

## 6,6'-Dichloro-3,3'-(butane-1,4-diylidioxy)-diflavone

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## Key indicators

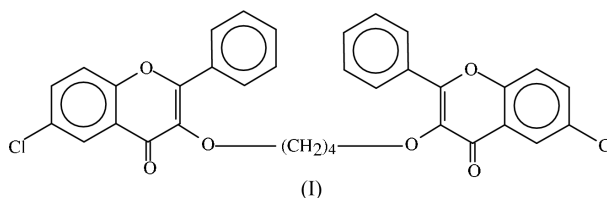
Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.011 \text{ \AA}$   
R factor = 0.056  
wR factor = 0.159  
Data-to-parameter ratio = 10.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Molecules of the title compound,  $\text{C}_{34}\text{H}_{24}\text{Cl}_2\text{O}_6$ , lie across crystallographic inversion centres. The flavone moiety is slightly folded, with the benzene and dihydropyrone rings making a dihedral angle of  $2.5 (2)^\circ$ . In the crystal, the molecules exist as cyclic intermolecular  $\text{C}-\text{H} \cdots \text{Cl}$  hydrogen-bonded dimers. The structure is further stabilized by van der Waals interactions.

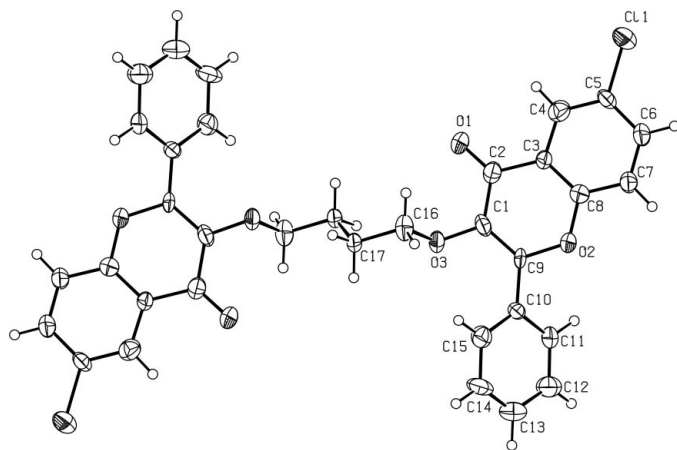
## Comment

Benzopyran derivatives are anti-inflammatory and anti-allergic, and are used to treat elephantiasis. These compounds are capable of inducing alteration in DNA structure and replication. Flavonoids exhibit antidiabetic (Hii & Howell, 1985; Basnet *et al.*, 1993; Ragunathan & Sulochana, 1994) and aldose reductase inhibitory activities (Varma & Kinoshita, 1976; Okuda *et al.*, 1984; Aida *et al.*, 1990). Chromanone compounds are used to treat angina pectoris (Hasegaida, 1967) and these compounds show vasodilating activity on the coronary vascular bed (Nagao *et al.*, 1972). Diethyl 2,6-dimethyl-4-(2-phenyl-4-oxo-4*H*-1-benzopyran-6-yl)-1,4-dihydropyridine-3,5-dicarboxylate, containing a 2-phenyl-4*H*-benzopyran-4-one (flavone), is known to have a coronary dilatory effect (Itz & Potzsch, 1963) and capillary resistance activity (Gabor, 1981).



The asymmetric unit of the title compound, (I), consists of one-half of the molecule, with the other half generated by a crystallographic inversion centre at  $(1-x, 1-y, 1-z)$ . The geometry of the benzopyran moiety is comparable with those observed in related structures (Thinagar *et al.*, 2000; Bruno *et al.*, 2001; Özbey *et al.*, 1999). The bond angles around C2 of the heterocyclic ring vary from  $113.1 (7)$  to  $123.6 (7)^\circ$ , indicating significant deviation from the ideal value. The  $\text{O2}-\text{C9}-\text{C10}-\text{C15}$  torsion angle [ $141.8 (7)^\circ$ ] shows a +anticlinal conformation of the phenyl ring with respect to the flavone moiety. The  $\text{O3}-\text{C16}$  bond is in a *trans* orientation with respect to the flavone moiety, as is evidenced by the  $\text{C1}-\text{O3}-\text{C16}-\text{C17}$  torsion angle of  $172.4 (6)^\circ$ . The pyran ring is planar within  $0.032 (8) \text{ \AA}$ , compared to the usual half-chair conformation (Alex *et al.*, 1993). However, the flavone moiety is not strictly planar, as the benzene and dihydropyrone rings form a dihedral angle of  $2.5 (2)^\circ$ . The O atom of the keto group

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**Figure 1**  
A view of the title molecule, showing 40% probability displacement ellipsoids and the atom-numbering scheme. Only the contents of the asymmetric unit are numbered.

deviates from the flavone moiety by  $-0.084$  (5). The dihedral angle between the planes of the flavone moiety and the phenyl ring is  $42.5$  (3) $^\circ$ .

