The H atoms of the organic moiety were placed at calculated positions and refined as riding using *SHELXL*97 (Sheldrick, 1997) defaults [C_{aryl}—H = 0.93, N—H = 0.86 Å and $U(H)_{eq} = 1.2U_{eq}$ of the parent atom]. A planar-trigonal geometry was assumed for the N—H bonds. Examination of the crystal structure with *PLATON* (Spek, 1995) showed that there were no solvent-accessible voids in the crystal lattice. All calculations were performed on a Pentium 150 MHz PC running LINUX.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997). Program(s) used to refine structure: *SHELXL*97. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

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(*S*,*S*)-*trans*-2,3-Tetralindiol

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Abstract

Enantiomerically pure *trans*-2,3-tetralindiol [(*R*,*R*)- or (*S*,*S*)-*trans*-1,2,3,4-tetrahydro-2,3-naphthalenediol, $C_{10}H_{12}O_2$] crystallizes in a layered structure, with two independent molecules in the asymmetric unit and an unbalanced arrangement of hydrogen bonds. The hydroxyl groups of one molecule form normal, if somewhat long, hydrogen bonds, but one of the O atoms in the other molecule accepts two protons while the second accepts none. The four O···O distances range from 2.817 (3) to 2.921 (3) Å.

Comment

Brianso (1976) reported the melting points of the resolved enantiomers of *trans*-2,3-tetralindiol to be 436 K, and the eutectic temperature of the racemic phase with one of the enantiomerically pure phases to be 403 K. This eutectic temperature is only 5 K lower than the melting point of the racemic material (T_{fus} 408–409 K; Verkade *et al.*, 1928). Crystals of *rac*-*trans*-2,3-tetralindiol are therefore only marginally more stable than the crystals of the pure enantiomers. Since crystals of racemic compounds are usually considerably more stable than crystals of the corresponding enantiomerically pure materials (Jacques *et al.*, 1981; Brock *et al.*, 1991), we decided to investigate.

The structure of the enantiomerically pure isomer, (I), has been published previously (Brianso, 1976), but its precision is so low (R = 0.13; structure said to be disordered) that its accuracy was in doubt. The structure of the racemic compound is unknown. We had no difficulties obtaining high-quality crystals of the enantiomerically pure material (recrystallization from toluene), but were unable to obtain suitable crystals of the racemic compound. The latter grew, from the many

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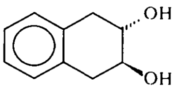
solvents we tried, either as clusters of exceptionally thin blades, or as tiny blocks with multiple re-entrant angles.



(I)

In crystals of the enantiomerically pure material, the two independent molecules form the expected hydrogen-bonded double layers (Fig. 2), but the bonds are surprisingly long (Table 1). The O···O distances in *cis*-2,3-tetralindiol (Klein *et al.*, 1983), which contains columns of molecules rather than double layers, are generally shorter [2.68 (1), 2.76 (1), 2.77 (1) and 2.82 (1) Å]. Moreover, the hydrogen-bonding pattern in the enantiomerically pure compound is unbalanced. Although atoms O1′ and O2′ each donate and accept one proton, atom O1 accepts two protons while atom O2 accepts none.

