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Structures of Three Tetrahydrofurofurane-Type Lignanes

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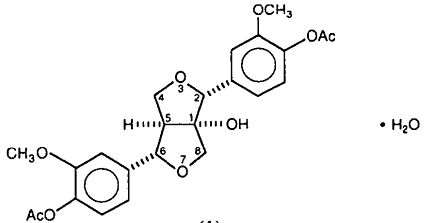
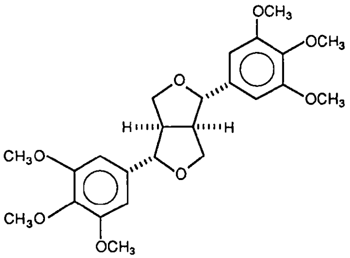
Abstract. (1) (+)-Pinoresinol, 1-hydroxy-4,4′-diacetate monohydrate {4,4′-(tetrahydro-1-hydroxy-1*H*,-3*H*-furo[3,4-*c*]furan-1,4-diyl)bis(2-methoxyphenyl) diacetate monohydrate}, $C_{24}H_{26}O_9.H_2O$, $M_r = 476.5$, orthorhombic, $P2_12_12_1$, $a = 7.230\ (2)$, $b = 7.702\ (1)$, $c = 42.420\ (5)$ Å, $V = 2362.2\ (11)$ Å^3, $Z = 4$, $D_x = 1.340$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54184$ Å, $\mu = 8.4$ cm^{-1}, $F(000) = 1008$, $T = 297$ K, $R = 0.043$ for 3834 observations with $I > 3\sigma(I)$ (of 4808 unique data). The two five-membered rings of the central dioxabicyclooctane system are *cis*-fused, each ring adopting the half-chair conformation with the C atom not carrying the OH group lying on both pseudodiads. The phenyl rings are planar with maximum deviations 0.006 (2) and 0.008 (2) Å, and the two methoxy substituents lie near these planes, with C—C—O—C torsion angles of 7.1 (4) and 2.7 (4)°. The acetate substituents are more nearly orthogonal to the phenyl rings, with C—C—O—C torsion angles of 87.2 (3) and 73.4 (3)°. The OH group donates a hydrogen bond to the water molecule, with O···O distance of 2.795 (3) Å and angle at H of 165 (3)°. (2) Yangambin {tetrahydro-1,4-bis(3,4,5-trimethoxyphenyl)-1*H*,3*H*-furo[3,4-*c*]furan}, $C_{24}H_{30}O_8$, $M_r = 446.5$, orthorhombic, $P2_12_12_1$, $a = 5.5333\ (5)$, $b = 12.5520\ (7)$, $c = 31.723\ (2)$ Å, $V = 2203.2\ (5)$ Å^3, $Z = 4$, $D_x = 1.346$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54184$ Å, $\mu = 8.0$ cm^{-1}, $F(000) = 952$, $T = 299$ K, $R = 0.028$ for 4311 observations having $I > 3\sigma(I)$ (of 4514 unique data). The two five-membered rings of yangambin are *cis*-fused with envelope conformations, with the O atoms at the flap positions. Maximum deviations from planarity are 0.0014 (12) Å for one phenyl substituent and 0.0156 (11) Å for the other. Both phenyl groups carry three methoxy groups, one of the three *para* to the tetrahydrofuran and the other two *meta* to tetrahydrofuran. The *para* methoxy groups are rotated out of the phenyl planes, with C—C—O—C torsion angles of 72.90 (14) and 79.83 (14)°, while the *meta* methoxy groups are more nearly coplanar with the phenyl rings, with analogous magnitudes ranging 0.0 (5)–6.1 (2)°. (3) (−)-Asarinin (episesamin) {5,5′-(tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diyl)bis(1,3-benzodioxole}, $C_{20}H_{18}O_6$, $M_r = 354.4$, monoclinic, $P2_1$, $a = 9.6127\ (10)$, $b = 5.6194\ (4)$, $c = 15.660\ (2)$ Å, $\beta = 103.687\ (7)$°, $V = 821.9\ (3)$ Å^3, $Z = 2$, $D_x = 1.432$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54184$ Å, $\mu = 8.4$ cm^{-1}, $F(000) = 372$, $T = 297$ K, $R = 0.030$ for 1761 observations with $I > 3\sigma(I)$ (of 1829 unique data). Unlike the other two compounds, which have both phenyl substituents *cis* to bridgehead H and thus *syn* to each other, asarinin has its two phenyl substituents *anti* to each other. The fused five-membered rings have the same envelope conformation as those of yangambin. The phenyl rings exhibit maximum deviations from planarity of 0.003 (2) and 0.004 (2) Å. One ketal ring is nearly coplanar with the phenyl group to which it is fused, its methylene C atom lying only 0.012 (3) Å out of the plane. The other ketal ring is in an envelope conformation with that C atom 0.220 (3) Å out of the plane.

