Fig. 2. Molecular conformations of (1) and (2). The shaded atoms refer to (1), the unshaded ones to (2), with superimposed atoms black.

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5,6-Benzochroman-4-one*

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Abstract. $C_{13}H_{10}O_2$, $M_r = 198\cdot2$, orthorhombic, *Pccn*, $a = 11\cdot6430$ (3), $b = 19\cdot4230$ (4), $c = 8\cdot5480$ (2) Å, $V = 1933\cdot06$ (8) Å^3, $Z = 8$, $D_m = 1\cdot35$ (2) (flotation), $D_x = 1\cdot362$ (1) Mg m^{-3}, $\lambda(Cu\,K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot698$ mm^{-1}, $F(000) = 832$, $T = 294$ K, $R = 0\cdot052$ for 1443 observed reflections. The dihydropyran ring system is a modified half-chair distorted toward the *d,e* diplanar arrangement. The benzene ring common to the

naphthalene and benzopyran groups deviates from planarity owing to fusion strains.

Introduction. The benzopyran nucleus is a widely prevalent ring system, present in a variety of naturally occurring compounds like carbohydrates, xanthones, flavones *etc.* The crystal structure determination of the title compound is part of a research programme which is being carried out on the conformation of the oxygen heterocyclic compounds.

Experimental. The title compound was prepared according to the procedure of Kasturi & Arunachalam (1970). Crystals from benzene–hexane mixture,

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* IUPAC name: 2,3-dihydro-1*H*-naphtho[2,1-*b*]pyran-1-one. *Cf. Acta Cryst.* (1986). C**42**, 1043–1044 for the structure of a similar compound.

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0·15 × 0·15 × 0·36 mm, CAD-4 diffractometer, monochromatized Cu $K\alpha$ radiation, cell parameters from $\sin^2\theta$ values for 25 reflections (θ range 40–50°), intensity data for $0 < \theta < 75°$, ω–2(5/6) θ scan, ω-scan angle (0·90 + 0·14 tanθ)°, extended by 25% on each side for background measurement, horizontal aperture (2·0 + tanθ) mm, vertical aperture 4 mm, three standard reflections (2,12,$\bar{1}$, $66\bar{1}$, 554) measured every 60 min displayed <2% average fluctuation, Lp but no absorption corrections, 2313 unique reflections, h 0→14, k 0→24, l 0→10, R_{int} from merging equivalent reflections = 0·032; 1443 (62%) with $I \geq 3\sigma(I)$, $\sigma^2(I) = C + 2B + [0\cdot04(C-B)]^2$, $C =$ scan count, $B =$ time-corrected background. Direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). An E map calculated from the set of phases with the highest figure of merit revealed the structure. Full-matrix least-squares refinement on $|F|$. Anisotropic temperature factors for C and O atoms, isotropic for H. H positions from $\Delta\rho$ map. $w = [\sigma^2(F_o) + 0\cdot003 F_o^2]^{-1}$, final R = 0·052, $wR = 0\cdot057$ for 1443 observed reflections, $S = 1\cdot26$, final $\Delta\rho$ map featureless, $(\Delta/\sigma)_{max} = 0\cdot43$, final $\Delta\rho$ excursions $-0\cdot21$, $0\cdot25$ e Å^{-3}. No correction for secondary extinction, scattering factors as in *SHELX* (Sheldrick, 1976). Calculations performed on an IBM 370 computer.

Discussion. A perspective view of the title compound with the atomic numbering is shown in Fig. 1. Atomic coordinates and equivalent isotropic temperature factors of non-H atoms are reported in Table 1* and bond distances, angles and selected torsion angles in Table 2. The average e.s.d. for bond lengths is 0·003 Å and for bond angles 0·2°.