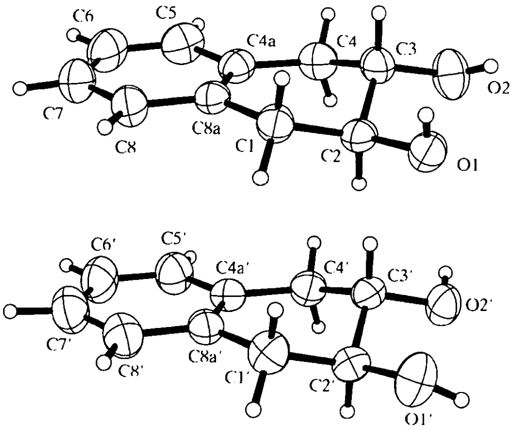
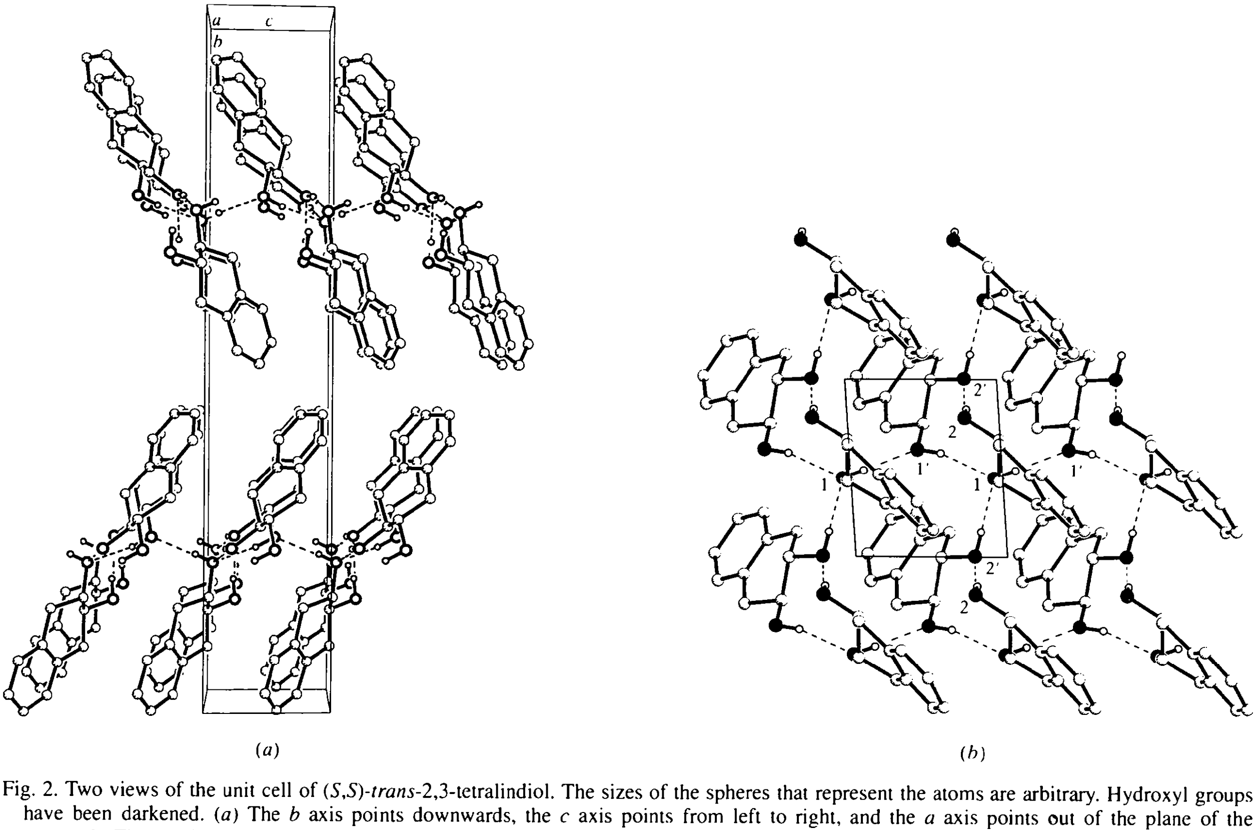


Fig. 1. Perspective drawing of the two independent molecules of (*S,S*)-*trans*-2,3-tetralindiol. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement. H atoms are shown as spheres of arbitrary radii.

The hydrogen-bonding pattern in the title compound is clearly not optimal. Formation of an ideal hydrogen-bonded network must be compromised by the necessity of accommodating some other structural feature. Perhaps the thickness mismatch between the aromatic and aliphatic ring systems is important. In the enantiomerically pure compound, the packing is 'loose' in the region of the aromatic rings, but increased internal motion in that part of the molecule is impossible because there is no appropriate low-frequency molecular deformation.



