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4-Hydroxy-4-methyl-2,6-diphenyl-5,6-dihydro-4*H*-1,3-thiazine

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.102
Data-to-parameter ratio = 14.5

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

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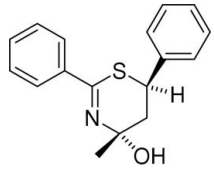
In the title compound, $C_{17}H_{17}NOS$, the phenyl ring at position 6 of the thiazine ring is *trans* to the hydroxy group. The thiazine ring is in a sofa conformation.

Comment

Mycobacterium tuberculosis is the main causative agent of a chronic and often fatal condition in humans known as tuberculosis (TB). It is estimated that one third of the world's population is TB-infected, with about eight million new cases annually, and of these, 3.1 million die annually. TB is currently the leading killer of youths, women, and AIDS patients in the world (Snider *et al.*, 1994). The antimycobacterial activities of 5,6-dihydro-4*H*-1,3-thiazine derivatives have been investigated. They show activity against *Mycobacterium tuberculosis* H37Rv (ATCC 27294) (Koketsu *et al.*, 2002). Confirmation of the conformation of the thiazine ring is essential for the study of structure–biological activity relationships of thiazine derivatives. 4,6-Disubstituted-4-hydroxy-5,6-dihydro-4*H*-1,3-thiazines are synthesized by the $BF_3{\cdot}Et_2O$-catalysed reaction of a primary thioamide with an α,β-unsaturated ketone (Koketsu *et al.*, 1999, 2002). They are obtained as diastereomers resulting from the asymmetric centres at positions 4 and 6 of the thiazine ring. In the present diastereomer, (I), the relationship between the OH group at position 4 and the phenyl group at position 6 is *trans*.



(I)

The molecular structure of (I) is shown in Fig. 1. The thiazine ring of (I) adopts a sofa conformation, with atom C3 deviating by 0.686 (3) Å from the plane of the remaining five atoms which lie in a common plane (r.m.s. deviation 0.007 Å). There are intermolecular O—H···N hydrogen bonds between neighbouring molecules (Table 2 and Fig. 2).

Experimental

4-Phenyl-3-buten-2-one (0.58 g, 4.0 mmol) was added to a solution of thiobenzamide (0.55 g, 4.0 mmol) in dry dichloromethane (40 ml) at room temperature under an argon atmosphere. To this solution was added $BF_3{\cdot}Et_2O$ (1.2 mmol). The reaction mixture was stirred for 2 h, quenched with saturated sodium carbonate solution, and extracted with dichloromethane. The extracts were dried (Na_2SO_4) and

evaporated to dryness. Recrystallizations from ether/hexane (4:6) gave (I) as crystals (yield 0.65 g, 69%; m.p. 399.6–400.0 K). IR (KBr) 3188, 1590 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, δ, p.p.m.): 1.49 (3H, *s*, CH_3), 1.89 (1H, *t*, J = 13.2 Hz), 2.30 (1H, *d*, J = 17.2 Hz), 3.63 (1H, *br*, OH), 4.52 (1H, *d*, J = 17.2 Hz, C6), 7.29–7.47 (8H, *m*, Ar), 7.81–7.84 (2H, *m*, Ar); ^{13}C NMR (100 MHz, $CDCl_3$, δ, p.p.m.): = 26.2, 38.6, 44.2, 85.8, 126.7, 127.7, 128.2, 128.8, 130.8, 138.2, 139.8, 158.1; MS (CI): m/z = 284 [M^++1].

Crystal data

$C_{17}H_{17}NOS$
M_r = 283.39
Orthorhombic, *Pbca*
a = 12.1452 (2) Å
b = 10.6688 (2) Å
c = 23.1573 (6) Å
V = 3000.60 (11) Å^3
Z = 8
D_x = 1.255 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10470 reflections
θ = 3.0–25.0°
μ = 0.21 mm^{-1}
T = 190 (2) K
Prism, colourless
0.13 × 0.13 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
φ or ω scans
Absorption correction: none
45044 measured reflections
2663 independent reflections
1718 reflections with $I > 2\sigma(I)$
R_{int} = 0.120
θ_{max} = 25.1°
$h = -14 \rightarrow 14$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.102
S = 1.02
2663 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.1974P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.762 (2)	C2—O1	1.425 (3)
S1—C4	1.826 (2)	C2—C11	1.516 (3)
C1—N1	1.283 (3)	C2—C3	1.522 (3)
C1—C5	1.483 (3)	C3—C4	1.522 (3)
N1—C2	1.479 (3)	C4—C12	1.509 (3)
C1—S1—C4	102.84 (10)	O1—C2—C3	106.48 (18)
N1—C1—C5	118.51 (19)	N1—C2—C3	113.38 (18)
N1—C1—S1	128.66 (17)	C11—C2—C3	110.97 (18)
C5—C1—S1	112.80 (16)	C2—C3—C4	113.10 (18)
C1—N1—C2	121.39 (18)	C12—C4—C3	113.88 (17)
O1—C2—N1	108.00 (17)	C12—C4—S1	107.72 (15)
O1—C2—C11	111.09 (18)	C3—C4—S1	109.60 (15)
N1—C2—C11	106.93 (18)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H9···N1^{i}	0.84	2.05	2.888 (2)	173

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

All H atoms were placed in idealized positions and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C)], with C—H = 0.95–0.99 Å and O—H = 0.84 Å. The displacement parameter of the hydroxy H atom was refined. In addition, the torsion angles involving the methyl and hydroxyl group were refined.

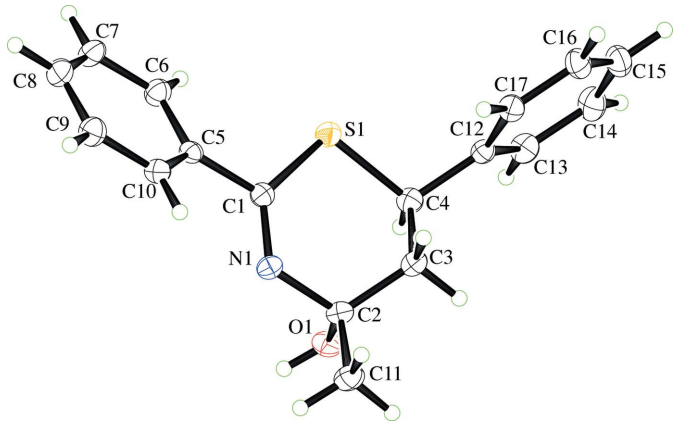


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

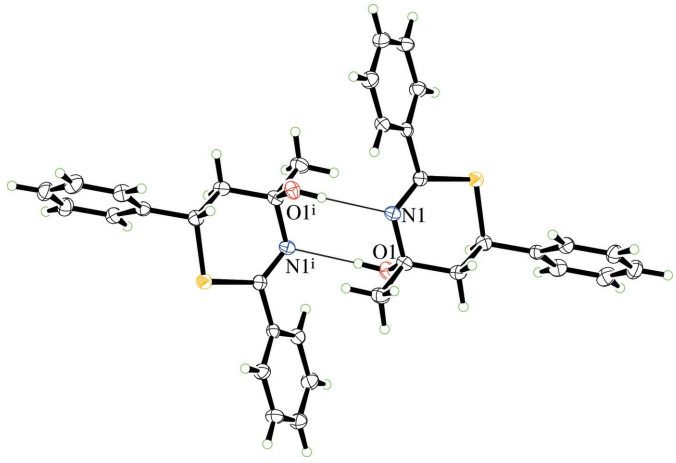


Figure 2
Hydrogen-bonded (dashed lines) dimeric structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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