In all essential details the geometry of the molecule in terms of the bond lengths and angles shows normal values. The C—O bond in the pyran ring is asymmetrical [C(1)—O(1), 1·430 (2); C(5)—O(1), 1·365 (3) Å]

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses for the non-hydrogen atoms*

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}(Å^2)
C(1)	0·9792 (2)	0·2853 (1)	0·0189 (3)	4·8
C(2)	1·0929 (2)	0·2510 (1)	0·0498 (3)	4·7
C(3)	1·0753 (2)	0·1820 (1)	0·1280 (2)	3·6
C(4)	0·9698 (1)	0·1452 (1)	0·0837 (2)	3·1
C(5)	0·8962 (1)	0·1762 (1)	−0·0231 (2)	3·4
C(6)	0·8017 (2)	0·1413 (1)	−0·0896 (2)	4·1
C(7)	0·7792 (2)	0·0757 (1)	−0·0458 (2)	4·1
C(8)	0·8470 (1)	0·0414 (1)	0·0671 (2)	3·4
C(9)	0·9434 (1)	0·0757 (1)	0·1338 (2)	3·1
C(10)	1·0091 (2)	0·0388 (1)	0·2451 (2)	3·8
C(11)	0·9802 (2)	−0·0270 (1)	0·2873 (3)	4·5
C(12)	0·8859 (2)	−0·0602 (1)	0·2217 (3)	4·7
C(13)	0·8209 (2)	−0·0267 (1)	0·1127 (3)	4·3
O(1)	0·9092 (1)	0·2419 (1)	−0·0769 (2)	4·4
O(2)	1·1480 (1)	0·1599 (1)	0·2171 (2)	5·2

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

O(1)–C(1)	1·430 (3)	C(6)–C(7)	1·354 (3)
O(1)–C(5)	1·365 (3)	C(7)–C(8)	1·414 (3)
C(1)–C(2)	1·504 (3)	C(8)–C(9)	1·424 (2)
C(2)–C(3)	1·515 (3)	C(9)–C(10)	1·416 (3)
C(3)–O(2)	1·217 (3)	C(10)–C(11)	1·370 (3)
C(3)–C(4)	1·471 (3)	C(11)–C(12)	1·391 (3)
C(4)–C(5)	1·389 (2)	C(12)–C(13)	1·365 (3)
C(4)–C(9)	1·449 (3)	C(13)–C(8)	1·412 (3)
C(5)–C(6)	1·412 (3)		

C(1)–O(1)–C(5)	114·9 (1)	C(5)–C(6)–C(7)	119·4 (1)
O(1)–C(1)–C(2)	109·8 (2)	C(6)–C(7)–C(8)	121·6 (1)
C(1)–C(2)–C(3)	110·4 (2)	C(7)–C(8)–C(9)	119·5 (1)
C(2)–C(3)–C(4)	115·4 (2)	C(7)–C(8)–C(13)	120·6 (1)
C(2)–C(3)–O(2)	119·8 (2)	C(13)–C(8)–C(9)	119·8 (1)
O(2)–C(3)–C(4)	124·8 (2)	C(8)–C(9)–C(4)	119·0 (1)
C(3)–C(4)–C(5)	118·3 (1)	C(8)–C(9)–C(10)	117·3 (1)
C(3)–C(4)–C(9)	123·6 (1)	C(10)–C(9)–C(4)	123·6 (1)
C(5)–C(4)–C(9)	117·8 (1)	C(9)–C(10)–C(11)	121·1 (1)
C(4)–C(5)–O(1)	123·9 (1)	C(10)–C(11)–C(12)	121·3 (2)
C(4)–C(5)–C(6)	122·5 (1)	C(11)–C(12)–C(13)	119·4 (2)
C(6)–C(5)–O(1)	113·6 (1)	C(12)–C(13)–C(8)	121·0 (2)

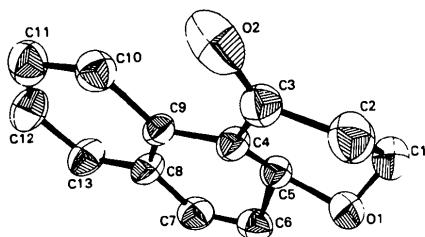| C(1)–C(2)–C(3)–C(4)	32·6 (2)	C(4)–C(5)–O(1)–C(1)	−19·9 (3)
C(2)–C(3)–C(4)–C(5)	−0·6 (2)	C(5)–O(1)–C(1)–C(2)	52·7 (2)
C(3)–C(4)–C(5)–O(1)	−7·4 (3)	O(1)–C(1)–C(2)–C(3)	−58·4 (2)

