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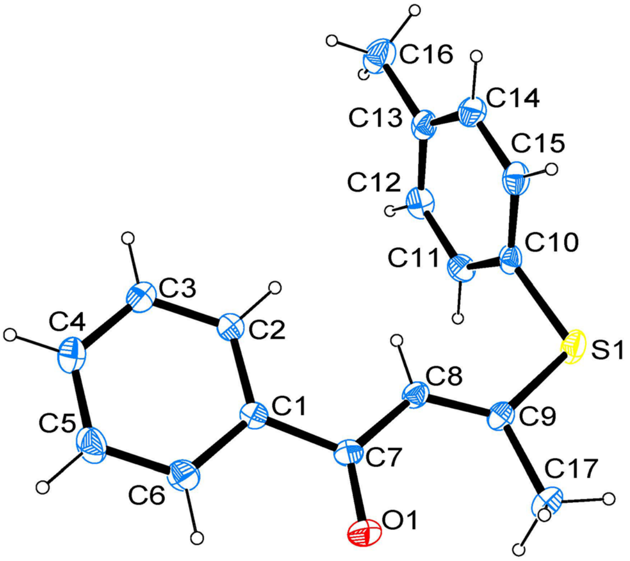
The crystal structure of (*E*)-1-phenyl-3-(*p*-tolylthio) but-2-en-1-one, $C_{17}H_{16}OS$



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Abstract

$C_{17}H_{16}OS$, monoclinic, $P2_1/n$ (no. 14), a = 5.4605(12) Å, b = 19.767(4) Å, c = 13.357(4) Å, β = 92.834(3)°, V = 1,439.9(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0398, w$R_{ref}(F^2)$ = 0.1144, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size	0.31 × 0.26 × 0.23 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.21 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7,262, 2,541, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,147
$N(param)_{refined}$:	175
Programs:	Bruker,[1] SHELX,[2,3] WinGX/ORTEP[4]

1 Source of material

All chemicals were purchased from commercial sources and used as received without further purification. An oven-dried flask was charged with 1-phenylbuta-2,3-dien-1-one (0.2 mmol), *p*-toluenethiol (0.2 mmol) and CH_3CN (2 mL). Then, after the mixture was stirred at room temperature for 3.5 h, the mixture was extracted with ethyl acetate. The combined organic layer was dried over Na_2SO_4, and concentrated in vacuum, and the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the pure title product. Finally, crystals were obtained by slow evaporation from petroleum ether (b.p.: 60–90 °C) solution at room temperature.

2 Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq} (parent C-atom).

3 Comment

Vinyl sulfides play an important role as versatile intermediates in organic chemistry and coordination chemistry, and they also are vital core structures for many natural products and biologically active compounds.[5–8] Due to the molecular structure of a compound determining its properties, considerable interest has been inclined toward the development of structures for access to vinyl sulfides.[9–13] Therefore, we have crystallized the title compound and report its crystal structure in this context.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	$U_{iso}*/U_{eq}$
C1	0.5988 (3)	0.35369 (8)	0.95850 (12)	0.0469 (4)
C2	0.5455 (3)	0.31101 (9)	0.87880 (14)	0.0607 (5)
H2	0.642271	0.272932	0.870230	0.073*
C3	0.3509 (4)	0.32377 (11)	0.81143 (15)	0.0717 (6)
H3	0.318389	0.294341	0.758096	0.086*
C4	0.2065 (4)	0.37912 (10)	0.82255 (15)	0.0697 (5)
H4	0.074617	0.387250	0.777397	0.084*
C5	0.2560 (4)	0.42262 (10)	0.90027 (17)	0.0750 (6)
H5	0.158807	0.460766	0.907646	0.090*
C6	0.4503 (4)	0.41012 (9)	0.96810 (15)	0.0641 (5)
H6	0.482029	0.439981	1.020963	0.077*
C7	0.8027 (3)	0.34223 (8)	1.03630 (12)	0.0508 (4)
C8	0.9408 (3)	0.27906 (8)	1.03330 (12)	0.0502 (4)
H8	0.888419	0.246969	0.986031	0.060*
C9	1.1368 (3)	0.26365 (8)	1.09323 (12)	0.0510 (4)
C10	1.1306 (3)	0.13821 (8)	0.99565 (13)	0.0532 (4)
C11	0.9234 (3)	0.10373 (9)	1.02104 (15)	0.0615 (5)
H11	0.865498	0.108228	1.085016	0.074*
C12	0.8021 (4)	0.06261 (10)	0.95175 (16)	0.0687 (5)
H12	0.661127	0.040166	0.969655	0.082*
C13	0.8836 (4)	0.05377 (9)	0.85660 (15)	0.0632 (5)
C14	1.0906 (4)	0.08811 (10)	0.83221 (15)	0.0682 (5)
H14	1.149352	0.083047	0.768468	0.082*
C15	1.2132 (3)	0.12995 (10)	0.90025 (14)	0.0652 (5)
H15	1.352751	0.152853	0.881825	0.078*
C16	0.7489 (5)	0.00794 (12)	0.78190 (19)	0.0974 (8)
H16A	0.661491	0.034889	0.732163	0.146*
H16B	0.635077	−0.019731	0.816040	0.146*
H16C	0.864623	−0.020447	0.750045	0.146*
C17	1.2511 (4)	0.30737 (10)	1.17439 (14)	0.0655 (5)
H17A	1.297136	0.350015	1.146504	0.098*
H17B	1.394106	0.285312	1.203590	0.098*
H17C	1.135417	0.314800	1.225082	0.098*
O1	0.8440 (3)	0.38557 (7)	1.10038 (10)	0.0750 (4)
S1	1.29935 (9)	0.18752 (3)	1.08569 (4)	0.0691 (2)

In the molecular structure (Figure), the dihedral angle between the almost planar vinylthio group (S1/C17/C7–C9) and the benzoyl group (O1/C1–C7) is 8.1°. The C(10)–S(1)–C(9)–C(8) and C(10)–S(1)–C(9)–C(17) torsion angles are – 5.54(18)° and 179.97(12)°. The bond lengths and angles are all in the expected ranges.[14] The thioether bond distances are 1.7526(18) Å for S(1)–C(9) and 1.7708(18) Å for S(1)–C(10), respectively, and the C(3)–S(1)–C(6) bond angle is 105.28(8)°. The bond lengths of C(8)–C(9), C(7)–C(8) and O(1)–C(7) are 1.340(2), 1.460(2) and 1.224(2) Å, respectively. The title compound forms a 2D structure by C–H$\cdots\pi$ and C–H$\cdots$O hydrogen bonds.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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