

5-Methoxy-4-(4-methoxyphenyl)-  
isochroman-3-ol

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

$R$  factor = 0.038

$wR$  factor = 0.114

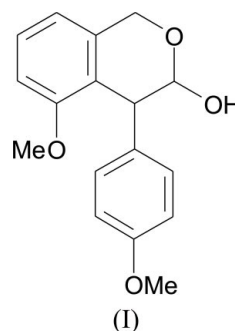
Data-to-parameter ratio = 10.5

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

In the title compound,  $\text{C}_{17}\text{H}_{18}\text{O}_4$ , the hydroxyl and methoxyphenyl substituents are in axial positions. The heterocyclic ring adopts a half-chair conformation. The molecules are linked by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, leading to dimerization.

## Comment

As a result of systematic studies on new precursors for obtaining benzo[*c*]pyran antibiotics (Moore, 1977; Moore & Czerniak, 1981), we have focused our attention on isochroman derivatives. In this paper, we present X-ray crystallographic analysis of the title compound, (I), as a continuation of our previous studies (Palusiak *et al.*, 2002*a,b*).

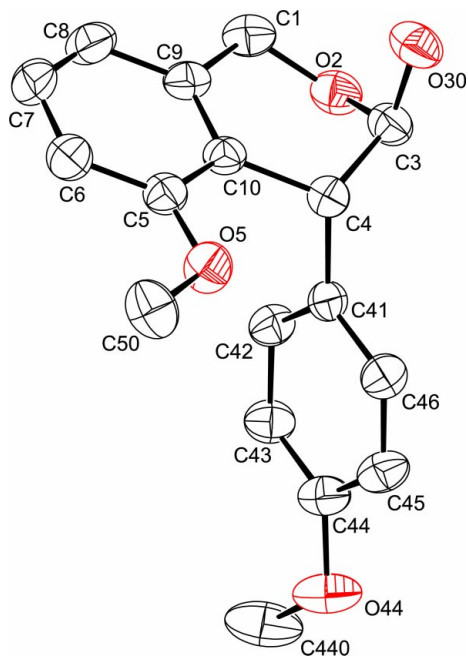


In (I), the isochroman moiety is substituted with a hydroxyl group in position 3, a 4-methoxyphenyl substituent in position 4 and a methoxy group in position 5. The heterocyclic ring has a half-chair conformation, with the twofold axis passing through the midpoint of the  $\text{O2}-\text{C3}$  bond. The asymmetry parameter (Nardelli, 1983)  $\Delta_2(\text{O2}-\text{C3})$  is 0.0209 (7). The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence  $\text{C1}-\text{O2}-\text{C3}-\text{C4}-\text{C10}-\text{C9}$  are  $Q = 0.472$  (2)  $\text{Å}$ ,  $\varphi_2 = 96.4$  (4) $^\circ$  and  $\theta_2 = 49.2$  (2) $^\circ$ . The substituents in positions 3 and 4 of the heterocyclic ring are in an axial conformation with respect to this ring. The torsion angles describing these orientations, *viz.*  $\text{O30}-\text{C3}-\text{C4}-\text{C10}$  and  $\text{C41}-\text{C4}-\text{C10}-\text{C9}$ , are presented in Table 1. The benzene ring of the isochroman system and the phenyl ring are nearly perpendicular, forming a dihedral angle of 86.51 (6) $^\circ$ . The atoms of the methoxy groups do not deviate significantly from the planes of their carrier rings. The maximum deviation of 0.156 (3)  $\text{Å}$  is observed for atom C440. Atom O30 of the hydroxyl group acts as a hydrogen-bond donor to O2 of an adjacent molecule (Table 2). Moreover, hydrogen bonding leads to dimerization (Fig. 2). As a result, an eight-membered ring is formed between molecules, whose topological motif corresponds to the first level graph-set descriptor  $R_2^2(8)$  (Bernstein *et al.*, 1995).

