(naphthyl)(phenyl)silyl] group: Okaya & Ashida (1966), Nyburg, Brook, Pascoe & Szymanski (1972), Hitchcock (1976), Larson, Sandoval, Cartledge & Fronczek (1983); structure of naphthalene: Cruickshank (1957).

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Structure of the Coumarin Angustifolin

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Abstract. Angustifolin, $C_{14}H_{14}O_3$, $M_r = 230{\cdot}3$, monoclinic, $P2_1/c$, $a = 7{\cdot}5555\ (9)$, $b = 14{\cdot}1532\ (11)$, $c = 11{\cdot}307\ (3)$ Å, $\beta = 99{\cdot}07\ (2)°$, $V = 1193{\cdot}9\ (6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}281$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 6{\cdot}92$ cm^{-1}, $F(000) = 488$, $T = 294$ K, $R = 0{\cdot}040$ for 2205 observations with $I > 3\sigma(I)$ (of 2455 unique data). Crystals were prepared *via* sigmatropic rearrangements from 7-hydroxycoumarin, which was isolated from *Ruta angustifolia* Pers., collected in San Agustin de Gaudalix, Madrid province, Spain. The aromatic ring exhibits a maximum deviation of 0·008 (1) Å from planarity, and the ten atoms of the ring system have a maximum deviation of 0·027 (1) Å from a common plane. The double bond of the prenyl group has a C=C distance of 1·286 (1) Å, and it forms a torsion angle having a magnitude of 31·2 (2)° with one of the C—C(methyl) bonds. Molecules related by the screw axis form linear hydrogen bonds involving the hydroxy group and the carbonyl O atom, having an O···O distance of 2·7064 (7) Å and an angle at H of 174·6 (11)°.

Experimental. Angustifolin was obtained as colorless needles. The sample was a fragment with dimensions 0·43 × 0·62 × 0·65 mm. Space group from absences $h0l$ with l odd and $0k0$ with k odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator, cell dimensions from setting angles of 25 reflections having $30 > \theta > 25°$. Data collection by ω–2θ scans designed for $I = 25\sigma(I)$, subject to maximum scan time = 90 s. Scan rates varied 0·82–3·30° min^{-1}. Reflections having $4 < 2\theta < 150°$, $0 \le h \le 9$, $0 \le k \le 17$, $-14 \le l \le 14$ measured, corrected for background, Lorentz–polarization, and absorption by ψ scans, minimum relative transmission 0·8861. Redundant $0kl$ and $0k\bar{l}$ data average ($R_{int} = 0{\cdot}020$), yielding 2455 unique data. Standard reflections 300, 080, 002, random variation, no decay correction. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ with 2205 data for which $I > 3\sigma(I)$ (250 unobserved reflections), using Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H positions refined with anisotropic thermal parameters; H-atom positions located from difference maps and refined with isotropic thermal parameters. Atomic scattering

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq} (Å^2)
O1	0·8872 (1)	0·37803 (6)	0·66449 (7)	3·67 (2)
O2	0·9087 (1)	0·22758 (7)	0·70957 (9)	5·08 (2)
O3	0·8651 (1)	0·70859 (6)	0·58099 (9)	5·00 (2)
C1	0·8444 (2)	0·28607 (9)	0·6362 (1)	3·60 (2)
C2	0·7257 (2)	0·26543 (8)	0·5247 (1)	3·27 (2)
C3	0·6702 (2)	0·33854 (8)	0·4513 (1)	3·31 (2)
C4	0·7193 (1)	0·43429 (8)	0·4803 (1)	3·07 (2)
C5	0·6649 (2)	0·51189 (9)	0·4068 (1)	3·49 (2)
C6	0·7156 (2)	0·60192 (8)	0·4415 (1)	3·61 (2)
C7	0·8221 (2)	0·61753 (8)	0·5534 (1)	3·52 (2)
C8	0·8780 (2)	0·54233 (9)	0·6284 (1)	3·51 (2)
C9	0·8270 (1)	0·45226 (8)	0·5898 (1)	3·09 (2)
C10	0·6707 (2)	0·16241 (8)	0·4990 (1)	3·77 (2)
C11	0·5826 (2)	0·1273 (1)	0·6031 (1)	4·50 (3)
C12	0·4158 (2)	0·1312 (1)	0·6138 (2)	5·17 (3)
C13	0·5403 (2)	0·1544 (1)	0·3812 (1)	4.64 (3)
C14	0·8367 (2)	0·1017 (1)	0·4878 (2)	5·17 (3)

factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Final $R = 0{\cdot}040$ (0·045 all data), $wR = 0{\cdot}061$, $S = 3{\cdot}564$ for 211 variables, extinction coefficient $g = 4{\cdot}1\ (2) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, maximum shift in final cycle $0{\cdot}03\sigma$, maximum residual density 0·18, minimum $-0{\cdot}19$ e Å^{-3}. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances, angles, and endocyclic torsion angles describing the conformation in Table 2. Fig. 1 shows the atom-numbering scheme, and Fig. 2 illustrates the molecular packing.

Related literature. Description of coumarins [Murray, Mendez & Brown (1982)]; isolation of angustifolin [Castillo del, Rodriguez-Luis & Secundino (1984)]; cytostatic effects on leukemic cells [Gosalvez, Canero & Blanco (1976) and Gonzalez, Darias, Alonso, Boada & Rodriguez-Luis (1977)]; synthesis of C3-prenylated coumarins [Massanet, Pando, Rodriguez-Luis & Salva (1987) and Hernandez-Galan, Massanet, Pando, Rodriguez-Luis & Salva (1988, 1989)]; crystal structure determination of: coumarin [Csoregh (1976) and Gavuzzo, Mazza & Giglio (1974)]; 7-hydroxy-5,6-dimethoxycoumarin (umckalin) [Wagner & Bladt (1975)]; 3-bromo-4-hydroxycoumarin monohydrate [Gaultier & Hauw (1965)]; 7-acetoxycoumarin [Gnanaguru, Ramasubbu, Venkatesan & Ramamurthy (1985)]; 7-ethoxycoumarin (7-*O*-ethylumbelliferone) [Ueno (1985)]; 6,7-dihydroxycoumarin (esculetin) [Ueno & Saito (1977)]; 7-hydroxy-6-methoxycoumarin [Kimura, Watson, Pachecho & Silva (1980)], (±)-3-(1-phenylpropyl)-4-hydroxycoumarin [Bravic, Gaultier & Hauw (1971)]; xanthotoxin [Stemple & Watson (1972)]; (±)-byakangelicol [Fayos (1976)]; bethancorol [Gonzalez, Fraga, Pino, Declercq, Germain & Fayos (1976)]; seselin [Kato (1970)]; (+)-*cis*-

* Lists of H-atom parameters, bond distances and angles involving H atoms, torsion angles, deviations from least-squares planes, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53148 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

O1—C1	1·3667 (8)	C4—C9	1·3935 (9)
O1—C9	1·3793 (7)	C5—C6	1·3700 (9)
O2—C1	1·2173 (8)	C6—C7	1·406 (1)
O3—C7	1·3533 (8)	C7—C8	1·3845 (9)
C1—C2	1·4575 (9)	C8—C9	1·3824 (9)
C2—C3	1·3507 (9)	C10—C11	1·524 (1)
C2—C10	1·5313 (9)	C10—C13	1·531 (1)
C3—C4	1·4294 (9)	C10—C14	1·542 (1)
C4—C5	1·3992 (9)	C11—C12	1·286 (1)

