O1*B*	0.1905 (2)	0.72636 (3)	0.8964 (2)	0.0306 (3)
O2*B*	−0.1015 (2)	0.67398 (3)	0.8514 (2)	0.0318 (3)
O3*B*	−0.2381 (2)	0.78377 (3)	0.8292 (2)	0.0285 (3)
C1*B*	0.0900 (3)	0.80473 (5)	0.8833 (3)	0.0332 (4)
C2*B*	−0.0474 (2)	0.77285 (4)	0.8591 (2)	0.0243 (3)
C3*B*	0.0009 (2)	0.73626 (4)	0.8666 (2)	0.0232 (3)
C4*B*	−0.1469 (2)	0.70756 (4)	0.8452 (2)	0.0237 (3)
C5*B*	−0.3431 (2)	0.72099 (5)	0.8163 (3)	0.0265 (3)
C6*B*	−0.3784 (2)	0.75781 (5)	0.8089 (3)	0.0299 (4)
O1*C*	0.4277 (2)	0.56081 (3)	0.4663 (2)	0.0267 (3)
O2*C*	0.7144 (2)	0.50828 (3)	0.4955 (2)	0.0355 (3)
O3*C*	0.8266 (2)	0.61682 (3)	0.4193 (2)	0.0272 (3)
C1*C*	0.5152 (3)	0.63870 (4)	0.4205 (3)	0.0303 (4)
C2*C*	0.6482 (2)	0.60656 (4)	0.4325 (2)	0.0236 (3)
C3*C*	0.6063 (2)	0.57026 (4)	0.4546 (2)	0.0212 (3)
C4*C*	0.7507 (2)	0.54141 (4)	0.4688 (2)	0.0239 (3)
C5*C*	0.9342 (2)	0.55404 (5)	0.4493 (3)	0.0271 (3)
C6*C*	0.9623 (2)	0.59056 (5)	0.4271 (3)	0.0287 (4)

Table 4. *Selected geometric parameters (Å, °) for (2)*

O1*A*—C3*A*	1.356 (2)	C2*B*—C3*B*	1.355 (2)
O2*A*—C4*A*	1.248 (2)	C3*B*—C4*B*	1.449 (2)
O3*A*—C6*A*	1.347 (2)	C4*B*—C5*B*	1.435 (2)
O3*A*—C2*A*	1.365 (2)	C5*B*—C6*B*	1.344 (2)
C1*A*—C2*A*	1.484 (2)	O1*C*—C3*C*	1.352 (2)
C2*A*—C3*A*	1.359 (2)	O2*C*—C4*C*	1.245 (2)
C3*A*—C4*A*	1.441 (2)	O3*C*—C6*C*	1.343 (2)
C4*A*—C5*A*	1.438 (2)	O3*C*—C2*C*	1.363 (2)
C5*A*—C6*A*	1.338 (2)	C1*C*—C2*C*	1.481 (2)
O1*B*—C3*B*	1.351 (2)	C2*C*—C3*C*	1.358 (2)
O2*B*—C4*B*	1.246 (2)	C3*C*—C4*C*	1.444 (2)
O3*B*—C6*B*	1.343 (2)	C4*C*—C5*C*	1.440 (2)
O3*B*—C2*B*	1.368 (2)	C5*C*—C6*C*	1.344 (2)
C1*B*—C2*B*	1.482 (2)		

Table 5. *Hydrogen-bonding parameters (Å, °)*

D	*A*	*D*···*A*	*D*—H†	H···*A*†	*D*—H···*A*†
Polymorph (1)					
O1*A*	O2*B*^{i}	2.738 (3)	0.86	2.035	146
O1*B*	O2*A*	2.752 (3)	0.81	2.102	138
C5*A*	O2*B*	3.193 (4)	0.96	2.410	139
C5*B*	O2*A*^{ii}	3.287 (4)	0.85	2.616	137
Polymorph (2)					
O1*A*	O2*B*	2.718 (1)	0.82	1.949	156
O1*B*	O2*A*	2.700 (1)	0.88	1.876	156
O1*C*	O2*C*^{iii}	2.731 (1)	0.86	1.925	155
C5*A*	O2*B*^{iv}	3.309 (2)	0.92	2.397	172
C5*B*	O2*A*^{v}	3.341 (2)	0.94	2.434	162
C5*C*	O2*C*^{vi}	3.293 (2)	0.93	2.364	175

† Uncertainties when H atoms were refined freely were approximately 0.05 Å. However, no e.s.d.'s are given here as in the final cycles, a riding model was used for the H atoms.