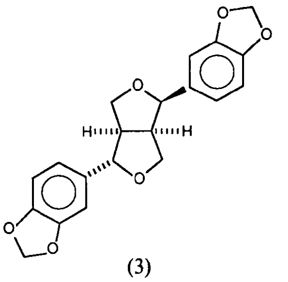
Experimental. Compound (1) was obtained as colorless needles by acetylation of (+)-1-hydroxypinoresinol from *Calamintha ashei* (Weatherby) Shinner (Lamiaceae). Yangambin (2) was isolated from *Rudbeckia maxima* Nutt., *R. nitida* Perdue and *R. scabrifolia* Brown (Asteraceae) which were collected in Alto, Texas, East Baton Rouge Parish, Louisiana and Vernon Parish, Louisiana, USA, respectively. The compound was obtained as colorless needles from chloroform. (−)-Asarinin (episesamin) (3) was isolated from *Pilocarpus gaudotianus* which was collected in Peninsula de Paraguaná, Eds. Falcón, Venezuela. Asarinin (3) was obtained as colorless needles from petroleum ether–ethyl acetate; m.p. 392–394 K, $[\alpha]_D^{25^\circ C} = -119^\circ$ (chloroform, $c = 0.1$).



(1)



(2)



(3)

Intensity data for all three compounds were obtained from fragments of colorless needles, on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Data collection parameters are summarized in Table 1. Variable scan rates were employed in the ω–2θ scans, and a maximum was set on the time spent on a weak reflection. Cell dimensions were determined from setting angles of 25 reflections, including measurements at $\pm 2\theta$. Intensity standards were remeasured every 10 000 s, and exhibited only random fluctuations. Two octants of data were collected within the specified θ limits for each compound. Data reduction included corrections for background, absorption, Lorentz and polarization effects. Absorption corrections were based on ψ scans.

Table 1. *Summary of data collection and structure refinement parameters*

	Pinoresinol derivative (1)	Yangambin (2)	Asarinin (3)
Crystal size (mm)	0.10 × 0.23 × 0.52	0.12 × 0.23 × 0.40	0.10 × 0.25 × 0.45
Reflections used for cell constants, θ range (°)	25–30	23–27	25–30
ω-scan width (°)	$0.50 + 0.14\tan\theta$	$0.60 + 0.14\tan\theta$	$0.65 + 0.14\tan\theta$
Scan speed (° min^{-1})	0.39–3.30	0.46–3.30	0.72–3.30
Maximum scan time per reflection (s)	120	120	90
Range for data collection			
θ	2–75	2–75	2–75
h	0,9	0,6	0,12
k	0,9	0,15	0,7
l	−53,53	−39,39	−19,19
Standard reflections	020, 004, 2,3,13	201, 020, 004	200, 020, 002
Empirical absorption correction			
Maximum transmission coefficient	0.9999	0.9986	0.9944
Minimum transmission coefficient	0.9175	0.9440	0.8954
Reflections measured			
Total	5789	5292	1987
Unique	4808	4514	1829
Observed [$I > 3\sigma(I)$]	3834	4311	1761
$R_{\rm int}$	0.018	0.011	0.009
R, wR	0.043, 0.048	0.028, 0.039	0.030, 0.042
Variables	308	410	307
Maximium shift/e.s.d. ratio (Δ/σ)	0.10	0.03	0.06
Minimum/maximum height in final ΔF (e Å^{-3})	−0.24/0.26	−0.19/0.31	−0.13/0.13
Goodness of fit, S	2.114	2.190	2.680
Extinction, g	$1.7(2) \times 10^{-6}$	$3.31(10) \times 10^{-6}$	$9.0(4) \times 10^{-6}$

Space groups for (1) and (2) were uniquely determined from systematic absences $h00$ with h odd, $0k0$ with k odd, and $00l$ with l odd. The space group of (3) was determined from systematic absences $0k0$ with k odd and knowledge that the compound is chiral. The structures were solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980).