(*a*) (*b*)

Fig. 2. Two views of the unit cell of (*S,S*)-*trans*-2,3-tetralindiol. The sizes of the spheres that represent the atoms are arbitrary. Hydroxyl groups have been darkened. (*a*) The *b* axis points downwards, the *c* axis points from left to right, and the *a* axis points out of the plane of the paper. (*b*) The *c* axis points from left to right and the *a* axis points upwards; the layer is at *y* ~0.30. The molecules incorporating atoms O1 and O2 are above the plane of the paper; the molecules incorporating atoms O1′ and O2′ are below the plane of the paper.

The question as to why the enantiomerically pure material is about as stable as the racemic material is unanswered. We produced only one crystal of the racemic material (from toluene) that had no re-entrant angles and which was large enough to produce measurable diffraction peaks. The cell constants [10.33 (2), 7.63 (2), 11.18 (2) Å, 90.3 (2), 95.7 (2), 90.2 (2)°, V = 877 (4) Å^3 at 293 (2) K] were determined from eight reflections with θ values in the range 2.8–11.4°. Another three reflections could not be indexed, which suggested the crystal was twinned. A hemisphere of data having $\theta_{\max} = 17.5°$ (Mo $K\alpha$ radiation) was measured, but only 266 of 1628 reflections had $I > \sigma(I)$, and the structure could not be solved. The molecular volume for the racemic material [219 (1) Å^3] is, however, substantially larger than for the enantiomerically pure material [211.1 (3) Å^3]. Larger molecular volume is usually correlated with higher energy.

Experimental

A sample of the title material was supplied by Professor A. Collet, Lyon, France. Crystals were grown from toluene solutions by slow evaporation, and were obtained as prisms elongated parallel to the c axis, with major faces {110}, (010), $\{\bar{1}\bar{1}0\}$, and $(0\bar{1}0)$.

Crystal data

$C_{10}H_{12}O_2$
$M_r = 164.20$
Monoclinic
$P2_1$
a = 5.9103 (4) Å
b = 28.2842 (19) Å
c = 5.0635 (3) Å
β = 93.975 (7)°
V = 844.42 (10) Å^3
$Z = 4$
D_x = 1.292 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 22 reflections
θ = 12.2–13.0°
μ = 0.083 mm^{-1}
T = 295 (1) K
Prism
0.5 × 0.2 × 0.2 mm
Colorless

Data collection

Enraf–Nonius CAD-4-VAX diffractometer
ω scans
Absorption correction: none
3779 measured reflections
1985 independent reflections
1368 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.031$
$\theta_{\max} = 27.49°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 36$
$l = -6 \rightarrow 6$
3 standard reflections
frequency: 60 min
intensity decay: <0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.076$
$S = 1.021$
1985 reflections
313 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.044P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.057$
$\Delta\rho_{\max}$ = 0.12 e Å^{-3}
$\Delta\rho_{\min}$ = −0.16 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C2	1.445 (3)	O1′—C2′	1.425 (3)
O2—C3	1.428 (3)	O2′—C3′	1.433 (3)
O1—C2—C3—O2	54.6 (3)	O1′—C2′—C3′—O2′	56.0 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1$^{\rm i}$	0.81 (3)	2.01 (3)	2.817 (3)	176 (3)
O2—H2···O2′	0.86 (3)	2.02 (3)	2.862 (3)	168 (3)
O1′—H1′···O1	0.89 (4)	2.06 (4)	2.921 (3)	162 (4)
O2′—H2′···O1$^{\rm ii}$	0.83 (3)	2.00 (3)	2.816 (3)	165 (3)

Symmetry codes: (i) $x, y, 1 + z$; (ii) $1 + x, y, z$.

All C—C and C—O bond lengths are normal. The range of the C—H bonds is 0.92 (3)–1.03 (3) Å. The $U_{\rm iso}$ values are 0.058 (9), 0.056 (9), 0.100 (13) and 0.051 (8) Å^2 for H1, H2, H1′ and H2′, and range from 0.029 (5) to 0.074 (9) Å^2 for the other H atoms. The absolute configuration was assigned arbitrarily.

Data collection: *CAD*-4 *Manual* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *Manual*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

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