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(3*S*,4*R*)-4-(2,5-Dimethoxyphenyl)-8-methoxyisochroman-3-ol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.109
Data-to-parameter ratio = 14.1

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

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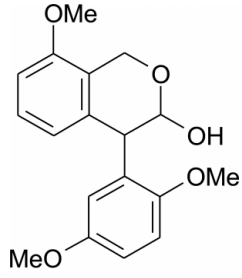
In the title compound, $C_{18}H_{20}O_5$, the hydroxyl and dimethoxyphenyl substituents are in axial positions. The heterocyclic ring is in a half-chair conformation. The molecules are linked by O—H···O hydrogen bonds, leading to the formation of a chain extended throughout the whole of the crystal.

Comment

Some members of the benzo[*c*]pyran family have been found in nature and have been shown to possess a variety of biological properties (Moore, 1977; Moore & Czerniak, 1981). The title isochroman, (I), has been synthesized as part of a research project on new precursors for obtaining this type of antibiotic (Epsztajn *et al.*, 2001). X-ray investigations were made in order to confirm the configurations of the chiral atoms C3 (*S*) and C4 (*R*), and to define the positions of the substituents attached to these atoms.



(I)

The molecule of the title compound consists of two condensed rings, phenyl and heterocyclic, with atom O2 in position 2 of the latter. There is a hydroxyl group in position 3, a 2,5-dimethoxyphenyl substituent in position 4 and a methoxy group in position 8. The heterocyclic ring has a half-chair conformation, with the twofold axis passing through the midpoint of the O2—C3 bond. The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence C1—O2—C3—C4—C10—C9 are Q = 0.477 (2) Å, φ_2 = −77.6 (4)° and q_2 = 132.6 (3)°, and the asymmetry parameter (Nardelli, 1983) Δ_2(O2—C3) is 0.0413 (8). The substituents in positions 3 and 4 of the heterocyclic ring are attached axially, with torsion angles O3—C3—C4—C10 and C41—C4—C10—C9 of −70.7 (2) and 101.8 (2)°, respectively. The phenyl rings are almost planar and form a dihedral angle of 85.09 (6)°. The O atom of the hydroxyl group acts as a hydrogen-bond donor to O2^{i} of an adjacent molecule [symmetry code: (i) −*x*+5/2, −*y*, *z*+1/2; see Table 2]. Finally, the linked molecules form a *C*(4) chain (Fig. 2) (Bernstein *et al.*, 1995).

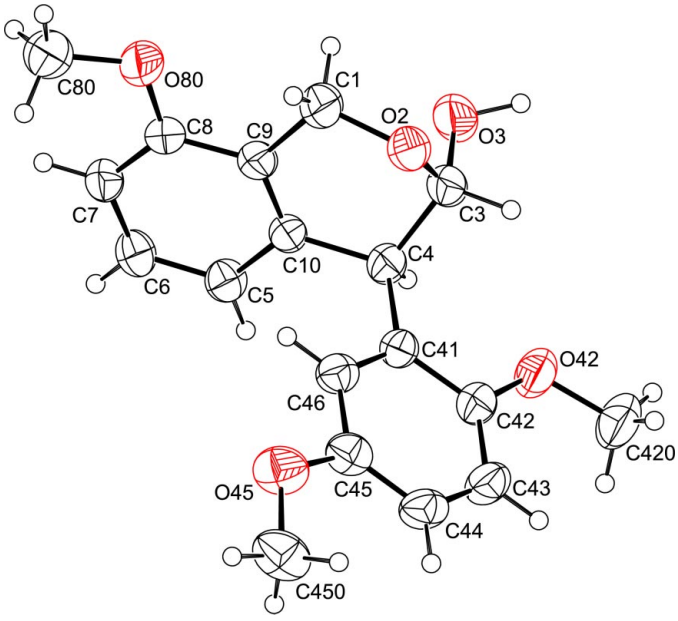


Figure 1
Displacement ellipsoid plot (*PLATON*; Spek, 1998) of title compound, with the atom-labelling scheme. Ellipsoids are drawn at the 40% probability level.

