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(*E*)-2-Acetyl-3-hydroxy-*N*-phenylbut-2-enethioamide

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The title compound, $C_{12}H_{13}NO_2S$, was prepared by the reaction of acetylacetone with phenyl isothiocyanate. In addition to the intramolecular O—H···O hydrogen bond, there is a weak intermolecular N—H···S hydrogen bond, which links the molecules into chains extended along the *c* axis. The crystal packing is further stabilized by van der Waals forces.

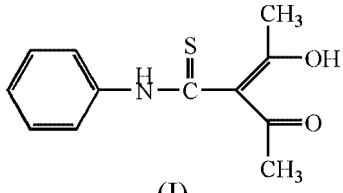
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.146
Data-to-parameter ratio = 17.8

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

Comment

Acetylacetone is a well known intermediate product, which can be used as an annexing agent in gasoline, a lubricant and a desiccant in paint. It has been found to possess fungicidal and insecticidal activities (Si, 1999). In our search of new derivatives of acetylacetone, we have synthesized the title compound, (I), by the reaction of acetylacetone and phenyl isothiocyanate. We report here its crystal structure (Fig. 1).



(I)

All bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). The hydroxy H atom is involved in an intramolecular O—H···O hydrogen bond (Table 1). The atoms of the acetylacetone group are essentially coplanar, with a maximum deviation from the mean plane (*p*1) of 0.023 (2) Å for atom O2. Atoms S1, N1, C6, C7 and C8 are also coplanar, with a maximum deviation from the mean plane (*p*2) of 0.032 (2) Å for atom N1. The phenyl ring C1–C6 makes dihedral angles with the mean planes *p*1 and *p*2 of 71.5 (2) and 18.9 (2)°, respectively, while the dihedral angle *p*1/*p*2 is 89.4 (1)°.

In the crystal structure, there is a weak intermolecular N—H···S hydrogen bond (Table 1), which links the molecules into chains extended along the *c* axis. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

The title compound was prepared by the reaction of acetylacetone (0.02 mol) with phenyl isothiocyanate (0.02 mol). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from a DMSO solution at room temperature.

Crystal data

$C_{12}H_{13}NO_2S$
M_r = 235.29
Orthorhombic, $P2_12_12_1$
a = 11.249 (2) Å
b = 10.506 (2) Å
c = 10.376 (2) Å
V = 1226.3 (4) Å^3
Z = 4
D_x = 1.274 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 (2) K
Block, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
5616 measured reflections
2675 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.0°
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.146
S = 0.94
2675 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1117P)^2 + 0.097P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.122 (12)
Absolute structure: Flack (1983), 1126 Friedel pairs
Flack parameter: −0.13 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···S1^{i}	0.86	2.56	3.408 (2)	169
O2—H2···O1	0.86 (1)	1.66 (2)	2.468 (5)	156 (4)

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

C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.96 Å and with U_{iso}(H) values of 1.2 or 1.5 times U_{eq}(C). The H atom attached to the O atom was located in a difference map and freely refined.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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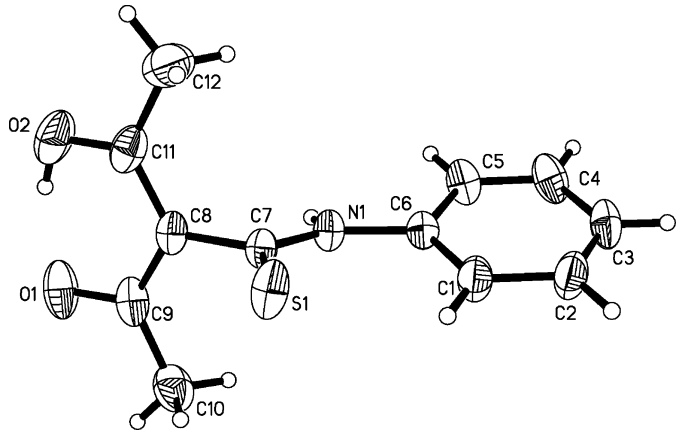

Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

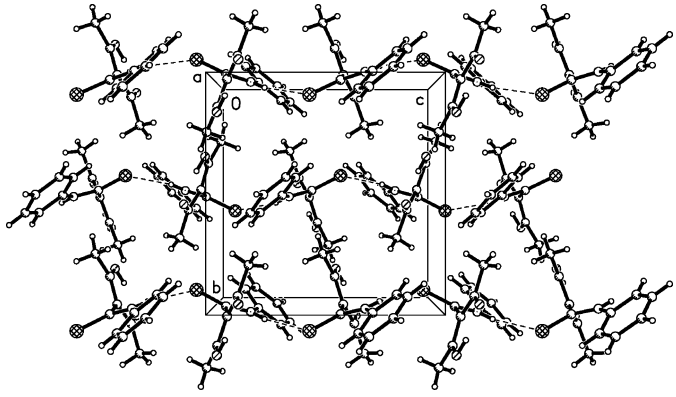

Figure 2
The crystal packing viewed down the *a* axis. The intermolecular hydrogen bonds are shown as dashed lines.

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