Structures were refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$, using the Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974), and data having $I > 3\sigma(I)$. Non-H atomic coordinates were refined with anisotropic thermal parameters and H-atom coordinates were located by ΔF syntheses. For (1), methyl H atoms were included as fixed contributions with C—H distance 0.95 Å and $B = 1.3\,B_{\rm eq}$ for the bonded atom, while other H atoms were refined with isotropic thermal parameters.

Secondary-extinction coefficients, g, were refined for all structures, with the correction factor $(1 + gI_c)^{-1}$ applied to F_c. Final values of the extinction coefficients, residual densities in final ΔF maps, R factors and other details of the refinements are given in Table 1. Absolute configurations were not determined.

Table 2. *Coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (1)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O3	0.9207 (3)	−0.4844 (2)	0.13660 (3)	5.18 (4)
O7	1.1224 (2)	−0.0475 (2)	0.10722 (3)	4.56 (4)
O9	0.6607 (3)	−0.2121 (3)	0.11044 (4)	5.22 (4)
O22	0.3299 (3)	−0.4954 (2)	0.20221 (4)	5.32 (4)
O24	0.3667 (3)	−0.2401 (2)	0.24404 (3)	4.86 (4)
O25	0.2120 (3)	−0.0502 (3)	0.21451 (5)	6.63 (5)
O28	1.1076 (3)	−0.2328 (2)	−0.03320 (3)	4.16 (3)
O30	1.1222 (2)	0.1121 (2)	−0.04019 (3)	4.43 (4)
O31	0.8285 (2)	0.0433 (2)	−0.05182 (4)	4.71 (4)
C1	0.8539 (3)	−0.2064 (3)	0.11743 (5)	3.92 (5)
C2	0.9035 (3)	−0.3105 (3)	0.14760 (5)	3.91 (5)
C4	1.0280 (4)	−0.4716 (4)	0.10819 (6)	5.77 (6)
C5	0.9638 (4)	−0.3037 (3)	0.09184 (5)	3.98 (5)
C6	1.1223 (3)	−0.1782 (3)	0.08293 (4)	3.58 (5)
C8	0.9324 (4)	−0.0241 (3)	0.11687 (5)	4.61 (6)
C10	0.7641 (3)	−0.2981 (3)	0.17376 (4)	3.84 (5)
C11	0.6163 (4)	−0.4116 (3)	0.17545 (4)	4.00 (5)
C12	0.4810 (4)	−0.3912 (3)	0.19845 (5)	4.01 (5)
C13	0.5001 (4)	−0.2566 (3)	0.21981 (4)	3.92 (5)
C14	0.6449 (4)	−0.1444 (3)	0.21858 (5)	4.88 (6)
C15	0.7804 (4)	−0.1652 (4)	0.19552 (5)	4.62 (6)
C16	1.1055 (3)	−0.0980 (3)	0.05035 (4)	3.21 (4)
C17	1.1029 (3)	−0.2071 (3)	0.02415 (4)	3.23 (4)
C18	1.1036 (3)	−0.1391 (3)	−0.00610 (4)	3.30 (4)
C19	1.1033 (3)	0.0400 (3)	−0.00980 (4)	3.61 (4)
C20	1.1030 (4)	0.1491 (3)	0.01591 (5)	4.34 (5)
C21	1.1045 (4)	0.0797 (3)	0.04602 (5)	4.05 (5)
C23	0.3180 (5)	−0.6479 (4)	0.18346 (7)	6.74 (7)
C26	0.2239 (4)	−0.1303 (3)	0.23836 (5)	4.54 (6)
C27	0.0915 (4)	−0.1317 (4)	0.26511 (6)	5.80 (7)
C29	1.1032 (4)	−0.4166 (3)	−0.03072 (5)	4.16 (5)
C32	0.9766 (4)	0.1006 (3)	−0.05952 (5)	3.70 (5)
C33	1.0233 (5)	0.1686 (4)	−0.09150 (5)	5.49 (6)
O1*W*	0.4197 (3)	−0.0189 (3)	0.14817 (4)	6.29 (5)

