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Key indicators

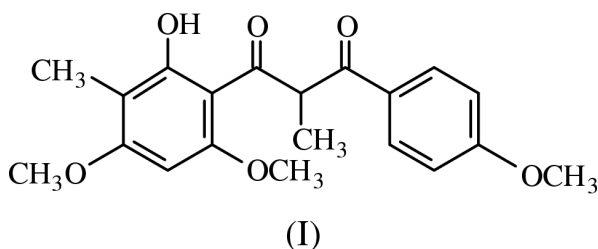
Single-crystal X-ray study
 $T = 183\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
Disorder in main residue
 R factor = 0.049
 wR factor = 0.144
Data-to-parameter ratio = 16.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.1-(2-Hydroxy-4,6-dimethoxy-3-methylphenyl)-
3-(4-methoxyphenyl)-2-methyl-1,3-propanedione

The crystal structure of the title compound, $\text{C}_{20}\text{H}_{22}\text{O}_6$, an intermediate in the synthesis of the flavone 2-(4-methoxyphenyl)-5,7-dimethoxy-3,8-dimethyl-4*H*-1-benzopyran-4-one, has been determined. The molecules are held together by van der Waals forces. There is an intramolecular hydrogen bond between the hydroxy group and the neighbouring keto O atom. The molecules in the crystal structure are disordered, with significantly different orientations of the methyl group in the disordered 4-methoxyphenyl substituent.

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Comment

In an investigation of the selective inhibition of certain rat heart enzymes caused by a series of lipophilic flavones, the inhibition effect of eight flavones, which are derivatives of either *O*-methyluteolin or *O*-methylapigenin, was studied (Gaillard *et al.*, 1996). The crystal structure of one of the *O*-methyluteolin derivatives, 2-(3,4-dimethoxyphenyl)-5,7-dimethoxy-3,8-dimethyl-4*H*-1-benzopyran-4-one, has recently been determined by Stomberg *et al.* (2002). A second one of these flavones, the *O*-methylapigenin derivative 2-(4-methoxyphenyl)-5,7-dimethoxy-3,8-dimethyl-4*H*-1-benzopyran-4-one, was prepared by acid-catalysed cyclization and dehydration of 1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)-3-(4-methoxyphenyl)-2-methyl-1,3-propanedione, (I). This paper describes the crystal structure of this synthetic intermediate, (I).



A perspective view of (I), together with the atom-numbering scheme, is shown in Fig. 1. The disorder in the methoxyphenyl group is shown in Fig. 2, and the packing of the molecules in Fig. 3. The molecules are held together by van der Waals forces. There is an intramolecular hydrogen bond between the hydroxy H atom H(O1) and the O atom O4 in the neighbouring carbonyl group. The benzylic C atom C7, the hydroxy O atom O1, and the methyl carbon atom C10 are twisted out of the C1/C2/C3/C4/C5/C6 ring plane by -0.094 (2), -0.014 (2) and 0.018 (3) \AA , respectively. The methyl C atoms in the methoxy groups are situated close to the respective benzene ring planes [torsion angles: C5—C4—

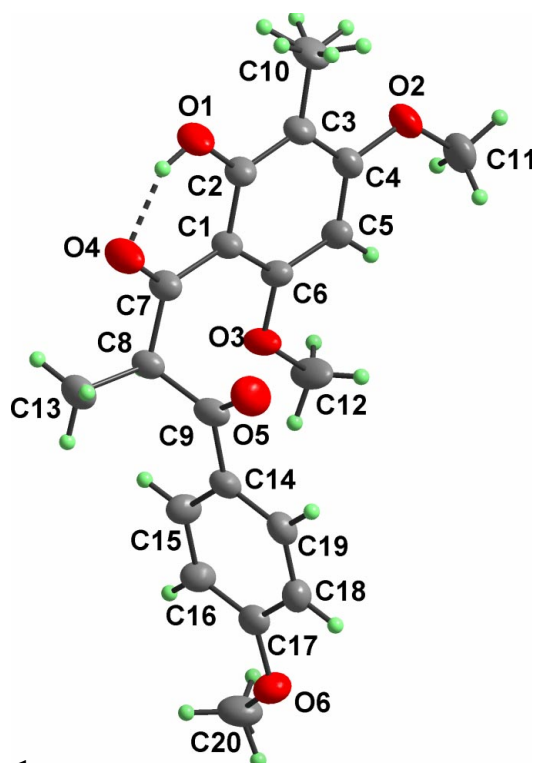


Figure 1

The atom-numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level. The disorder of the methyl group C10 is shown. A dashed line indicates the intramolecular hydrogen bond.

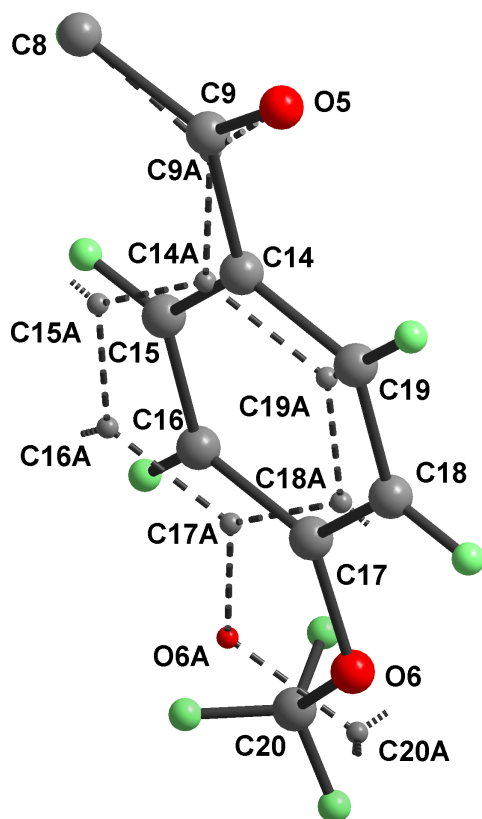


Figure 2

The disorder of the 4-methoxyphenyl group. The minor part, with occupancy 0.194 (3), is shown with smaller radii of atoms and dashed bonds.

