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(*S*)-4-(4-Hydroxyphenyl)-2,2,4-trimethylchroman

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Key indicators

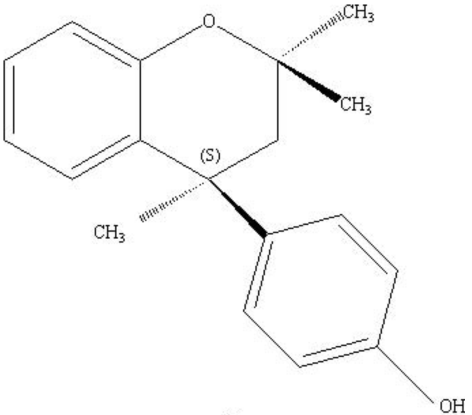
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.100
Data-to-parameter ratio = 10.2

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

The enantiomerically pure title compound, $C_{18}H_{20}O_2$, crystallizes with one molecule in the asymmetric unit. The packing arrangement is significantly different from that of its racemate. Linear hydrogen-bonded chains are aligned along the [100] direction. Edge-to-face π–π interactions occur between adjacent hydrogen-bonded columns.

Comment

As part of our continuing studies of the structural properties of Dianin's compound, we have isolated the *S* isomer, (I) (Fig. 1). As described elsewhere (Esterhuysen *et al.*, 2005), purification of (*S*,*S*)-4-(2,2,4-trimethylchroman-4-yl)phenyl camphanate, (II), and the crystallographic determination of the absolute stereochemistry allowed us to further purify the enantiomerically pure *S*-Dianin's compound by removal of the camphanate group. Although it is well known that the supramolecular packing of enantiomerically pure Dianin's compound does not form clathrate species (Brienne & Jaques, 1975), there has been no description of the structure. Molecules of (I) are connected by O—H···O hydrogen bonds (Table 1), forming infinite chains running along [100]. Two adjacent chains are held together strongly by edge-to-face π–π interactions to form double strands (centroid-to-centroid distances of 5.781 and 5.021 Å) (Fig. 2). Within a double strand, the two individual chains are related by a 2_1 axis. These double strands further pack in a herring-bone-type motif (Fig. 3).



(I)

Experimental

Compound (II) (see *Comment*) (610 mg, 1.36 mmol) and sodium hydroxide (487 mg, 12.2 mmol) were dissolved/suspended in methanol/water (4:1, 10 ml) and stirred at ambient temperature for 24 h. The solvent was removed under reduced pressure and the residue dissolved in water (35 ml). This solution was acidified by

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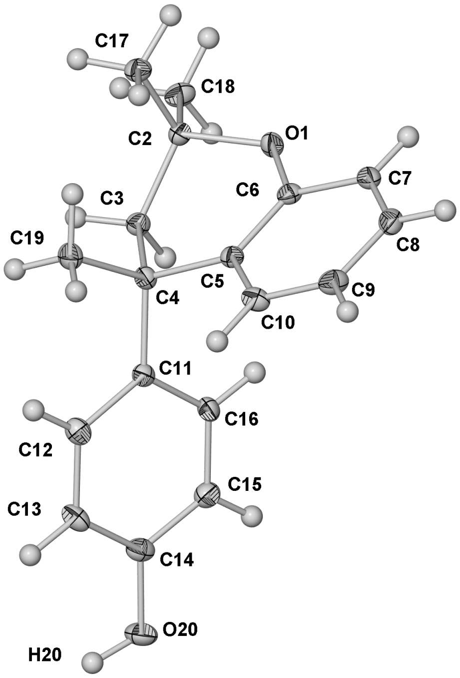

Figure 1
The molecular structure of compound (I), showing atom labels and 50% probability ellipsoids for non-H atoms.