Atomic parameters for non-H atoms are given in Tables 2–4, the molecular structures are shown in Figs. 1–3, and selected bond distances, angles and torsion angles are given in Table 5.*

Table 3. *Coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O3	0.6922 (2)	−0.12277 (7)	0.70498 (2)	3.91 (2)
O7	0.2102 (1)	−0.05107 (8)	0.78727 (2)	3.60 (2)
O15	0.9366 (2)	0.22988 (7)	0.61519 (3)	3.64 (2)
O17	0.6514 (2)	0.19228 (7)	0.54991 (2)	3.62 (2)
O19	0.3083 (2)	0.04134 (8)	0.55249 (2)	4.26 (2)
O27	0.1911 (2)	0.03801 (8)	0.95149 (3)	4.40 (2)
O29	0.5226 (2)	0.19364 (7)	0.95282 (3)	3.88 (2)
O31	0.8085 (2)	0.23066 (7)	0.88704 (3)	4.07 (2)
C1	0.5083 (2)	0.01830 (8)	0.74213 (3)	2.67 (2)
C2	0.5068 (2)	−0.04352 (9)	0.70018 (3)	2.93 (2)
C4	0.6648 (3)	−0.1595 (1)	0.74723 (3)	3.85 (2)
C5	0.6239 (2)	−0.06020 (9)	0.77385 (3)	2.59 (2)
C6	0.4354 (2)	−0.07174 (8)	0.80860 (3)	2.62 (2)
C8	0.2568 (2)	0.0383 (1)	0.76052 (4)	3.95 (2)
C9	0.5495 (2)	0.01856 (9)	0.66023 (3)	2.84 (2)
C10	0.7323 (2)	0.09535 (9)	0.65790 (3)	2.93 (2)
C11	0.7641 (2)	0.15346 (9)	0.62095 (3)	2.77 (2)
C12	0.6134 (2)	0.13562 (9)	0.58629 (3)	2.85 (2)
C13	0.4363 (2)	0.0564 (1)	0.58838 (3)	3.10 (2)
C14	0.4035 (2)	−0.00068 (9)	0.62556 (3)	3.10 (2)
C16	1.0954 (2)	0.2520 (1)	0.64917 (4)	3.77 (2)
C18	0.4661 (3)	0.2681 (1)	0.54067 (4)	4.33 (3)
C20	0.1207 (3)	−0.0371 (1)	0.55252 (4)	4.12 (3)
C21	0.4649 (2)	0.00088 (8)	0.84648 (3)	2.54 (2)
C22	0.3124 (2)	−0.01565 (9)	0.88093 (3)	2.93 (2)
C23	0.3278 (2)	0.0494 (1)	0.91609 (3)	3.04 (2)
C24	0.4963 (2)	0.13267 (9)	0.91731 (3)	2.97 (2)
C25	0.6486 (2)	0.14899 (9)	0.88290 (3)	2.92 (2)
C26	0.6326 (2)	0.08324 (9)	0.84769 (3)	2.88 (2)
C28	0.0034 (3)	−0.0400 (1)	0.95100 (4)	4.16 (2)
C30	0.3435 (3)	0.2745 (1)	0.95758 (5)	5.06 (3)
C32	0.9787 (3)	0.2468 (1)	0.85400 (4)	3.84 (2)

