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Methyl (3-hydroxy-4-oxo-2-phenyl-1,5-benzothiazepin-5-yl)acetate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.001 Å
R factor = 0.045
wR factor = 0.048
Data-to-parameter ratio = 11.8

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

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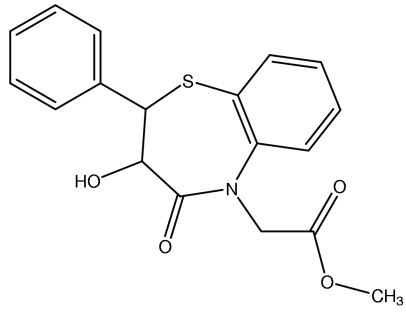
The structure of the title compound, $C_{18}H_{17}NO_4S$, has been established by an X-ray crystallographic study. The N and S atoms of the thiazine ring are almost in the benzo plane, whereas the three C atoms are above the plane.

Comment

A number of 1,5-benzothiazepine derivatives have been shown to exhibit pharmacological properties as calcium antagonists (Akiyoshi *et al.*, 1992), coronary vasodilators (Hirozumi *et al.*, 1991), antihypertensives (Hiroshi & Hirshi, 1992) and blood-platelet aggregation inhibitors (Hirozumi & Trunehiro, 1990). The success of this class of products has stimulated significant activity in related chemical synthesis, directed at the synthesis of both analogous and enantiomerically pure compounds.

The thiazepine ring in the title compound, (I), is fused with the benzo ring and has four substituents: phenyl in position 2, hydroxy in position 3, oxo in position 4 and acetate in position 5. The seven-membered ring contains a planar N5—C6—C11—S1 fragment (r.m.s. deviation: 0.014 Å), almost coplanar with the adjacent benzo ring [angle 5.3 (3)°]. The three other atoms of the benzothiazepine system (C2, C3 and C4) are on the same side of the N5—C6—C11—S1 fragment.



(I)

Experimental

A mixture of 2-aminothiophenol (110 mmol, 13.75 g) with ethyl 3-phenylglycidate (110 mmol, 21.12 g) was heated with stirring at 393 K for 1 h under a nitrogen atmosphere and then at 433 K for 16 h. The cooled mixture was dissolved in ethanol (30 ml) and allowed to stand at 278 K overnight. The precipitated needles were filtered off, washed with ethanol and recrystallized from ethanol to give 7.4 g (25%) of 3-hydroxy-2-phenyl-1,5-benzothiazepin-4(5*H*)-one. To a solution of this compound (5 mmol, 1.36 g) in acetone (40 ml) was added K_2CO_3 (20 mmol, 2.76 g) and methyl chloroacetate (40 mmol, 4.34 g). The mixture was refluxed and the reaction was monitored by thin-layer chromatography (ether/$CHCl_3$). After filtration, the solvent was

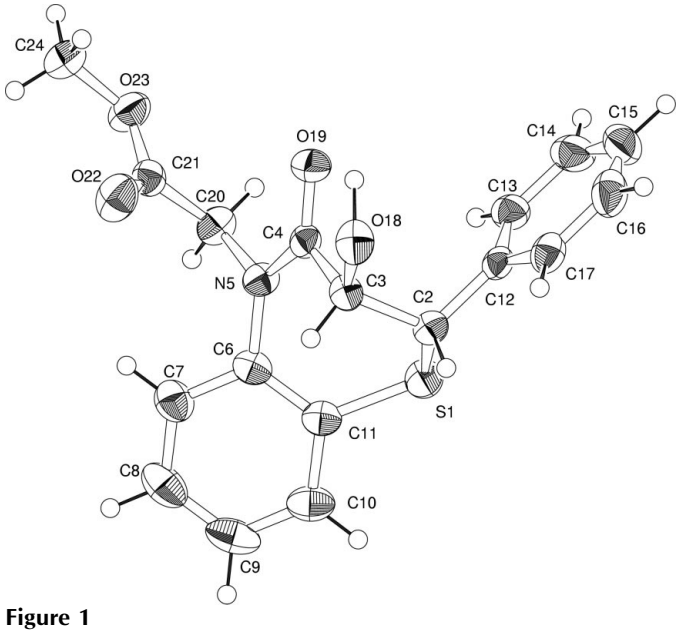


Figure 1
Perspective view of the title molecule showing the labelling of the atoms, with displacement ellipsoids at the 50% probability level.

evaporated *in vacuo* to give 1.20 g (70%) of methyl (3-hydroxy-4-oxo-2-phenyl-1,5-benzothiazepin-5-yl) acetate, (I), m.p. 429 K. Analysis calculated for $C_{18}H_{17}NO_2S$: C 62.96, H 4.99, N 4.08%; found C 62.88, H 5.10, N 4.21%. Mass FAB+ (NBA): 344 (M + 1).

Crystal data

$C_{18}H_{17}NO_4S$	D_x = 1.416 Mg m^{-3}
M_r = 343.40	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 10319 reflections
a = 13.6308 (8) Å	θ = 1–26.4°
b = 15.1336 (9) Å	μ = 0.22 mm^{-1}
c = 16.1540 (10) Å	T = 298 K
β = 151.086 (3)°	Prism, violet
V = 1611.2 (2) Å^3	0.4 × 0.3 × 0.2 mm
Z = 4	

Data collection

KappaCCD diffractometer	R_{int} = 0.046
φ scans	θ_{max} = 26.4°
3302 measured reflections	$h = -16 \rightarrow 16$
3156 independent reflections	$k = -18 \rightarrow 0$
2569 reflections with $I > 3\sigma(I)$	$l = -20 \rightarrow 16$

Refinement

R = 0.045	H-atom parameters not refined
wR = 0.048	$w = 1/[\sigma^2(F_o^2) + 0.03F_o^2]$
S = 1.36	$(\Delta/\sigma)_{max}$ = 0.015
2569 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
217 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *maXus* (Mackay *et al.*, 1999); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *maXus*.

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