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# Ethyl 3,3-bis(benzylsulfanyl)acrylate

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**Key indicators**

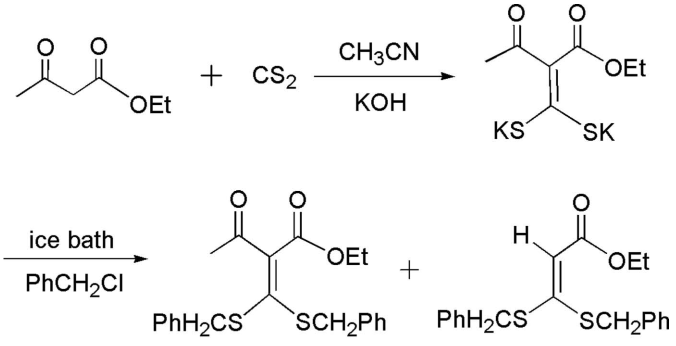
Single-crystal X-ray study
*T* = 294 K
Mean σ(C–C) = 0.005 Å
*R* factor = 0.037
*wR* factor = 0.113
Data-to-parameter ratio = 15.8

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

In the crystal structure of the title compound, $C_{19}H_{20}O_2S_2$, the molecules form stacks along the *b* axis, with short intermolecular S···C distances of 3.453 (3) and 3.560 (3) Å. Weak intermolecular C—H···O interactions provide further stabilization of the crystal packing.

## Comment

α-Acetyl dithioacetal ketene compounds constitute an important class of organic synthethic intermediates (Dieter, 1986; Junjappa *et al.*, 1990). It was reported (Li *et al.*, 2005; Zhao *et al.*, 2000) that the analogue of α-acetyl-α-ethoxycarbonyldibenzylthioacetal, (1), could be prepared by the reaction shown in the scheme. However, when the two original compounds were mixed first before being added to the reaction system, the title compound, (2), was obtained in 99% yield. We present here the crystal structure of (2).



(1) (2)

The bond lengths and angles in (2) are as expected for this type of compound. In the crystal structure, the molecules form stacks along the *b* axis, with short intermolecular S···C distances of 3.453 (3) and 3.560 (3) Å. Weak intermolecular C—H···O interactions (Table 1) provide further stabilization of the crystal packing (Fig. 2).

## Experimental

A mixture of ethyl acetoacetate (26.0 g, 0.20 mol) and carbonyl disulfide (15.2 g, 0.20 mol) was added dropwise to a solution of ground potassium hydroxide (22.4 g, 0.40 mol) in acetonitrile (150 ml) over a period of 50 min with a temperature no higher than 283 K. The reaction mixture was stirred for 2 h and then benzyl chloride (50.0 g, 0.40 mol) was added slowly. After stirring for a further 4 h, the resulting solution was filtered and the solid obtained was recrystallized from ethanol, yielding light-yellow single crystals of (2).

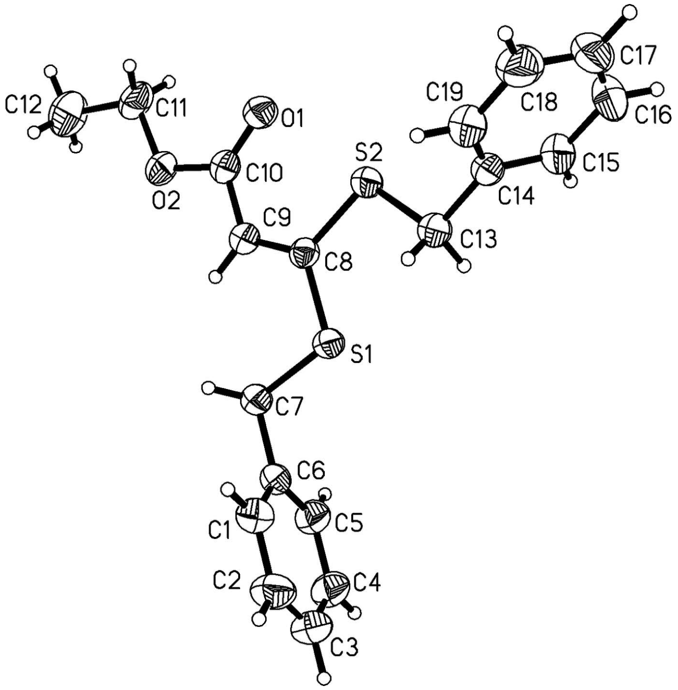


**Figure 1**
A view of compound (2), with displacement ellipsoids drawn at the 35% probability level.

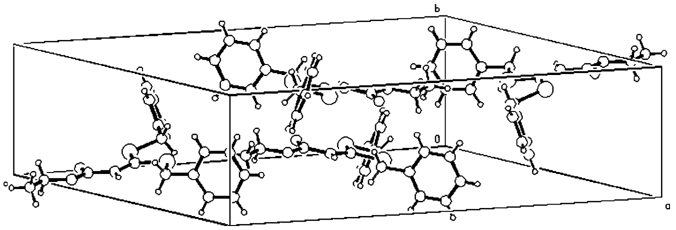


**Figure 2**
The molecular packing of (2).

*Crystal data*

$C_{19}H_{20}O_2S_2$
$M_r = 344.49$
Monoclinic, $P2_1/c$
$a = 14.582$ (6) Å
$b = 6.909$ (3) Å
$c = 22.653$ (7) Å
$\beta = 125.482$ (18)°
$V = 1858.4$ (13) Å$^3$
$Z = 4$

$D_x = 1.231$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 1699 reflections
$\theta = 2.8$–23.1°
$\mu = 0.29$ mm$^{-1}$
$T = 294$ (2) K
Prism, light-yellow
0.34 × 0.20 × 0.12 mm

*Data collection*

Bruker APEX-II CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.932$, $T_{\max} = 0.965$
9650 measured reflections

3297 independent reflections
2313 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\max} = 25.0°$
$h = -17 \rightarrow 16$
$k = -8 \rightarrow 7$
$l = -26 \rightarrow 26$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.113$
$S = 1.06$
3297 reflections
209 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 0.3616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.19$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1···O1$^{\rm i}$ | 0.93 | 2.54 | 3.414 (3) | 156 |
| C13—H13$A$···O2$^{\rm i}$ | 0.97 | 2.64 | 3.592 (3) | 166 |

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement as riding, with $U_{\rm iso}$(H) set at $1.2U_{\rm eq}$(C) for $CH_2$ and CH H atoms, and $1.5U_{\rm eq}$(C) for $CH_3$ H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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