Symmetry codes: (i) $\frac{1}{2} - x, y, \frac{1}{2} - z$; (ii) $\frac{1}{2} - x, y, \frac{1}{2} + z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $1 + x, y, z$; (v) $x - 1, y, z$; (vi) $2 - x, 1 - y, 1 - z$.

Data collection: *XSCANS* (Fait, 1991) for (1); *CAD*-4 *Software* (Enraf–Nonius, 1989) for (2). Cell refinement: *XSCANS* for (1); local program for (2). Data reduction: *XSCANS* for (1); local program for (2). Program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1990*a*) for (1); *SHELXS*86 (Sheldrick, 1990*b*) for (2). For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL/PC XP*; software used to prepare material for publication: *SHELXTL/PC*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,2-Dimethyl-6-nitro-3,4-dihydro-2*H*-benzo[*b*]pyran-4-one *O*-Benzoyloxime

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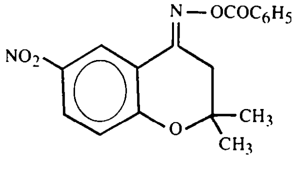
Abstract

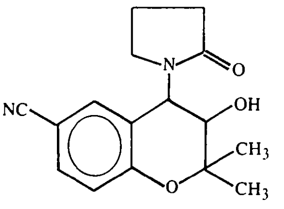
The title compound, $C_{18}H_{16}N_2O_5$, has a pyran ring in a half-chair conformation fused to a benzene ring. The substituent groups at C3 and C5 are approximately coplanar with the benzopyran nucleus, with dihedral angles of 8.5 (5) and 4.2 (5)°, respectively. The benzoyloxy carbonyl group and the benzene ring of the benzopyran moiety both adopt a *cis* configuration.

Comment

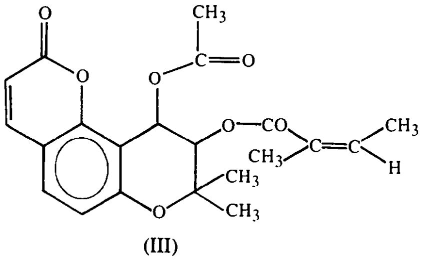
There is interest at present in the structures of benzopyran compounds and their biological activity as potassium-channel activators which relax smooth muscle and lower blood pressure (Arch *et al.*, 1991; Buckle *et al.*, 1990; Buckle, Arch *et al*, 1991; Buckle, Eggleston *et al.*, 1991; Burrell, Cassidy, Evans, Lightowler & Stemp, 1990; Cassidy *et al.*, 1989). A representative compound is cromakalim [(II); Cassidy *et al.*, 1989]. Praeruptorin C, (III), isolated from the Chinese herbal drug 'Qian-Hu', is also a benzopyran compound and has been shown to cause inhibition of the calcium-induced tension development in vascular smooth muscle and myocardial muscle (Rao, Shen & Zou, 1988). As part of a systematic study based on the molecular features of compounds (II) and (III), the title compound, (I), has been prepared.



(I)



(II)



(III)

Tests show that (I) is active in inhibiting calcium entry into smooth muscle cells induced by high K$^+$ concentrations. The single-crystal X-ray analysis of (I) shows that it contains a pyran ring in a half-chair conformation in the solid state; atoms C1 and C2 lie 0.294 (4) and −0.359 (4) Å, respectively, from the mean plane through atoms C3, C9, C8 and O1 [maximum deviation 0.002 (4) Å].