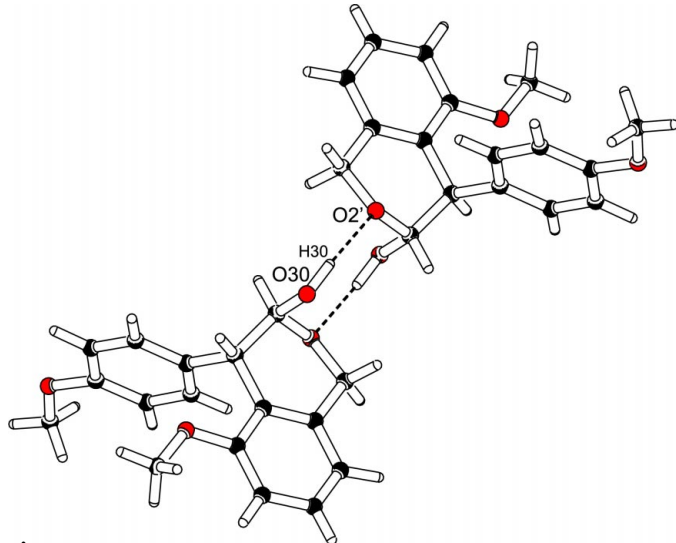
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**Figure 1**  
View of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.



**Figure 2**  
Intermolecular hydrogen bonding in the crystal structure of (I).

## Experimental

The synthesis of (I) has been described elsewhere (Epszajn *et al.*, 2001). Crystals were obtained from ethanol, by slow evaporation at room temperature.

### Crystal data

$C_{17}H_{18}O_4$   
 $M_r = 286.31$   
Triclinic,  $P\bar{1}$   
 $a = 5.881$  (2) Å  
 $b = 8.182$  (2) Å  
 $c = 15.983$  (3) Å  
 $\alpha = 78.03$  (2)°  
 $\beta = 89.28$  (2)°  
 $\gamma = 72.49$  (2)°  
 $V = 716.4$  (3) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.327$  Mg m<sup>-3</sup>  
Cu  $K\alpha$  radiation  
Cell parameters from 25 reflections  
 $\theta = 38.2$ – $39.9$ °  
 $\mu = 0.77$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Plate, colourless  
 $0.50 \times 0.40 \times 0.10$  mm

### Data collection

Rigaku AFC-5S diffractometer  
 $\omega$  scans  
Absorption correction: analytical  
(de Meulenaer & Tompa, 1965)  
 $T_{\min} = 0.775$ ,  $T_{\max} = 0.925$   
2695 measured reflections  
2505 independent reflections  
1974 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 67.5$ °  
 $h = -6 \rightarrow 7$   
 $k = -9 \rightarrow 8$   
 $l = -19 \rightarrow 18$   
3 standard reflections  
every 150 reflections  
intensity decay: <2%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.114$   
 $S = 1.12$   
2505 reflections  
239 parameters  
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.0312P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.14$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>  
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.0072 (12)

**Table 1**

Selected geometric parameters (Å, °).

O5—C5	1.364 (2)	O2—C3	1.429 (2)
O5—C50	1.426 (2)	O2—C1	1.434 (2)
O44—C44	1.368 (2)	O30—C3	1.389 (2)
O44—C440	1.416 (3)		
C5—O5—C50	118.0 (2)	O2—C1—C9	113.6 (2)
C44—O44—C440	118.1 (2)	O30—C3—O2	111.8 (2)
C3—O2—C1	113.0 (2)	O30—C3—C4	107.7 (2)
O44—C44—C43	125.5 (2)	O2—C3—C4	111.0 (2)
O44—C44—C45	115.3 (2)		
C50—O5—C5—C6	-0.8 (2)	C440—O44—C44—C43	-5.1 (3)
C41—C4—C10—C9	-108.2 (2)	C10—C4—C3—O30	74.4 (2)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O30—H30 $\cdots$ O2 <sup>i</sup>	0.97 (4)	1.96 (4)	2.917 (2)	170 (3)

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

The methyl H atoms were constrained to their parent C atom using a riding model. The positions of the others H atoms were found and refined isotropically; C—H distances are in the range 0.935 (18)–1.01 (2) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998); software used to prepare material for publication: *PARST97* (Nardelli, 1996).

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