Table 4. *Coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (3)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
C1	0.1474 (2)	0.0411 (4)	0.6306 (1)	3.73 (3)
C2	0.2415 (2)	0.0043 (4)	0.7239 (1)	3.79 (3)
O3	0.3849 (1)	0†	0.71252 (7)	4.09 (3)
C4	0.3776 (2)	−0.1360 (4)	0.6344 (1)	4.02 (4)
C5	0.2406 (2)	−0.0538 (4)	0.56975 (9)	3.41 (3)
C6	0.2603 (2)	0.1636 (4)	0.51539 (9)	3.27 (3)
O7	0.1394 (1)	0.3116 (3)	0.51445 (7)	4.53 (3)
C8	0.1187 (2)	0.2993 (5)	0.6017 (1)	4.61 (4)
C9	0.2264 (2)	0.1866 (4)	0.7914 (1)	3.68 (3)
C10	0.3353 (2)	0.3505 (4)	0.8247 (1)	3.75 (3)
C11	0.3117 (2)	0.5105 (4)	0.8859 (1)	3.96 (4)
C12	0.1860 (2)	0.5142 (5)	0.9138 (1)	4.32 (4)
C13	0.0783 (2)	0.3559 (6)	0.8821 (1)	5.37 (5)
C14	0.1004 (2)	0.1926 (6)	0.8203 (1)	4.99 (5)
O15	0.4027 (2)	0.6846 (4)	0.92841 (9)	5.69 (3)
C16	0.3278 (2)	0.8020 (6)	0.9848 (1)	5.45 (5)
O17	0.1930 (2)	0.6912 (4)	0.97593 (9)	5.77 (4)
C18	0.2719 (2)	0.1183 (4)	0.4222 (1)	3.31 (3)
C19	0.2246 (2)	0.2923 (4)	0.3579 (1)	3.56 (3)
C20	0.2433 (2)	0.2465 (4)	0.2751 (1)	3.74 (3)
C21	0.3045 (2)	0.0389 (4)	0.2551 (1)	3.86 (3)
C22	0.3519 (2)	−0.1326 (4)	0.3158 (1)	4.41 (4)
C23	0.3347 (2)	−0.0891 (4)	0.4012 (1)	4.08 (4)
O24	0.2061 (2)	0.3871 (4)	0.20155 (8)	5.38 (3)
C25	0.2683 (3)	0.2759 (6)	0.1379 (1)	6.07 (5)
O26	0.3069 (2)	0.0385 (4)	0.16724 (7)	5.08 (3)

† Coordinate fixed to define origin of structure with non-centrosymmetric space group.

Related literature. A description of lignanes is given by Haworth (1936). Synthesis of (+), (−) and (±) forms of pinoresinol dimethyl ether is reported by Erdtman (1936). Characterization of lignanes having a 2,6-diaryl-*cis*-3,7-dioxobicylco[3.3.0]octane structure is described by Adjangba (1963) and Hearon & MacGregor (1955). Crystal structure determinations of the lignanes (−)-syringaresinol (Bryan & Fallon, 1976), (−)-3,6-bis-(3,4-dimethoxyphenyl)tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diol (Ghisalberti, Jefferies, Skelton & White, 1987) and (+)-pinoresinol dimethyl ether (Vasquez, Fronczek & Fischer, 1990), have been performed. Characterization of yangambin (2) (Pelter, Ward, Rao & Sastry, 1976) and its isolation from *Rudbeckia* species (Vasquez, Quijano, Fronczek, Macias, Urbatsch, Cox & Fischer, 1990) have been described. The crystal structure of sesamin, the epimer at C2 of asarinin, has recently been reported (Baures, Miski & Eggleston, 1992). For a review of lignanes see Massanet, Pando, Rodríguez-Luís & Zubía (1989).

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* Complete lists of distances, angles, torsion angles, least-squares planes, H-atom parameters, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55355 (88 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0597]

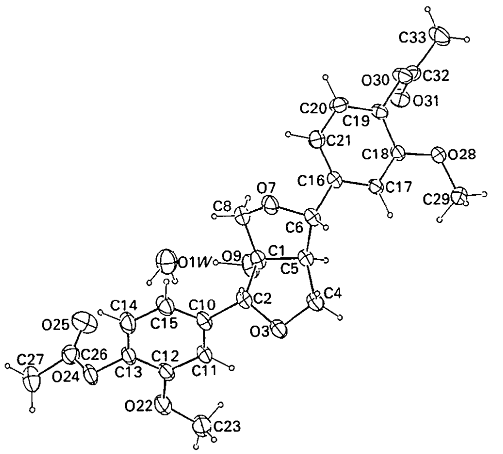

Fig. 1. The structure of pinoresinol derivative (1) monohydrate with thermal ellipsoids drawn at the 30% probability level and H atoms represented by circles of arbitrary radii.