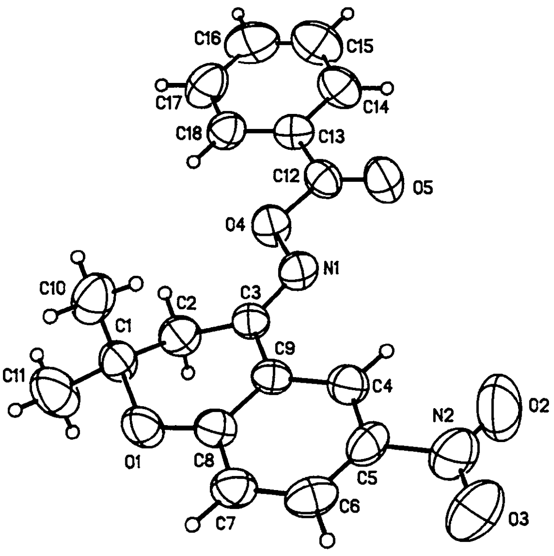


Fig. 1. The molecular structure and numbering scheme for compound (I). Displacement ellipsoids are plotted at the 50% probability level.

The benzoyloxime group is almost planar [maximum deviation 0.080 (4) Å]. In contrast to the molecular conformation of (II) (Cassidy *et al.*, 1989), in which the substituent pyrrolidone ring is orthogonal to the benzopyran nucleus, the benzoyloxime plane of (I) is nearly parallel to the benzopyran nucleus, with a dihedral angle of 8.5 (5)°. The nitro group is also approximately coplanar with the benzopyran nucleus, with a dihedral angle of 4.2 (5)°. The benzoyl carbonyl group and the benzene ring of benzopyran both adopt a *cis* configuration. The bond lengths of compound (I) are unexceptional.

Experimental

Acetone (40 ml, 685 mmol) was added to a benzene solution of 3-acetyl-4-hydroxynitrobenzene (15 g, 86 mmol), followed by the dropwise addition of pyrrolidene (3.7 ml, 43 mmol). The solution was stirred at room temperature for 20 h and the intermediate product was recrystallized from isopropyl ether as a pale-yellow solid (m.p. 374–376 K). A mixture of this product (10 g, 45 mmol) and hydroxylamine hydrochloride (15 g, 210 mmol) in dry methanol (150 ml) was refluxed for 10 h and the solvent removed *in vacuo*. The residue was washed with water and recrystallized from benzene to give a white solid (m.p. 434–436 K). This product (0.47 g, 2 mmol) was treated in dry pyridine (10 ml) with benzoyl chloride (1.12 g, 8 mmol) at 273 K and the mixture kept at room temperature for 10 h before treatment with excess dilute hydrochloric acid. The filtered product, (I), was recrystallized from absolute ethanol (m.p. 429–431 K). Satisfactory analyses (C, H, N) were obtained.

Crystal data

$C_{18}H_{16}N_2O_5$
$M_r = 340.3$
Triclinic
$P\bar{1}$
$a = 7.0320(10)$ Å
$b = 10.667(2)$ Å
$c = 11.879(2)$ Å
$\alpha = 102.40(3)°$
$\beta = 102.92(3)°$
$\gamma = 95.16(3)°$
$V = 839.3(2)$ Å^3
$Z = 2$
$D_x = 1.347$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4.05–9.87°$
$\mu = 0.093$ mm^{-1}
$T = 293$ K
Section cut from needle
0.40 × 0.30 × 0.20 mm
Dark yellow

Data collection

Rigaku AFC-6*S* diffractometer
$2\theta/\theta$ scans
Absorption correction: none
2827 measured reflections
2827 independent reflections
1364 observed reflections [$F > 4\sigma(F)$]

$\theta_{max} = 25°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 10$
$l = -14 \rightarrow 13$
3 standard reflections monitored every 150 reflections intensity decay: 0.9%