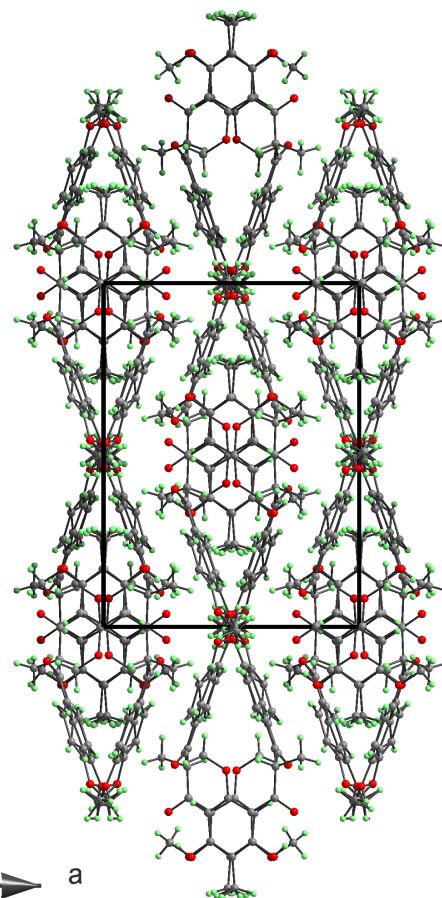


Figure 3

A packing diagram, in projection along the *c* axis.

O2—C11 -4.8 (2)°, C5—C6—O3—C12 1.6 (2)°, C16—C17—O6—C20 11.4 (4)° and C16A—C17A—O6A—C20A 172.6 (10)°].

Experimental

The synthesis of 1-(2-hydroxy-4,6-dimethoxy-3-methylphenyl)-3-(4-methoxyphenyl)-2-methyl-1,3-propanedione, (I), has been described by Gaillard *et al.* (1996), who also published $^1\text{H-NMR}$ data for the substance. Crystals suitable for X-ray analysis were obtained from benzene (m.p. 445–446 K).

Crystal data

$\text{C}_{20}\text{H}_{22}\text{O}_6$
 $M_r = 358.38$
 Monoclinic, $I2/c$
 $a = 12.2304$ (2) Å
 $b = 16.4299$ (2) Å
 $c = 17.8325$ (2) Å
 $\beta = 93.999$ (1)°
 $V = 3574.61$ (8) Å³
 $Z = 8$

$D_x = 1.332$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 8192 reflections
 $\theta = 1.7\text{--}30.6^\circ$
 $\mu = 0.10$ mm⁻¹
 $T = 183$ (2) K
 Needle, colourless
 $0.60 \times 0.12 \times 0.10$ mm

Data collection

Siemens SMART CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.943$, $T_{\max} = 0.990$
 24968 measured reflections

5495 independent reflections
 3717 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$
 $\theta_{\text{max}} = 30.6^\circ$
 $h = -17 \rightarrow 17$
 $k = -23 \rightarrow 23$
 $l = -25 \rightarrow 25$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 1.3054P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.144$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 0.99$	$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$
5495 reflections	$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
340 parameters	
H-atom parameters constrained	

Table 1
Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1 \cdots O4$	0.84	1.74	2.4899 (14)	148

Initial refinement of the structure converged with unacceptably high R -values ($R = 0.095$ for observed reflections and $wR^2 = 0.306$ for all reflections), and there were seven residual peaks in the electron density difference map larger than $0.42 \text{ e } \text{\AA}^{-3}$ (maximum $1.64 \text{ e } \text{\AA}^{-3}$). These peaks appeared close to the 4-methoxyphenyl group and indicated disorder. Six of the seven largest peaks formed a planar ring and they were taken to be the phenyl C atoms, while the largest residual peak could be attributed to the methoxy O atom. The methoxy C atom was also found (peak height $0.26 \text{ e } \text{\AA}^{-3}$). No other atom, except the benzylic C atom attached to the 4-methoxyphenyl group, could be resolved into two parts. Another kind of disorder was found in the methyl group C10. Refinement with geometrical similarity restraints on disordered atoms led to occupancies of 0.806:0.194(3).

H atoms were refined isotropically and were constrained to the ideal geometry, using an appropriate riding model. For the hydroxyl

group, the O—H distance (0.84 \AA) and C—O—H angle (109.5°) were kept fixed, and the torsion angle was chosen to match the observed electron density. For methyl groups, the C—H distances (0.96 \AA) and C—C—H angles (109.5°) were kept fixed, while the torsion angles were allowed to refine, with the starting position based on the threefold averaged circular Fourier synthesis. For methyl group C10, an idealized disordered methyl group was assumed, with two positions rotated from each other by 60° and with a half-occupancy for H atoms.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SADABS* (Sheldrick, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXTL*.

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