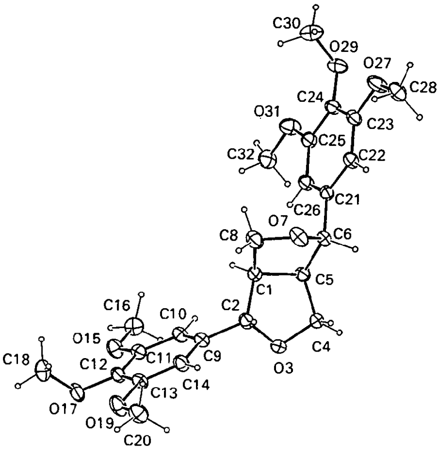

Fig. 2. The structure of yangambin (2) with 30% ellipsoids.

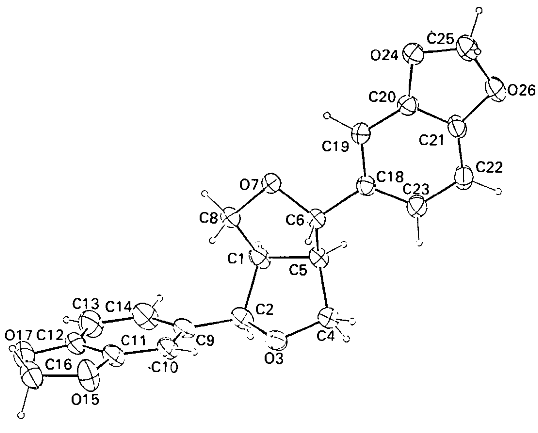

Fig. 3. The structure of asarinin (3) with 30% ellipsoids.

Table 5. *Selected bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

	(1)	(2)	(3)
C1—O9	1.429 (3)	–	–
C1—C2	1.553 (3)	1.541 (2)	1.540 (2)
C1—C5	1.540 (3)	1.547 (2)	1.549 (3)
C1—C8	1.515 (4)	1.529 (2)	1.526 (4)
C2—O3	1.423 (3)	1.437 (2)	1.431 (2)
O3—C4	1.436 (3)	1.426 (2)	1.430 (2)
C4—C5	1.539 (4)	1.523 (2)	1.530 (2)
C5—C6	1.546 (3)	1.525 (2)	1.527 (3)
C6—O7	1.441 (3)	1.441 (2)	1.426 (2)
O7—C8	1.445 (3)	1.430 (2)	1.429 (2)
C2—C1—C5	102.1 (2)	104.09 (8)	104.0 (1)
C2—C1—C8	113.9 (2)	114.03 (10)	115.7 (2)
C5—C1—C8	104.3 (2)	103.47 (9)	103.6 (2)
O3—C2—C1	103.6 (2)	104.66 (8)	104.6 (1)
C2—O3—C4	105.0 (2)	104.35 (9)	105.2 (1)
O3—C4—C5	105.8 (2)	105.79 (9)	105.4 (2)
C1—C5—C4	104.3 (2)	102.84 (8)	103.2 (1)
C1—C5—C6	104.5 (2)	104.34 (8)	103.5 (1)
C4—C5—C6	114.3 (2)	115.14 (10)	114.2 (1)
C5—C6—O7	105.2 (2)	103.60 (8)	105.3 (1)
C6—O7—C8	106.8 (2)	105.29 (9)	105.0 (1)
C1—C8—C7	104.2 (2)	105.16 (10)	105.8 (2)
C5—C1—C2—O3	− 34.0 (2)	− 22.52 (11)	24.1 (2)
C1—C2—O3—C4	45.1 (2)	40.64 (11)	− 41.0 (2)
C2—O3—C4—C5	− 37.3 (3)	− 43.04 (13)	41.7 (2)
O3—C4—C5—C1	14.2 (3)	47.23 (13)	− 24.7 (2)
C4—C5—C1—C2	11.8 (3)	− 2.54 (12)	0.3 (2)
C8—C1—C5—C6	10.3 (2)	− 3.61 (11)	2.3 (2)
C1—C5—C6—O7	13.1 (2)	27.43 (10)	− 25.8 (2)
C5—C6—O7—C8	− 33.3 (2)	− 42.70 (11)	40.9 (2)
C6—O7—C8—C1	40.3 (2)	40.69 (11)	− 39.2 (2)
O7—C8—C1—C5	− 30.3 (2)	− 21.73 (11)	21.8 (2)

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[4-(1-Hydroxyethyl)-5-phenanthryl]methyl Acetate