Refinement

Refinement on F
$R = 0.0486$
$wR = 0.0556$
$S = 1.60$
1364 reflections
226 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{\max} = 0.007$
$\Delta\rho_{\max} = 0.160$ e Å^{-3}
$\Delta\rho_{\min} = -0.230$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.4307 (6)	0.7354 (3)	0.7698 (3)	0.054 (2)
C2	0.2578 (6)	0.6907 (3)	0.6608 (3)	0.059 (2)
C3	0.2778 (5)	0.5633 (3)	0.5850 (3)	0.045 (2)
C4	0.3237 (5)	0.3358 (3)	0.5972 (3)	0.052 (2)
C5	0.3882 (6)	0.2513 (3)	0.6651 (4)	0.055 (2)
C6	0.4775 (6)	0.2916 (4)	0.7842 (4)	0.059 (2)
C7	0.5025 (6)	0.4204 (4)	0.8386 (3)	0.056 (2)
C8	0.4356 (5)	0.5089 (4)	0.7733 (3)	0.049 (2)
C9	0.3485 (5)	0.4678 (3)	0.6516 (3)	0.042 (2)
C10	0.6193 (6)	0.7679 (4)	0.7365 (4)	0.075 (2)
C11	0.3884 (8)	0.8456 (4)	0.8597 (4)	0.096 (2)
C12	0.1011 (5)	0.5796 (4)	0.2959 (3)	0.051 (2)
C13	0.0532 (5)	0.6868 (4)	0.2399 (3)	0.050 (2)
C14	−0.0258 (6)	0.6526 (4)	0.1182 (3)	0.067 (2)
C15	−0.0701 (7)	0.7458 (6)	0.0568 (4)	0.090 (3)
C16	−0.0386 (7)	0.8731 (6)	0.1169 (5)	0.091 (3)
C17	0.0376 (6)	0.9088 (4)	0.2380 (4)	0.078 (2)
C18	0.0838 (5)	0.8147 (4)	0.2995 (3)	0.058 (2)
O1	0.4583 (4)	0.6332 (2)	0.8352 (2)	0.061 (1)
O2	0.2716 (7)	0.0795 (3)	0.5016 (3)	0.138 (2)
O3	0.4050 (5)	0.0370 (3)	0.6661 (3)	0.109 (2)
O4	0.1695 (3)	0.6249 (2)	0.4180 (2)	0.054 (1)
O5	0.0849 (4)	0.4682 (3)	0.2460 (2)	0.068 (1)
N1	0.2352 (4)	0.5235 (3)	0.4715 (3)	0.050 (1)
N2	0.3518 (6)	0.1115 (3)	0.6054 (4)	0.082 (2)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.519 (5)	C8—O1	1.348 (4)
C1—O1	1.471 (5)	C12—C13	1.470 (6)
C2—C3	1.498 (5)	C12—O4	1.384 (4)
C3—C9	1.474 (6)	C12—O5	1.191 (5)
C3—N1	1.279 (5)	O4—N1	1.429 (4)
C8—C9	1.393 (5)		
C2—C1—O1	110.7 (3)	C13—C12—O4	110.6 (3)
C1—C2—C3	111.3 (3)	C13—C12—O5	126.3 (3)
C2—C3—C9	114.7 (3)	O4—C12—O5	123.1 (4)
C2—C3—N1	129.7 (4)	C1—O1—C8	119.0 (3)
C9—C3—N1	115.5 (3)	C12—O4—N1	110.8 (3)
C9—C8—O1	123.2 (4)	C3—N1—O4	109.8 (3)
C3—C9—C8	119.5 (3)		

H atoms were introduced in calculated positions and given fixed isotropic U values.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Octyloxybiphenyl-4′-yl 4-Methoxybenzoate

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Abstract

The title compound, $C_{28}H_{32}O_4$, has the phase sequence crystal–nematic–isotropic liquid. The crystal has an 'imbricated' structure, *i.e.* a half-and-half overlapping of molecules. Molecular long axes are oriented in one direction, resulting in a polar structure.

Comment

Liquid crystalline behaviour is influenced by molecular structure, such as the direction of polar groups, as well as the kind of polar groups. For example, the title

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