

**(1*S*\*,2*R*\*,2'*R*\*)-2-Phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol at 193 K**Jarno Kansikas<sup>a\*</sup> and Kaija Sipilä<sup>b</sup><sup>a</sup>Department of Chemistry, Laboratory of Inorganic Chemistry, PO Box 55, 00014 University of Helsinki, Finland, and <sup>b</sup>Department of Chemistry, Laboratory of Organic Chemistry, PO Box 55, 00014 University of Helsinki, FinlandCorrespondence e-mail:  
jarno.kansikas@helsinki.fi**Key indicators**Single-crystal X-ray study  
*T* = 193 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
*R* factor = 0.046  
*wR* factor = 0.107  
Data-to-parameter ratio = 15.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-Phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol,  $\text{C}_{20}\text{H}_{24}\text{O}_2\text{S}_2$ , was synthesized as a mixture of four diastereoisomers. An isomer which crystallizes in the centrosymmetric monoclinic space group  $P2_1/c$  with the relative configuration (1*S*\*,2*R*\*,2'*R*\*) is presented. Intermolecular hydrogen bonds between the hydroxyl group and the O atom of the tetrahydropyran ring of a neighbouring molecule [ $\text{O} \cdots \text{O} 2.751 (2) \text{ \AA}$ ] form zigzag chains in the *c*-axis direction. The tetrahydropyran ring has a chair conformation with an axial S-side chain.

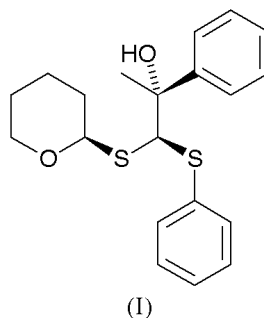
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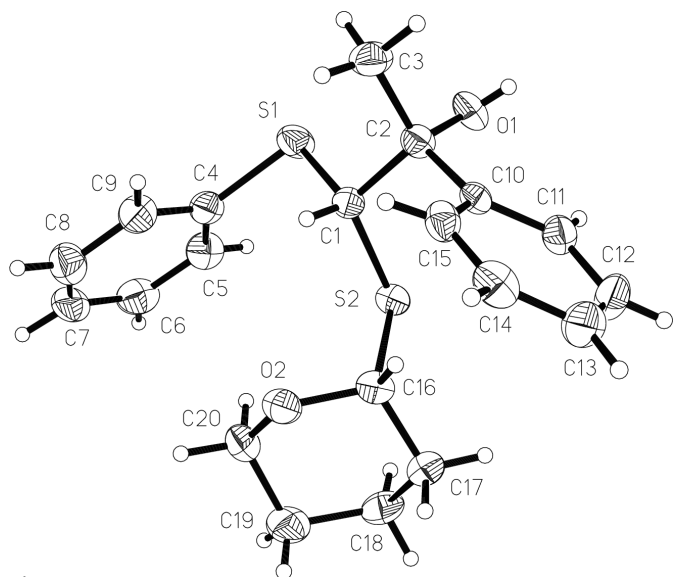
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**Comment**

According to the crystallographic atomic labelling of 2-phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol (Fig. 1), the C atom bearing the S-side chain in the tetrahydropyran ring is C16 and thus the relative configuration of the title compound may also be named (1*S*\*,2*R*\*,16*R*\*), (I).