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Abstract. $C_{19}H_{18}O_3$, $M_r = 294.33$, monoclinic, $P2_1/c$, $a = 7.7157\ (10)$, $b = 27.019\ (3)$, $c = 7.6219\ (10)$ Å, $\beta = 107.473\ (10)°$, $V = 1515.6\ (7)$ Å^3, $Z = 4$, $D_x = 1.290$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.09$ mm^{-1}, $F(000) = 624$, $T = 293$ K, $R = 0.057$ for 2038 reflections. The bulky substituents cause the aromatic ring system to be helical, with a torsion angle C(4)—C(4a)—C(4b)—C(5) of −32° (standard phenanthrene numbering). Atoms C(4) and C(5), bearing the substituents, deviate furthest from the mean aromatic plane (±0.40 Å). The central ring is the most deformed, with a maximum torsion angle of −23° about C(4a)—C(4b). The molecules are linked in pairs by hydrogen bonds from the OH group to the carbonyl O atom, with O···O 2.99 Å.

Experimental. A colourless prism 0.8 × 0.3 × 0.15 mm was mounted in a glass capillary. Using a Stoe–Siemens four-circle diffractometer, 4364 intensities (ω scans) were registered to $2\theta_{max} = 50°$, with monochromated Mo $K\alpha$ radiation. Of 2647 unique reflections ($R_{int} = 0.016$, index ranges h −8 to 8, k 0 to 32, l 0 to 9) 2039 [$F > 4\sigma(F)$] were considered observed. The cell constants were refined from $\pm\omega$ angles of 56 reflections in the 2θ range 20–22°. Three check reflections showed no significant intensity variation. No absorption correction was applied.

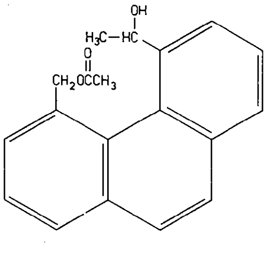

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	417 (3)	6515 (1)	966 (3)	62 (1)
C(2)	292 (3)	6170 (1)	−364 (3)	65 (1)
C(3)	1851 (3)	6012 (1)	−736 (3)	53 (1)
C(4)	3569 (3)	6165 (1)	304 (3)	40 (1)
C(4a)	3739 (3)	6471 (1)	1859 (3)	39 (1)
C(4b)	5459 (3)	6611 (1)	3221 (3)	42 (1)
C(5)	7036 (3)	6310 (1)	3749 (3)	44 (1)
C(6)	8639 (3)	6507 (1)	4878 (3)	61 (1)
C(7)	8713 (5)	6977 (1)	5606 (3)	76 (1)
C(8)	7160 (5)	7245 (1)	5327 (3)	70 (1)
C(8a)	5505 (4)	7064 (1)	4188 (3)	54 (1)
C(9)	3833 (5)	7302 (1)	4155 (3)	64 (1)
C(10)	2237 (4)	7098 (1)	3262 (3)	64 (1)
C(10a)	2113 (3)	6685 (1)	2058 (3)	49 (1)
C(41)	5126 (3)	6058 (1)	−483 (3)	45 (1)
C(42)	4837 (4)	6325 (1)	−2295 (3)	62 (1)
O(4)	5433 (3)	5544 (1)	−656 (3)	69 (1)
C(51)	6999 (3)	5762 (1)	3405 (3)	50 (1)
O(52)	7549 (2)	5538 (1)	5227 (2)	61 (1)
C(52)	7716 (3)	5057 (1)	5319 (3)	59 (1)
O(53)	7482 (4)	4808 (1)	3967 (3)	124 (1)
C(53)	8262 (4)	4863 (1)	7224 (3)	74 (1)

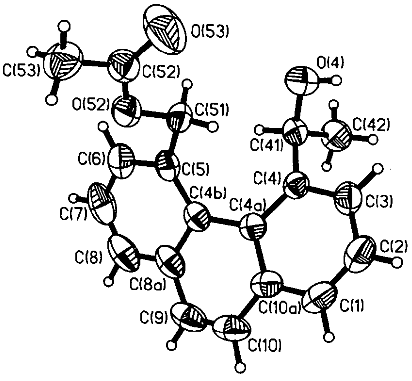

Fig. 1. The molecule of the title compound in the crystal. Thermal ellipsoids are drawn at the 50% level. H-atom radii are arbitrary.

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