reported (Martin *et al.*, 1989, and references therein). The crystal structure of a precursor of (1) and (2) has also been reported (Lynch, Pacofsky, Martin & Davis, 1989).

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Structure of 6-(4-*n*-Propoxybenzoyloxy)flavone

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Abstract. 4-Oxo-2-phenyl-4*H*-1-benzopyran-6-yl 4-propoxybenzoate, $C_{25}H_{20}O_5$, $M_r = 400{\cdot}43$, triclinic, $P\bar{1}$, $a = 16{\cdot}306\,(11)$, $b = 14{\cdot}830\,(12)$, $c = 13{\cdot}918\,(20)$ Å, $\alpha = 107{\cdot}24\,(9)$, $\beta = 99{\cdot}80\,(9)$, $\gamma = 72{\cdot}33\,(6)°$, $V = 3050\,(5)$ Å^3, $Z = 6$, $D_x = 1{\cdot}306$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}849$ cm^{-1}, $F(000) = 1260$, $T = 298$ K, $R = 0{\cdot}076$, $wR = 0{\cdot}047$ for 10 683 unique observed reflections. There are no unusual bond distances or angles. The flavone skeleton is twisted with respect to the 4-*n*-propoxybenzoyloxy group in the molecule.

Experimental. The title compound, 6-(4-*n*-propoxybenzoyloxy)flavone, was prepared by a conventional dehydrochloride reaction of 6-hydroxyflavone and 4-*n*-propoxybenzoyl chloride in a mixture of benzene and pyridine (Hirose, Tsuya, Nishigaki, Idaka & Yano, 1989) and purified by recrystallization from acetone. A single crystal was obtained as a transparent and elongated rod by slow evaporation from acetone solution. A crystal with approximate dimensions 0·5 × 0·5 × 0·5 mm was mounted on a Rigaku ROTA AFC-5 diffractometer equipped with a graphite monochromator. The lattice parameters were obtained from the setting angles of 25 reflections with $5 < 2\theta < 10°$, Mo $K\alpha$ radiation. Intensities were measured within the ranges $0 < 2\theta < 65°$, $-20 \le h \le 20$, $-20 \le k \le 20$, $0 \le l \le 15$, using ω–2θ scans. Intensities of three standard reflections (520, $42\bar{4}$, 624) were measured every 100 reflections (maximum correction on I was $< 1{\cdot}8\%$). Lorentz and polarization corrections but no absorption correction were applied. 10683 unique observed reflections were measured with $F_o \ge 2\sigma(F_o)$. The structure was determined by direct methods using *RANTAN* (Yao, 1981). The H-atom positions were geometrically obtained, non-H atoms refined with anisotropic thermal parameters. Final refinement including H atoms was made by full-matrix least-squares minimization using *UNICS* (Sakurai, 1967). Atomic scattering factors are from *International Tables for X-ray Crystallography* (1974, Vol. IV). The function $\sum w(F_o - F_c)^2$ was minimized, where $w = 1/\sigma(F_o)^2$. Final $R = 0{\cdot}076$, $wR = 0{\cdot}047$, $S = 5{\cdot}41$,