We have earlier reported the structure of another diastereoisomer (1*S*\*,2*S*\*,16*R*\*), (II) (Kansikas *et al.*, 1995). A very closely related compound 1-phenyl-2-phenylthio-2-(tetrahydropyran-2-ylthio)ethanol, ( $\text{C}_{19}\text{H}_{22}\text{O}_2\text{S}_2$ ), where the methyl group is replaced by an H atom, also forms four diastereoisomers. Applying the above labelling scheme, atom C15 is now in the tetrahydropyran ring bonded to S. Three of the ethanol diastereoisomers crystallize in non-centrosymmetric space groups as conglomerates of enantiomeric crystals with the configurations (1*S*,2*S*,15*R*), (III) (Kansikas *et al.*, 1996), (1*R*,2*S*,15*S*), (IV), and (1*S*,2*S*,15*S*), (V) (Kansikas & Sipilä, 2000). The fourth diastereoisomer crystallizes in a centrosymmetric space group with the relative configuration (1*R*\*,2*S*\*,15*R*\*), (VI) (Sipilä *et al.*, 2001). Some interatomic distances, angles and torsion angles for (I) are listed in Table 1. The S—C distances fall well within the values found in the compounds (II)–(VI), but the C1—S1—C4 angle of  $99.21 (1)^\circ$  is the smallest among them. Torsion angles around the S atoms in compounds (I)–(VI) vary considerably and that gives rise to

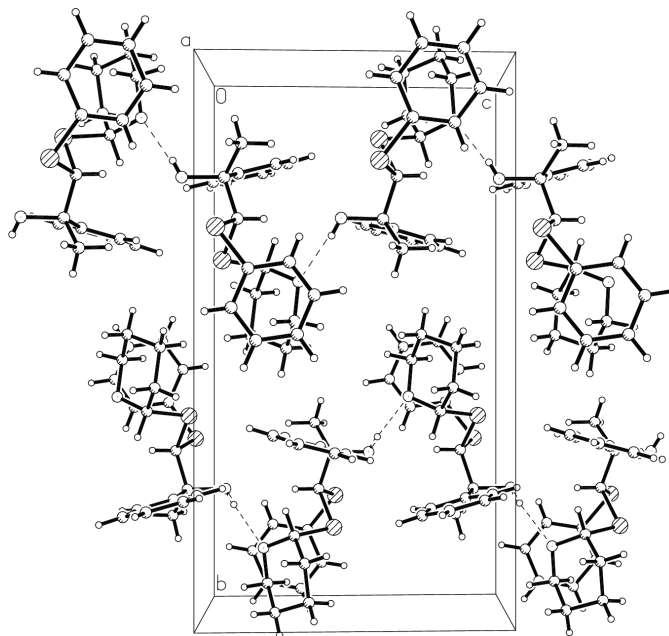


**Figure 1**  
View of (I) showing the crystallographic atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

very different appearances of these otherwise rather similar molecules. In the compound (I) the hydroxyl group forms an intermolecular hydrogen bond to the O atom of the tetrahydropyran ring of the neighbouring molecule at the equivalent position ( $x, \frac{1}{2} - y, z - \frac{1}{2}$ ) (Fig. 2 and Table 2). A comparable intermolecular hydrogen bond, but between the molecules with the symmetry code ( $x, y, z - 1$ ), is found in the ethanol derivative (V). The O...O distances are 2.751 (2) Å for (I) and 2.764 (3) Å for (V). The title isomer (I) crystallizes in the space group  $P2_1/c$  (No. 14), whereas the ethanol derivative (IV) with the same relative configuration crystallizes in the non-centrosymmetric space group  $P2_12_12_1$  (No. 19) and possesses an intramolecular hydrogen bond between the hydroxyl group and the O atom of the tetrahydropyran ring similar to (II) and (III).

## Experimental

2-Phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol was synthesized as a mixture of four diastereoisomers according to a procedure reported previously (Kansikas *et al.*, 1995). The diastereoisomers (1*S*\*,2*R*\*,2'*R*\*)-2-phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol, (I), and (1*S*\*,2*S*\*,2'*R*\*)-2-phenyl-1-phenylthio-1-(tetrahydropyran-2-ylthio)propan-2-ol, (II) (Kansikas *et al.*, 1995), were separated by high-pressure liquid chromatography (HPLC) from the crude product and recrystallized from absolute ethanol. The relative amounts of the diastereoisomers in elution order were: 31% (II) and 19% (I). The remaining two diastereoisomers (50% of the total amount) could not be separated by this procedure. HPLC separation was performed with an ISCO 2350 liquid chromatograph equipped with a Shimadzu SPD-6A UV spectrophotometric detector and a Shimadzu C-R6A Chromatopac. Components were monitored measuring the absorption at 254 nm. The column used was Merck Lichrocart Si 60 (250 × 10 mm ID), 5 μm, the mobile phase 2% ethyl acetate in dichloromethane; flow rate 7 ml min<sup>-1</sup>. The NMR spectra were recorded on a Varian Gemini