C1—O1—C9	122·63 (5)	O3—C7—C8	123·53 (6)
O1—C1—O2	115·73 (6)	C6—C7—C8	120·42 (6)
O1—C1—C2	118·92 (6)	C7—C8—C9	118·20 (6)
O2—C1—C2	125·33 (7)	O1—C9—C4	119·67 (6)
C1—C2—C3	117·83 (6)	O1—C9—C8	117·40 (5)
C1—C2—C10	117·81 (6)	C4—C9—C8	122·93 (6)
C3—C2—C10	124·35 (6)	C2—C10—C11	107·66 (6)
C2—C3—C4	122·78 (6)	C2—C10—C13	110·74 (6)
C3—C4—C5	124·53 (6)	C2—C10—C14	110·22 (6)
C3—C4—C9	118·07 (6)	C11—C10—C13	110·37 (7)
C5—C4—C9	117·39 (6)	C11—C10—C14	109·95 (7)
C4—C5—C6	121·15 (6)	C13—C10—C14	107·91 (7)
C5—C6—C7	119·90 (6)	C10—C11—C12	127·73 (8)
O3—C7—C6	116·06 (6)		

O2—C1—C2—C10	2·5 (2)	C3—C2—C10—C13	−1·7 (2)
C1—C2—C10—C11	56·87 (14)	C2—C10—C11—C12	89·8 (2)
C1—C2—C10—C13	177·61 (11)	C13—C10—C11—C12	−31·2 (2)

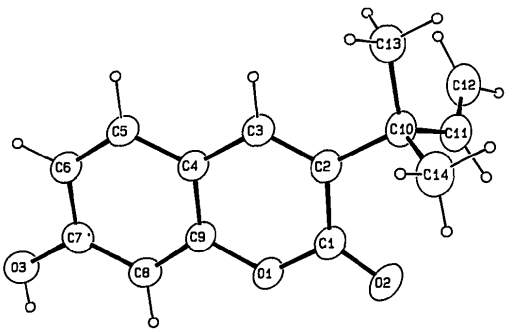

Fig. 1. The molecular structure of angustifolin, with thermal ellipsoids drawn at the 40% probability level, and H atoms represented with arbitrary radius.

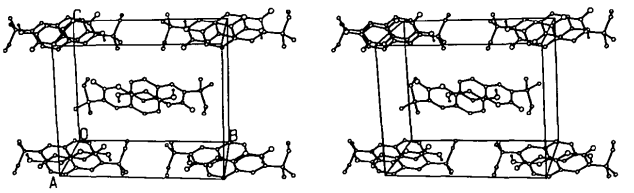

Fig. 2. Stereoview of the unit cell.

khellactone [Bernotat-Wulf, Niggli, Ulrich & Schmid (1969)]; *trans*-4′-methylkhellactone [Macias, Massanet, Rodriguez-Luis, Salva & Fronczek (1989)].

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Structure of a Glycouril Derivative: Tetrahydro-1,6:3,4-di(methanoxymethano)-3a,6a-dimethylimidazo[4,5-*d*]imidazole-2,5(1*H*,3*H*)-dione

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Abstract. $C_{10}H_{14}N_4O_4$, $M_r = 254{\cdot}25$, monoclinic, $C2/c$, $a = 13{\cdot}569$ (3), $b = 6{\cdot}462$ (1), $c = 12{\cdot}820$ (2) Å, $\beta = 103{\cdot}61$ (2)°, $V = 1092{\cdot}5$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}546$ g cm^{-3}, m.p. 573 K (decomposition), λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}1$ cm^{-1}, $F(000) = 536$, $T = 295$ K, $R = 0{\cdot}034$ for 759 observed reflections with $I > 2{\cdot}5\sigma(I)$. The molecule lies on a twofold rotational axis and contains four fused heterocyclic ring systems. The five-membered imidazolidone ring has a distorted envelope conformation and the heteroatomic six-membered ring adopts a chair conformation. The least-squares planes of the five-membered rings are at an angle of 76·1 (1)°.

Experimental. Crystals were obtained by heating an aqueous solution of 3a,6a-dimethylglycouril (Himes, Hubbard, Mighell & Fatiadi, 1978) and formaldehyde, adjusting to pH 1 with concentrated hydrochloric acid and recrystallizing from dimethyl sulfoxide (Niele & Nolte, 1988). Colorless, transparent, plate-shaped crystal of dimensions 0·8 × 0·3 × 0·01 mm was used for data collection on an Enraf–

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