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Disordered 6-hydroxyflavanone

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.041
wR factor = 0.095
Data-to-parameter ratio = 12.4

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

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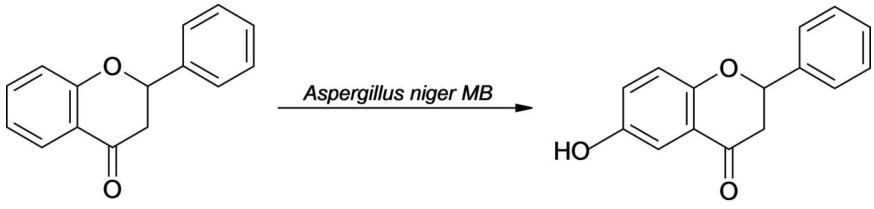
In contrast to the previously reported crystal structure of 6-hydroxyflavanone [Shoja *et al.* (1998). *Z. Kristallogr. New Cryst. Struct.* **213**, 373–374], in the title crystal structure, $C_{15}H_{12}O_3$, both *S* and *R* enantiomers appear to occupy randomly the four crystallographic sites of the unit cell in an approximate 3:1/1:3 ratio.

Comment

Shoja *et al.* (1998) described a fully ordered crystal structure of 6-hydroxyflavanone [space group $P2_1/c$, a = 5.311 (7), b = 21.747 (3), c = 10.122 (2) Å, β = 92.48 (4)°, T = 295 K]. Similar cell dimensions have been determined for the structure presented in the current paper. However, the present structure of 6-hydroxyflavanone (I) displays both enantiomers somewhat randomly, not systematically, arranged in the unit cell.



(I)

There are known crystal structures of various flavanone derivatives in which enantiomers occupy equivalent sites in a random way. This type of disorder appears in the crystal structures of 5-hydroxy-7,4′-dimethoxyflavanone (Miles *et al.*, 1989), 7-hydroxy-4′-methoxyflavanone (Kendi *et al.*, 1995), and naringenin (Cox *et al.*, 2003). Cox *et al.* (2003) suggest that the integrity of the crystal structures is maintained by the close overlap of equivalent atom positions in the two enantiomers, which can easily substitute for each other.

The molecular structure of (I), together with the numbering scheme employed, is presented in Fig. 1. In the present analysis, atoms at position 2 in the pyrone ring [C2 and H2 (major component) and C2*A* and H2*A* (minor component)] and phenyl ring [C1′–C6′ (major component) and C1*A*′–C6*A*′ (minor component)] are clearly resolved. Thus, two enantiomers occupy equivalent sites in the unit cell, but not in a systematic way. The ratio of the two enantiomers (*R*:*S*) in the asymmetric unit is 0.75:0.25, which gives a 3:1/1:3 ratio in the crystal structure overall. The disorder does not appear to have a significant influence on the hydrogen-bonding motif (Table 1), which is similar to that observed in the previously reported crystal structure of 6-hydroxyflavanone (Shoja *et al.*, 1998).

Experimental

The title compound was obtained during microbiological transformation of flavanone (see scheme in *Comment*) using *Aspergillus niger MB* race. Crystals of 6-hydroxyflavanone were grown from a hexane–acetone (10/1) solution under ambient conditions.

Crystal data

$C_{15}H_{12}O_3$
M_r = 240.25
Monoclinic, $P2_1/c$
a = 5.251 (2) Å
b = 21.809 (4) Å
c = 10.025 (3) Å
β = 93.34 (3)°
V = 1146.1 (6) Å^3
Z = 4
D_x = 1.392 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
Block, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Kuma KM-4-CCD diffractometer
ω scans
Absorption correction: none
7454 measured reflections
2233 independent reflections
1456 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.095
S = 0.91
2233 reflections
180 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3H···O2^{i}	0.96	1.80	2.7553 (17)	173

Symmetry code: (i) $x+1, -y+\frac{3}{2}, z-\frac{1}{2}$.

Non-H atoms with occupancy factors 0.75 or 1 were refined with anisotropic displacement parameters. The occupancy factors for C2/C1′–C6′ and C2*A*/C1*A*′–C6*A*′ were initially refined and subsequently fixed at 0.75 (for C2 and C1′–C6′) and 0.25 (for C2*A* and C1*A*′–C6*A*′). The geometry of the minor component of the phenyl ring was fixed as a regular hexagon with C—C = 1.39 Å. All H atoms were included in idealized positions and were refined as riding, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

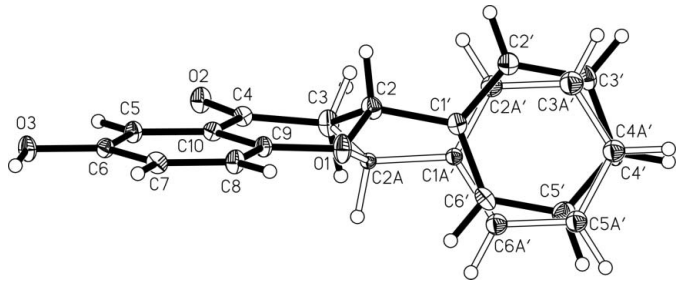


Figure 1
The molecular structure of the overlapping enantiomers of (I). The atoms of the *S* enantiomer are joined by open bonds. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2001); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Bruker, 1999); software used to prepare material for publication: *SHELXL97*.

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