113·4 (1)
C6—C5—C10	119·9 (1)	C15—C16—C17	111·8 (1)
C5—C6—C7	120·0 (1)	C16—C17—C18	120·8 (2)
C6—C7—C8	121·0 (1)	C16—C17—C22	120·6 (2)
C7—C8—C9	118·4 (1)	C18—C17—C22	118·5 (2)
O1—C9—C8	117·0 (1)	C17—C18—C19	120·2 (2)
O1—C9—C10	121·1 (1)	C18—C19—C20	120·4 (3)
C8—C9—C10	121·9 (1)	C19—C20—C21	120·2 (2)
C4—C10—C5	123·7 (1)	C20—C21—C22	120·4 (3)
C4—C10—C9	117·5 (1)	C17—C22—C21	120·3 (2)
C5—C10—C9	118·7 (1)		

The structure of (II) shows no unusual close contacts between molecules. The phenyl ring is disposed pseudoequatorially on the dihydropyran ring and *cis* to the equatorial methyl, the conformation with the

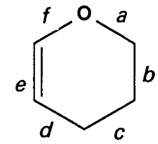
Table 4. *Bond distances* (Å) *and angles* (°) *for* (II)

O1—C2	1·375 (2)	C8—C9	1·389 (3)
O1—C9	1·371 (2)	C9—C10	1·388 (2)
O2—C2	1·214 (2)	C11—C12	1·549 (3)
O3—C4	1·342 (2)	C11—C15	1·523 (2)
O3—C13	1·474 (2)	C12—C13	1·491 (3)
C2—C3	1·443 (2)	C13—C14	1·492 (3)
C3—C4	1·367 (2)	C15—C16	1·387 (2)
C3—C11	1·500 (2)	C15—C20	1·379 (2)
C4—C10	1·444 (2)	C16—C17	1·382 (3)
C5—C6	1·382 (3)	C17—C18	1·361 (3)
C5—C10	1·404 (2)	C18—C19	1·377 (3)
C6—C7	1·389 (3)	C19—C20	1·385 (3)
C7—C8	1·393 (3)		

C2—O1—C9	122·0 (1)	C4—C10—C5	123·4 (1)
C4—O3—C13	115·2 (1)	C4—C10—C9	117·7 (1)
O1—C2—O2	116·8 (2)	C5—C10—C9	118·8 (2)
O1—C2—C3	118·5 (1)	C3—C11—C12	109·2 (1)
O2—C2—C3	124·7 (2)	C3—C11—C15	112·9 (1)
C2—C3—C4	119·0 (1)	C12—C11—C15	107·7 (2)
C2—C3—C11	118·5 (1)	C11—C12—C13	112·0 (2)
C4—C3—C11	122·3 (1)	O3—C13—C12	107·7 (1)
O3—C4—C3	123·6 (1)	O3—C13—C14	106·3 (2)
O3—C4—C10	115·6 (1)	C12—C13—C14	114·4 (2)
C3—C4—C10	120·8 (1)	C11—C15—C16	121·0 (1)
C6—C5—C10	119·8 (2)	C11—C15—C20	121·2 (1)
C5—C6—C7	120·4 (2)	C16—C15—C20	117·6 (2)
C6—C7—C8	120·7 (2)	C15—C16—C17	121·1 (2)
C7—C8—C9	118·3 (2)	C16—C17—C18	120·6 (2)
O1—C9—C8	117·0 (1)	C17—C18—C19	119·2 (2)
O1—C9—C10	121·0 (2)	C18—C19—C20	120·3 (2)
C8—C9—C10	122·0 (2)	C15—C20—C19	121·1 (2)

Table 5. *Torsion angles* (°) *in the dihydropyran rings*

E.s.d.'s are at most about 0·4°; common configuration.



	a	b	c	d	e	f
(I)	−42·2	57·4	−42·7	14·3	0·1	14·6
(II)	−52·0	62·5	−38·7	4·2	6·6	18·5

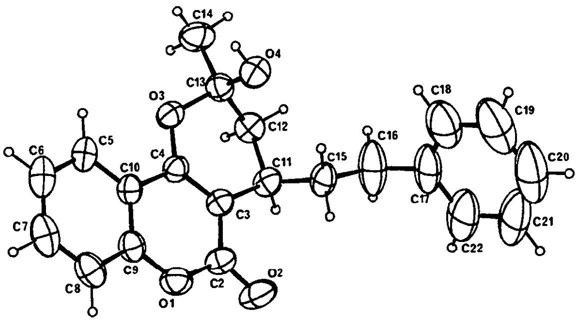


Fig. 1. A drawing of (I) with 50% probability ellipsoids for the non-H atoms.

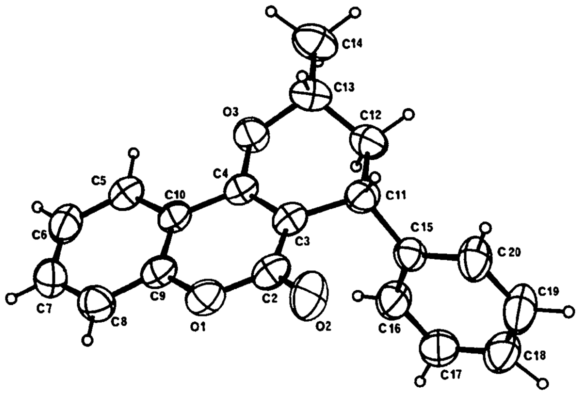


Fig. 2. A drawing of (II) with 50% probability ellipsoids for the non-H atoms.

least diaxial interactions. A plane through the phenyl ring is inclined at 80° with respect to the coumarin ring plane. The dihydropyran ring has a *d,e*-diplanar conformation, with ring displacement asymmetry $\Delta C_2 = 0{\cdot}0569\ (13)$. Intraring bond distances and angles are generally similar to those found in related structures having axial 2-hydroxy groups (Valente, Eggleston & Schomaker, 1987) including the relatively long O—C(sp^3) bond (O3—C13) 1·474 (2) Å. The persistence of this feature suggests that it may be inherent to chroman dihydropyrans. Dihydropyran-ring torsion angles are given in Table 5; a drawing of (II) is given in Fig. 2.

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Structure of the 1:1 Molecular Complex of Durene* with 1,2,4,5-Tetracyanobenzene†

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Abstract. $C_{10}H_{14}.C_{10}H_2N_4$, $Mr = 312{\cdot}4$, monoclinic, $P2_1/a$, $a = 15{\cdot}249\ (6)$, $b = 7{\cdot}750\ (3)$, $c = 7{\cdot}520\ (2)$ Å, $\beta = 105{\cdot}28\ (4)°$, $V = 857{\cdot}3\ (1{\cdot}1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}21$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}41$ cm^{-1}, $T = 293$ K, $F(000) = 328$, $wR = 0{\cdot}038$ for 912 observed reflections. The structure is formed by mixed stacks along **c** with uniformly spaced donor and acceptor molecules. Durene–TCNB is isostructural with the low-temperature phases of anthracene–TCNB and naphthalene–TCNB. The two molecules are ordered on their site. The angle between the long molecular axis and the *ac* plane is 28·2° for durene and 62·7° for TCNB.

Introduction. Formation of weak π-molecular complexes by planar aromatic donors (D) and acceptors (A) usually leads to columnar structures. This quasi-one-dimensional structure with charge-transfer (CT) forces acting along the D–A stack axis is responsible

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* 1,2,4,5-Tetramethylbenzene.

† 1,2,4,5-Benzenetetracarbonitrile.

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