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Molecule *A*				
O1	0·2651 (2)	1·1072 (2)	−0·2632 (2)	10·2
O2	0·3586 (1)	0·7456 (1)	−0·0582 (1)	7·0
O3	0·2270 (1)	0·8346 (1)	−0·0101 (1)	5·6
O4	0·2109 (1)	0·5813 (1)	0·2018 (1)	4·6
O5	0·2276 (1)	0·8549 (1)	0·3594 (1)	7·4
C1	0·2868 (4)	1·2799 (3)	−0·3050 (5)	13·8
C2	0·2934 (3)	1·1875 (3)	−0·3730 (4)	12·2
C3	0·3239 (3)	1·0980 (3)	−0·3289 (3)	13·0
C4	0·2803 (2)	1·0298 (2)	−0·2142 (2)	7·0
C5	0·3478 (2)	0·9457 (2)	−0·2280 (2)	7·2
C6	0·3533 (2)	0·8769 (2)	−0·1757 (2)	6·3
C7	0·2906 (2)	0·8919 (2)	−0·1116 (2)	5·9
C8	0·2236 (2)	0·9786 (2)	−0·0994 (2)	5·4
C9	0·2181 (2)	1·0474 (2)	−0·1502 (2)	6·1
C10	0·2989 (2)	0·8160 (2)	−0·0594 (2)	5·0
C11	0·2275 (1)	0·7685 (2)	0·0442 (2)	4·6
C12	0·2280 (2)	0·8010 (2)	0·1461 (2)	4·7
C13	0·2221 (1)	0·7396 (2)	0·2018 (2)	4·2
C14	0·2163 (1)	0·6460 (2)	0·1515 (2)	4·2
C15	0·2159 (1)	0·7123 (2)	0·0478 (2)	4·2
C16	0·2214 (2)	0·6741 (2)	−0·0062 (2)	5·1
C17	0·2218 (2)	0·7720 (2)	0·3115 (2)	5·2
C18	0·2149 (2)	0·6997 (2)	0·3569 (2)	5·4
C19	0·2112 (1)	0·6095 (2)	0·3041 (2)	4·3
C20	0·2089 (1)	0·5304 (2)	0·3457 (2)	4·4
C21	0·2188 (2)	0·4356 (2)	0·2865 (2)	5·5
C22	0·2199 (2)	0·3606 (2)	0·3274 (2)	6·5
C23	0·2108 (2)	0·3793 (2)	0·4279 (2)	6·3
C24	0·2001 (2)	0·4739 (2)	0·4879 (2)	6·3
C25	0·1993 (2)	0·5488 (2)	0·4476 (2)	5·5
Molecule *B*				
O1′	0·1149 (1)	0·4247 (1)	−0·1175 (1)	10·4
O2′	0·1380 (1)	0·1529 (1)	0·1638 (1)	7·7
O3′	−0·0006 (1)	0·1730 (1)	0·0997 (1)	5·5
O4′	−0·0350 (1)	−0·0642 (1)	0·3200 (1)	4·8
O5′	−0·1766 (1)	0·2213 (1)	0·3931 (1)	5·3
C1′	0·0249 (3)	0·5631 (3)	−0·3075 (4)	11·2
C2′	0·0860 (3)	0·5219 (3)	−0·2303 (3)	10·1
C3′	0·0482 (2)	0·4635 (2)	−0·1843 (2)	7·7
C4′	0·1001 (2)	0·3671 (2)	−0·0675 (2)	5·6
C5′	0·1707 (2)	0·3265 (2)	−0·0080 (2)	6·3
C6′	0·1632 (2)	0·2685 (2)	0·0473 (2)	5·7
C7′	0·0843 (2)	0·2490 (2)	0·0454 (2)	4·7
C8′	0·0149 (2)	0·2884 (2)	−0·0149 (2)	5·4
C9′	0·0215 (2)	0·3471 (2)	−0·0718 (2)	6·2
C10′	0·0803 (2)	0·1871 (2)	0·1087 (2)	5·2
C11′	−0·0107 (2)	0·1136 (2)	0·1566 (2)	4·8
C12′	−0·0660 (2)	0·1545 (2)	0·2305 (2)	4·2
C13′	−0·0766 (1)	0·0959 (2)	0·2876 (2)	3·7
C14′	−0·0307 (1)	−0·0023 (2)	0·2660 (2)	4·2
C15′	0·0234 (2)	−0·0445 (2)	0·1881 (2)	5·2
C16′	0·0333 (2)	0·0141 (2)	0·1333 (2)	5·5
C17′	−0·1334 (1)	0·1351 (2)	0·3706 (2)	3·9
C18′	−0·1332 (1)	0·0650 (2)	0·4240 (2)	4·2
C19′	−0·0858 (1)	−0·0287 (2)	0·3993 (2)	4·1
C20′	−0·0782 (1)	−0·1054 (2)	0·4499 (2)	4·3
C21′	−0·1197 (2)	−0·0814 (2)	0·5373 (2)	5·6
C22′	−0·1115 (2)	−0·1528 (2)	0·5868 (2)	6·8
C23′	−0·0635 (2)	−0·2467 (2)	0·5492 (3)	7·6
C24′	−0·0212 (2)	−0·2712 (2)	0·4632 (3)	7·5
C25′	−0·0285 (2)	−0·2009 (2)	0·4129 (2)	6·1
Molecule *C*				
O1″	−0·3805 (1)	0·8776 (1)	−0·2289 (2)	8·6
O2″	−0·4807 (1)	0·7866 (1)	0·1500 (1)	8·5
O3″	−0·4473 (1)	0·6383 (1)	0·0395 (1)	6·5
O4″	−0·4981 (1)	0·4538 (1)	0·3058 (1)	5·5
O5″	−0·2510 (1)	0·4105 (1)	0·2443 (1)	7·0
C1″	−0·4276 (4)	0·9918 (4)	−0·3766 (4)	15·2
C2″	−0·3489 (3)	0·9855 (3)	−0·3070 (4)	13·1
C3″	−0·3676 (2)	0·9741 (3)	−0·2071 (3)	10·2
C4″	−0·3978 (2)	0·8479 (2)	−0·1534 (2)	6·3
C5″	−0·4074 (2)	0·7525 (2)	−0·1798 (2)	6·2
C6″	−0·4255 (2)	0·7158 (2)	−0·1091 (2)	5·8
C7″	−0·4360 (2)	0·7717 (2)	−0·0098 (2)	5·5
C8″	−0·4259 (2)	0·8657 (2)	0·0154 (2)	6·3
C9″	−0·4062 (2)	0·9032 (2)	−0·0543 (2)	6·7
C10″	−0·4582 (2)	0·7378 (2)	0·0693 (2)	6·1
C11″	−0·4613 (2)	0·5948 (2)	0·1102 (2)	5·7