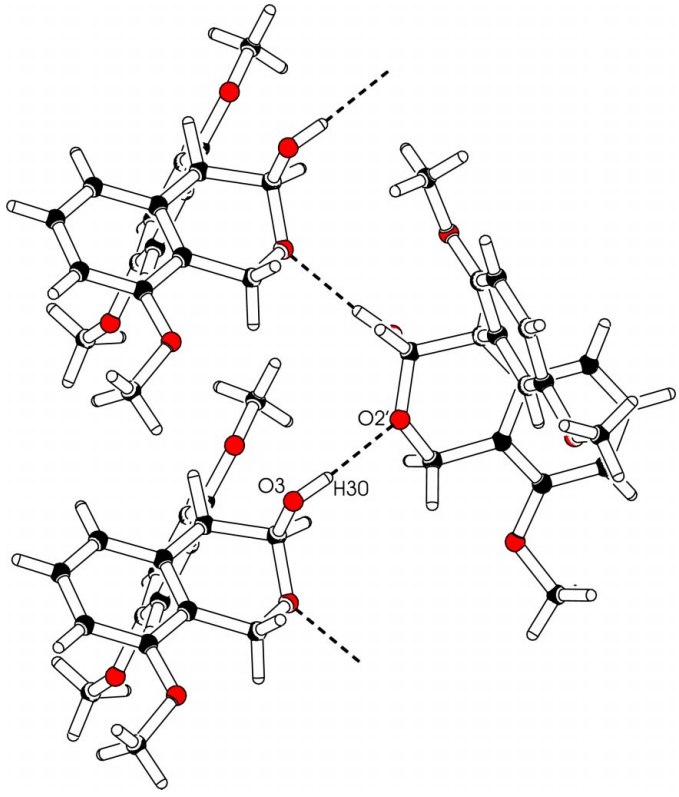


Figure 2
The intermolecular hydrogen bonding in the crystal structure of the title compound.

Experimental

The synthesis of the title compound has been described elsewhere (Epsztajn *et al.*, 2001). Crystals were obtained by slow evaporation from a methanol solution at room temperature.

Crystal data

$C_{18}H_{20}O_5$
M_r = 316.34
Orthorhombic, $P2_12_12_1$
a = 11.920 (1) Å
b = 18.114 (2) Å
c = 7.383 (1) Å
V = 1594.2 (3) Å^3
Z = 4
D_x = 1.318 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 20 reflections
θ = 9.5–12.3°
μ = 0.79 mm^{-1}
T = 293 (2) K
Needle, colourless
0.50 × 0.28 × 0.18 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
T_{min} = 0.778, T_{max} = 0.879
6547 measured reflections
3063 independent reflections
2599 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 72.6°
$h = -14 \rightarrow 14$
$k = -22 \rightarrow 22$
$l = -8 \rightarrow 8$
3 standard reflections
every 150 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.109
S = 1.02
3063 reflections
217 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0726P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0125 (9)
Absolute structure: Flack (1983), 1281 Friedel pairs
Flack parameter = −0.1 (2)

Table 1
Selected geometric parameters (Å, °).

O2—C3	1.426 (3)	O45—C450	1.416 (2)
O2—C1	1.436 (2)	C42—O42	1.367 (2)
O80—C8	1.369 (2)	O3—C3	1.406 (2)
O80—C80	1.420 (3)	O42—C420	1.416 (3)
O45—C45	1.383 (3)		
C3—O2—C1	113.8 (2)	O3—C3—O2	110.7 (2)
C8—O80—C80	117.9 (2)	O3—C3—C4	110.0 (2)
C45—O45—C450	118.2 (2)	O2—C3—C4	109.8 (2)
C42—O42—C420	118.8 (2)		
C9—C10—C4—C41	101.8 (2)	O3—C3—C4—C10	−70.7 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H30···O2^{i}	0.98 (4)	1.90 (4)	2.881 (2)	171 (3)

Symmetry code: (i) $\frac{5}{2} - x, -y, \frac{1}{2} + z$.

Atoms H30 and H3 were refined isotropically. The other H atoms were constrained to ride on their parent C atoms using AFIX in *SHELXL*97 (Sheldrick, 1997).

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:

*SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998); software used to prepare material for publication: *PARST*97 (Nardelli, 1996).

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