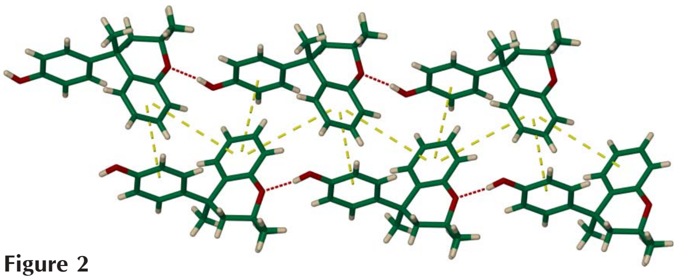

Figure 2
The crystal packing in compound (I) in capped-stick representation, showing the hydrogen-bonded chains. Intermolecular hydrogen bonding is shown as red dashed lines and intermolecular π–π interactions are shown as yellow dashed lines.

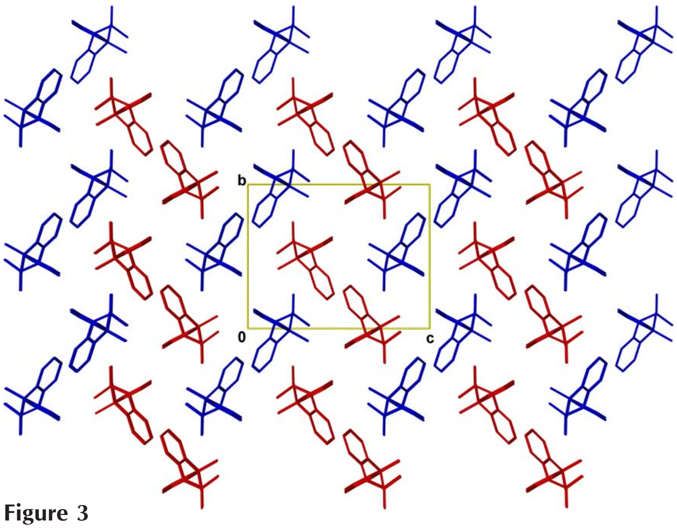

Figure 3
Herring-bone-type packing of the double strands of compound (I). Molecules are shown in capped-stick representation and viewed along [100]. H atoms have been removed for clarity.

adding concentrated HCl dropwise, causing the solution to become turbid. The pH was adjusted to 4.5, after which the turbid mixture was extracted with dichloromethane (2 × 25 ml). The combined extracts were dried over anhydrous $MgSO_4$ and the solvent was then removed under reduced pressure. The colourless oily residue (410 mg) contained traces of crystals. The oily residue was dissolved in methanol for recrystallization. The resulting crystals were found to be the racemic clathrate. The crystals were filtered off and the mother liquors reduced to an oil. The formation and subsequent filtration of the racemic crystals provided an extra step for the purification of the enantiomeric excess of compound (I). The residue (346 mg) yielded crystals from methanol/water (*ca* 9:1). Compound (I) 302 mg (83%), m.p. 411–412 K, $[\alpha]_D^{27.6}$ −61.1° (1.4) (MeOH, $c = 0.95$) [literature value 413 K, $[\alpha]_D^{25}$ −24° (Brienne & Jaques, 1975)].

Crystal data

$C_{18}H_{20}O_2$
$M_r = 268.34$
Orthorhombic, $P2_12_12_1$
$a = 10.029$ (1) Å
$b = 10.476$ (1) Å
$c = 13.2088$ (13) Å
$V = 1387.8$ (2) Å^3
$Z = 4$
$D_x = 1.284$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8768 reflections
$\theta = 2.5$–28.2°
$\mu = 0.08$ mm^{-1}
$T = 100$ (2) K
Plate, colourless
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.980$, $T_{\max} = 0.996$
8768 measured reflections

1885 independent reflections
1758 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\max} = 28.2°$
$h = -13 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.100$
$S = 1.06$
1885 reflections
185 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.2927P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O20—H20···O1$^{\rm i}$	0.93 (3)	2.05 (3)	2.9671 (19)	171 (1)

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

Non-hydroxyl H atoms were positioned geometrically and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2 (1.5 for methyl groups) times $U_{\rm eq}$(C). The hydroxyl H atom was freely refined. In the absence of dispersion effects, Friedel pairs (1325) were merged before the final refinement.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Atwood & Barbour, 2003; Barbour, 2001); software used to prepare material for publication: *X-SEED*.

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