The molecular structure of (I) is stabilized by intramolecular C—H $\cdots$ O hydrogen bonds and the crystal packing is stabilized by C—H $\cdots$ Cl intermolecular hydrogen bonds (Table 2). The intramolecular C16—H16A $\cdots$ O1 hydrogen bond is involved in  $R_1^1(5)$  ring formation. In the crystal, an intermolecular C4—H4 $\cdots$ Cl( $-x, 1 - y, -z$ ) hydrogen bond is involved in cyclic dimer formation, with an  $R_2^2(8)$  ring descriptor (Bernstein *et al.*, 1995).

## Experimental

A suspension of 6-chloro-3-hydroxy-2-(2-furyl)-4-oxo-4H-1-benzopyran (1 g, 0.0038 mol), 1,4-dibromobutane (0.0019 mol), tetra-*n*-butylammonium iodide (1 g) and freshly ignited  $K_2CO_3$  (1 g) was refluxed in dry acetone (30 ml) for 4 h. The colour of the reaction mixture changed from yellow to colourless. The reaction mixture, after filtration and distillation of acetone, was poured into cold water to obtain a mixture of the monochromone and bischromone. This was then crystallized from acetone to obtain pure bischromone.

### Crystal data

$C_{34}H_{24}Cl_2O_6$   
 $M_r = 599.43$   
Triclinic,  $P\bar{1}$   
 $a = 5.335$  (9)  $\text{\AA}$   
 $b = 9.242$  (8)  $\text{\AA}$   
 $c = 13.969$  (9)  $\text{\AA}$   
 $\alpha = 89.27$  (1) $^\circ$   
 $\beta = 82.68$  (1) $^\circ$   
 $\gamma = 86.47$  (1) $^\circ$   
 $V = 681.8$  (14)  $\text{\AA}^3$

$Z = 1$   
 $D_x = 1.460$   $\text{Mg m}^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 25 reflections  
 $\theta = 2\text{--}22^\circ$   
 $\mu = 0.29$   $\text{mm}^{-1}$   
 $T = 293$  (2) K  
Block, colourless  
 $0.30 \times 0.29 \times 0.27$  mm

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $w/2\theta$  scans  
2312 measured reflections  
2056 independent reflections  
685 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.122$

$\theta_{\text{max}} = 23.7^\circ$   
 $h = -6 \rightarrow 0$   
 $k = -10 \rightarrow 10$   
 $l = -15 \rightarrow 15$   
3 standard reflections every 100 reflections  
intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.159$   
 $S = 0.91$   
2056 reflections  
191 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.29$   $\text{e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.27$   $\text{e \AA}^{-3}$   
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.018 (4)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C2	1.220 (7)	O3—C16	1.470 (7)
O2—C8	1.378 (7)	C16—C17	1.484 (8)
O2—C9	1.388 (7)	C17—C17 <sup>i</sup>	1.541 (13)
O3—C1	1.358 (7)		
O1—C2—C3	123.6 (7)	O2—C8—C7	116.3 (7)
O1—C2—C1	123.2 (7)	C1—C9—C10	127.5 (6)
C3—C2—C1	113.1 (7)	O2—C9—C10	111.9 (6)
C8—C3—C4	116.6 (7)	C15—C10—C11	117.0 (7)
C8—C3—C2	122.4 (7)	C15—C10—C9	122.6 (7)
C3—C8—C7	123.1 (7)		
C16—O3—C1—C2	$-69.2$ (8)	C1—O3—C16—C17	172.4 (6)
C1—C9—C10—C15	$-45.6$ (12)	O3—C16—C17—C17 <sup>i</sup>	$-61.0$ (10)
O2—C9—C10—C11	$-36.3$ (10)		

Symmetry code: (i)  $1 - x, 1 - y, 1 - z$ .

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C16—H16A $\cdots$ O1	0.97	2.51	3.091 (10)	118
C4—H4 $\cdots$ Cl1 <sup>ii</sup>	0.93	2.76	3.688 (10)	174

Symmetry code: (ii)  $-x, 1 - y, -z$ .

All H atoms were fixed geometrically and allowed to ride on the corresponding C atoms, with C—H distances of 0.93 or 0.97  $\text{\AA}$ , and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The large  $R_{\text{int}}$  value (0.122) and low ratio of observed to unique reflections (0.33), are a result of the poor diffraction quality of the crystal. The intensity data collection was restricted to  $\theta_{\text{max}}$  of  $23.7^\circ$ , as the reflections were found to be rather weak at higher angles.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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