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the title compound. Thermal ellipsoids are at the 50% probability level.
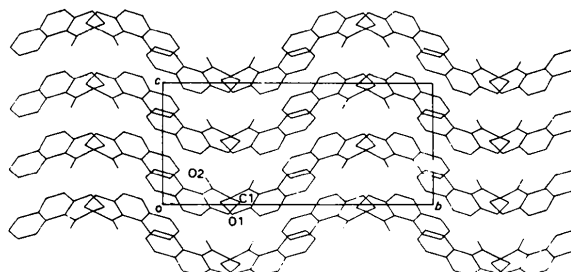

Fig. 2. Molecular packing of the title compound viewed along the a axis.

owing to the effect of conjugation between the oxygen and the benzene ring (Spek, Kojić-Prodić & Labadie, 1984). This conjugation could be justified by the coplanarity of the ether oxygen O(1) with the benzene ring [O(1)—C(5)—C(6)—C(7), −179·9 (2); O(1)—C(5)—C(4)—C(9), 178·2 (2)°] which causes an enlargement of the angle O(1)—C(5)—C(4), 123·9 (1)°, and narrowing of the angle O(1)—C(5)—C(6), 113·6 (1)°. Similar observations were noted in the solid-state structures of a variety of compounds incorporating the pyran ring (Cantrell & Hockstein, 1982; Cantrell, Stalzer & Becker, 1974; Ponnuswamy & Trotter, 1984) and anisole moiety (Domiano, Nardelli, Balsamo, Macchia & Macchia, 1979). The C(5)—O(1)—C(1), C(4)—C(3)—C(2) angles reflect the ring strain due to the neighbouring sp^2 atoms (Gzella, Jaskólski, Rychlewska & Kosturkiewicz, 1984).

The conformation of the dihydropyran ring is a modified half-chair distorted toward the d,e diplanar arrangement (Valente, Santarsiero & Schomaker, 1979). The corresponding asymmetry parameter (Duax, Weeks & Rohrer, 1976) $\Delta C_2[C(1)—C(2)] = 19·7°$.

On account of fusion strains the benzene ring [atoms C(4) to C(9)] common to the naphthalene and benzopyran groups deviates from planarity [$\sum(\Delta/\sigma)^2 = 360$]; maximum deviations from the plane are 0·019 (1), −0·015 (1), 0·013 (2) Å for C(4), C(5) and C(7) respectively.

The molecular packing projected on the bc plane is illustrated in Fig. 2. There is a shortened intramolecular distance C(10)···O(2), 2·864 (3) Å. C(10)—H, 0·98 (2); O(2)···H, 2·19 (2) Å; angle C(10)—H···O(2), 125·5 (15)° which corresponds, according to the geometrical characteristics, to a weak C—H···O hydrogen bond (Taylor & Kennard, 1982; Berkovitch-

Yellin & Leiserowitz, 1984; Dvorkin, Malinowsky, Simonov, Andronati, Kuzmina & Yavorsky, 1985).

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Ethyl 3-Phenyl-4H-pyrazolo[3,2-c][1,4]benzothiazine-2-carboxylate (I) and 4-[Chloro-(ethoxycarbonyl)methyleneamino]-2-methylene-2,3-dihydro-4H-1,4-benzothiazine 1,1'-Dioxide (II)

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Abstract. (I): $C_{19}H_{16}N_2O_2S$, $M_r = 336·4$, monoclinic, $P2_1/c$, $a = 9·686$ (2), $b = 7·660$ (2), $c = 21·989$ Å, $\beta = 98·31$ (2)°, $V = 1614·3$ (6) Å³, $Z = 4$, $D_m = 1·37$, $D_x = 1·384$ Mg m⁻³; λ(Mo Kα) = 0·71069 Å, $\mu = 0·20$ mm⁻¹, $F(000) = 704$, room temperature, $R = 0·045$, $wR = 0·031$ for 2299 observed reflections

† Deceased.