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Disordered 4′-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 = 0.050
wR factor = 0.089
Data-to-parameter ratio = 18.2

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

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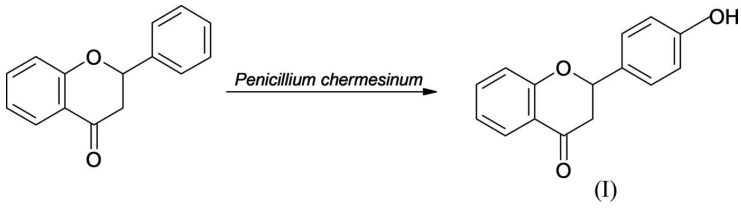
Unlike the crystals of 4′-hydroxyflavanone, $C_{15}H_{12}O_3$ obtained from a methanol solution [Shoja (1998). *Z. Kristallogr. New Cryst. Struct.* **213**, 85–86], in crystals grown from a hexane–acetone mixture (10:1), both the *S* and *R* enantiomers appear to occupy in a random way the four symmetry-equivalent sites of the unit cell in an approximate 4:1/1:4 ratio.

Comment

Shoja (1998) has described the fully ordered crystal structure of 4′-hydroxyflavanone [space group $P2_1/c$, a = 5.307 (2), b = 15.520 (20), c = 14.827 (6)Å and β = 102.35 (2)°, T = 295 K], the crystals being obtained from a methanol solution. Here we report the crystallization from a hexane–acetone (10:1) solution, leading to the formation of the crystal structure of the title compound (I), in which both enantiomers occupy symmetry-equivalent sites randomly.



(I)

Accomodation of enantiomers at the same sites is not systematical and is known for various flavanone derivatives, *e.g.* for 5-hydroxy-7,4′-dimethoxyflavanone (Miles *et al.*, 1989) or for 7-hydroxy-4′-methoxyflavanone (Kendi *et al.*, 1995). For naringenin, both ordered (Spek, 1988) and disordered (Cox *et al.*, 2003) crystal structures have been reported. Cox *et al.* (2003) suggest that the integrity of the disordered crystal structure is maintained by the close overlap of equivalent atom positions in the two enantiomers, which can easily substitute for each other.

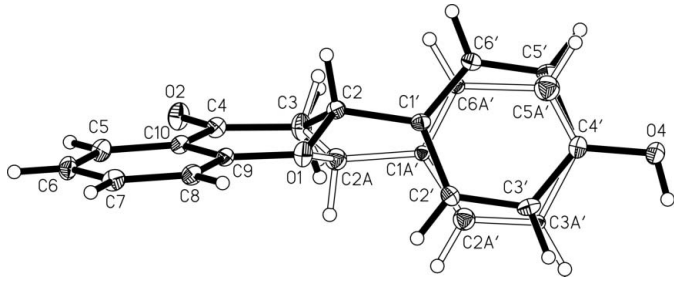


Figure 1
The molecular structure of the overlapping enantiomers of (I). The atoms of the *S* enantiomer are joined by open bonds. Displacement ellipsoids are drawn at the 30% probability level.

The molecular structure of (I), together with the numbering scheme employed, is presented in Fig. 1. Atoms at position 2 of the pyrone ring and the atoms of the benzene ring are disordered over two sites (C2 and C1′, C2′, C3′, C5′, C6′ with 0.80 occupancy and C2*A* and C1*A*′, C2*A*′, C3*A*′, C5*A*′, C6*A*′ with 0.20 occupancy). The disorder results from the two enantiomers occupying equivalent sites in the unit cell, but not in a systematic way. However, the hydrogen-bonding (Table 1) motif is similar to that observed in the previously reported ordered crystal structure of 4′-hydroxyflavanone (Shoja, 1998).

Experimental

The title compound was obtained during microbiological transformation of flavanone (see Scheme) using *Penicillium chermesinum* race. Crystals of 4′-hydroxyflavanone were grown from a hexane–acetone (10:1) solution of (I) under ambient conditions.

Crystal data

$C_{15}H_{12}O_3$
M_r = 240.25
Monoclinic, $P2_1/c$
a = 5.247 (2) Å
b = 15.374 (3) Å
c = 14.870 (5) Å
β = 102.62 (3)°
V = 1170.5 (7) Å^3
Z = 4
D_x = 1.363 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
Needle, colourless
0.35 × 0.15 × 0.15 mm

Data collection

Kuma KM-4-CCD diffractometer
ω scans
Absorption correction: none
17651 measured reflections
3396 independent reflections
1923 reflections with $I > 2\sigma(I)$
R_{int} = 0.084
θ_{max} = 30.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.089
S = 0.98
3396 reflections
187 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0283P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2^{i}	0.84	1.87	2.7043 (16)	175

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

The occupancy factors for C2/C1′/C2′/C3′/C5′/C6′ and C2*A*/C1*A*′/C2*A*′/C3*A*′/C5*A*′/C6*A*′ were intially refined but finally fixed at 0.80 and 0.20, respectively. Non-H atoms with occupancy factors equal to 0.20 were refined with isotropic displacement parameters. Non-H atoms with occupancy factors 0.8 were refined with anisotropic displacement parameters. All H atoms were included at idealized positions and were treated as riding atoms, with C—H distances of 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

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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