Table 1 (*cont.*)

	x	y	z	B_{eq}
C12″	−0·3894 (2)	0·5361 (2)	0·1500 (2)	5·2
C13″	−0·4002 (2)	0·4869 (2)	0·2165 (2)	4·7
C14″	−0·4828 (2)	0·4994 (2)	0·2409 (2)	5·0
C15″	−0·5548 (2)	0·5589 (2)	0·1997 (2)	6·7
C16″	−0·5440 (2)	0·6063 (2)	0·1342 (2)	6·8
C17″	−0·3264 (2)	0·4231 (2)	0·2617 (2)	5·1
C18″	−0·3486 (2)	0·3775 (2)	0·3277 (2)	5·1
C19″	−0·4306 (2)	0·3931 (2)	0·3485 (2)	4·7
C20″	−0·4579 (2)	0·3512 (2)	0·4171 (2)	5·0
C21″	−0·4005 (2)	0·2733 (2)	0·4503 (2)	6·8
C22″	−0·4236 (2)	0·2350 (2)	0·5178 (3)	8·2
C23″	−0·5045 (2)	0·2740 (2)	0·5521 (2)	8·1
C24″	−0·5610 (2)	0·3496 (3)	0·5185 (2)	5·4
C25″	−0·5397 (2)	0·3893 (2)	0·4518 (2)	5·8

Table 2. *Selected bond distances* (Å) *and angles* (°) (*average values from molecules A, B and C*)

O2—C10	1·193 (4)	C10—O3—C11	116·8 (2)
O3—C10	1·374 (3)	O2—C10—O3	122·0 (3)
O3—C11	1·405 (3)	O2—C10—C7	126·6 (3)
C7—C10	1·472 (4)	O3—C10—C7	111·4 (2)

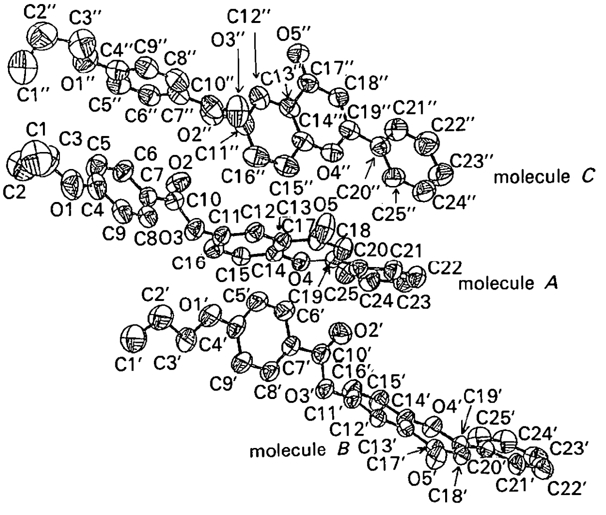

Fig. 1. *ORTEP*II view showing independent molecules *A*, *B* and *C* in the unit cell projected onto the *ac* plane with the numbering of the atoms. Thermal ellipsoids are drawn at 50% probability.