**Figure 2**  
Fraction of the molecular packing showing the hydrogen-bond scheme of (I) in the *c*-axis direction.

200 spectrometer using tetramethylsilane as an internal standard. The assignments are based on chemical-shift data and DEPT measurements. <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ 1.4–1.9 (6H, *m*; CH<sub>2</sub>), 1.80 (*s*, CH<sub>3</sub>), 3.4–3.6 and 3.6–3.7 (2H, *m*; OCH<sub>2</sub>), 4.7 (1H, *s*, SCHS), 4.9 (1H, *s*, OH), 5.2–5.3 (1H, *m*, OCHS), 7.2–7.6 (10H, *m*; aromatic H). <sup>13</sup>C NMR (50 MHz; CDCl<sub>3</sub>): δ 21.7 and 25.1 and 30.7 (CH<sub>2</sub>), 29.5 (CH<sub>3</sub>), 65.1 (OCH<sub>2</sub>), 68.5 (SCHS), 78.2 (CPh), 80.1 (OCS), 125.1–147.3 (aromatic C).

### Crystal data

C<sub>20</sub>H<sub>24</sub>O<sub>2</sub>S<sub>2</sub>  
*M<sub>r</sub>* = 360.51  
 Monoclinic,  $P2_1/c$   
*a* = 9.2440 (18) Å  
*b* = 19.239 (4) Å  
*c* = 10.682 (2) Å  
 $\beta$  = 93.37 (3)°  
*V* = 1896.5 (6) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.263 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 4–10°  
 $\mu$  = 0.29 mm<sup>-1</sup>  
*T* = 193 (2) K  
 Prismatic, colourless  
 0.36 × 0.30 × 0.25 mm

### Data collection

Rigaku AFC-7S diffractometer  
 $\omega/2\theta$  scans  
 3637 measured reflections  
 3418 independent reflections  
 2438 reflections with  $I > 2\sigma(I)$   
*R<sub>int</sub>* = 0.029  
 $\theta_{\max}$  = 25.3°

*h* = 0 → 11  
*k* = 0 → 23  
*l* = -12 → 12  
 3 standard reflections every 100 reflections  
 intensity decay: <0.1%

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.046  
*wR*(*F*<sup>2</sup>) = 0.107  
*S* = 1.05  
 3418 reflections  
 217 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.1099P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{Å}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

S1—C4	1.778 (3)	C1—S2	1.819 (2)
S1—C1	1.831 (2)	S2—C16	1.828 (2)
O1—C2	1.423 (3)	O2—C16	1.424 (3)
C1—C2	1.546 (3)	O2—C20	1.431 (3)
C4—S1—C1	99.20 (11)	C1—S2—C16	101.66 (11)
C2—C1—S2	115.16 (17)	C16—O2—C20	114.32 (19)
C2—C1—S1	108.91 (16)	O1—C2—C1	104.76 (19)
S2—C1—S1	107.30 (12)	O2—C16—S2	112.52 (16)
C4—S1—C1—C2	-162.48 (17)	S1—C1—C2—O1	-54.5 (2)
C4—S1—C1—S2	72.25 (15)	S2—C1—C2—C3	-176.06 (17)
C2—C1—S2—C16	96.02 (18)	S1—C1—C2—C3	63.4 (2)
S1—C1—S2—C16	-142.57 (12)	S1—C1—C2—C10	-173.97 (16)
S2—C1—C2—O1	66.0 (2)	C1—S2—C16—O2	67.04 (18)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1A $\cdots$ O2 <sup>i</sup>	0.84	1.92	2.751 (2)	168

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

Data collection: AFC-7S software; cell refinement: AFC-7S software; data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

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