$(\Delta/\sigma)_{max} = 1·167$ (x coordinate of C3). Maximum peak in final difference Fourier synthesis is 0·32 e Å^{-3}. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Table 2 shows selected bond lengths and angles for the non-H atoms. Fig. 1, obtained using *ORTEP*II (Johnson, 1976), shows the molecular structures and numbering scheme. The flavone skeleton is twisted with respect to the 4-*n*-propoxybenzoyloxy group in the molecule. The angle C10—O3—C11—C12 in

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53641 (55 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecule *A* is 63·91°, that in molecule *B* is 65·11°, and that in molecule *C* is 75·43° (see Fig. 1).

Related literature. The crystal structures of flavone derivatives have been reported by several workers (Cantrell & Stalzer, 1982; Castleden, Hall, Nimgirawath, Thadaniti & White, 1985).

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10,11-Dihydroquinidine Sesquihydrate

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Abstract. $C_{20}H_{26}N_2O_2.1{\cdot}5H_2O$, $M_r = 353{\cdot}44$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}358$ (1), $b = 17{\cdot}631$ (1), $c = 17{\cdot}686$ (2) Å, $V = 3853$ (1) Å^3, $Z = 8$, $D_m = 1{\cdot}20$ (2), $D_x = 1{\cdot}217$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}91$ cm^{-1}, $F(000) = 1528$, $T = 293$ K, $R = 0{\cdot}0522$, $wR = 0{\cdot}0631$ for 4147 unique observed reflections. The asymmetric unit contains two alkaloid and three water molecules. The two independent molecules have identical geometries with the exception of the disordered ethyl chain in molecule *B*. The bond lengths and angles are in the expected ranges. The molecules are hydrogen bonded by O—H···N and O—H···O intermolecular contacts.

Experimental. 10,11-Dihydroquinidine is found with other alkaloids in *Cinchona* barks. Small amounts of this dihydrobase can be found in commercial quinidine (Suszko-Purzycka & Trzebny, 1965). The title compound was isolated from commercial quinidine by the mercuric acetate process (Thorn & Dirscherl, 1935) (based on the difference in solubility of the dihydroquinidine and the addition product of mercuric acetate to the vinyl alkaloid in aqueous ammonia). Further purification was carried out using column chromotagraphy, in the same way as for the quinine (Pniewska & Suszko-Purzycka, 1989). The alkaloid was obtained from the toluene–methanol eluate and recrystallized from acetone. Colourless prismatic crystals were grown by slow evaporation of acetone solution. D_m was determined by flotation in KI solution. A crystal 0·52 × 0·40 × 0·40 mm was used for diffractometer measurements. Data were collected with an Enraf–Nonius CAD-4 diffractometer using Cu $K\alpha$ radiation and a graphite monochromator. Unit-cell parameters were refined from the setting angles of 25 reflections having $20 < \theta < 30°$. 4426 reflections were measured with $1 < \theta < 75°$; index ranges: $0 \le h \le 15$, $0 \le k \le 22$, $0 \le l \le 22$, ω–2θ scan method, $\Delta\omega(\theta) = (1{\cdot}05 + 0{\cdot}14\tan\theta)°$. 0·5% intensity variation for three standard reflections measured every 1 h; no absorption correction. 4386 unique reflections of which 4147 with $F > 3\sigma(F)$. Structure solved by direct methods with *SHELXS*86 (Sheldrick, 1986) and refined with *SHELX*76 (Sheldrick, 1976) programs. Most non-H atoms were revealed from the first *E* map; C(10*B*) and C(11*B*) positions and the O atoms of the water molecules were located from a difference Fourier map. Anisotropic refinement of all non-H atoms gave non-positive-definite temperature tensors and implausible bond lengths for the C atoms of the ethyl group in molecule *B*. A difference Fourier map showed two alternative sites for the C(10*B*) and C(11*B*) atoms; site occupation refinement converged to 0·50 and these values and isotropic thermal parameters were used in further refinement. H atoms were located from difference maps and refined isotropically, except those for the disordered C(10*B*) and C(11*B*) atoms. Water H atoms could not be located and were omitted. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1{\cdot}0/[\sigma^2(F_o) + 0{\cdot}007334F_o^2]$; final value for isotropic extinction parameter $g = 2{\cdot}5\,(2) \times 10^{-6}$,

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