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Acetyl Benzyl Disulfide

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Abstract. *S*-Benzylthio thioacetate, $C_9H_{10}OS_2$, $M_r = 198\cdot31$, monoclinic, $P2_1$, $a = 5\cdot7112\ (5)$, $b = 8\cdot1912\ (5)$, $c = 10\cdot6486\ (8)$ Å, $\beta = 92\cdot230\ (8)°$, $V = 497\cdot8\ (1)$ Å^3, $Z = 2$, $D_m = 1\cdot32\ (1)$, $D_x = 1\cdot323$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 0\cdot47$ mm^{-1}, $F(000) = 208$, $T = 299\ (1)$ K, $R_F = 0\cdot024$ for 812 reflections. In this molecule, the S—S [2·024 (1) Å] and S—C [1·802 (3), 1·825 (4) Å] distances are typical and the C—S—S—C dihedral angle [−80·5 (1)°] lies at the lower end of the range reported for related compounds. The acetyl and disulfide groups are coplanar to within ±0·01 Å. As a consequence, the molecule exhibits a short [3·078 (2) Å] intramolecular S···O contact.

Experimental. The title compound was obtained by treatment of acetyl sulfenyl chloride with benzyl mercaptan as described by Tsurugi & Nakabayashi (1959). Acetyl sulfenyl chloride was obtained by chlorination of acetyl sulfide (Bohme & Clement, 1952) while acetyl sulfide was prepared from thioacetic acid and acetyl chloride following the method of Bonner (1950). Colorless, diffraction quality crystals by evaporation from petroleum ether, D_m by flotation; crystal 0·15 × 0·25 × 0·45 mm; Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$ radiation; θ–2θ scan. Reciprocal-lattice symmetry and systematic absences consistent with space groups $P2_1$ and $P2_1/m$; $P2_1$ from successful solution of structure. Cell constants from setting angles of 25 reflections with $10\cdot62 \le \theta \le 18\cdot13°$; data corrected for Lorentz, polarization and absorption (empirical) effects, transmission factors 0·98–1·00. Variation in intensity of three standard reflections ±1·8%; 939 unique reflections measured with $4 \le 2\theta \le 50°$; 812 with $I \ge 3\sigma(I)$ used in refinement. Data collected: $h, k, \pm l$ ($0 \le h \le 6$, $0 \le k \le 9$, $-12 \le l \le 12$).

Structure solved by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and difference Fourier techniques. Full-matrix least-squares refinement on F; non-H atoms anisotropic; H atoms isotropic; H-atom temperature factors not refined; secondary-extinction parameter refined [final $g = 7\cdot5\ (5) \times 10^{-6}$]; $w = 4F_o^2/[\sigma^2(F_o^2) + 0\cdot0016F_o^4]$. Final $R_F = 0\cdot024$, $wR_F = 0\cdot030$, $S = 1\cdot08$, $(\Delta/\sigma)_{\max} = 0\cdot12$, $\Delta\rho_{\max} = 0\cdot14$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs from Enraf–Nonius (1983) *SDP*.

Atomic parameters are given in Table 1, bond lengths and angles in Table 2. Fig. 1 shows the molecular structure and labelling scheme.*

Related literature. As part of a project to prepare and study novel copper persulfide complexes (John, Bharadwaj, Krogh-Jespersen, Potenza & Schugar, 1986), the title compound was prepared as a potential source of benzyl persulfide, $PhCH_2S_2^-$. The S—S and S—C distances are similar to those in dibenzoyldisulfane [PhC(O)SSC(O)Ph; S—S, 2·021 (2); S—C,

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

Anisotropic refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B(Å^2)
S(1)	−0·1391 (1)	0	0·26868 (6)	4·54 (1)
S(2)	0·0445 (1)	0·1699 (1)	0·36926 (7)	4·69 (1)
O	−0·3572 (4)	−0·0251 (3)	0·4822 (2)	5·46 (5)
C(1)	−0·3468 (5)	−0·0756 (4)	0·3778 (3)	3·99 (5)
C(2)	−0·4971 (5)	−0·2044 (5)	0·3207 (3)	5·40 (7)
C(3)	−0·1484 (6)	0·3475 (4)	0·3607 (3)	4·36 (6)
C(4)	−0·1769 (5)	0·4227 (4)	0·2335 (3)	3·62 (5)
C(5)	−0·0069 (5)	0·5237 (5)	0·1875 (3)	5·18 (7)
C(6)	−0·0358 (6)	0·5952 (5)	0·0717 (4)	7·20 (9)
C(7)	−0·2378 (8)	0·5640 (5)	−0·0001 (3)	7·26 (9)
C(8)	−0·4057 (6)	0·4648 (5)	0·0434 (3)	6·25 (9)
C(9)	−0·3757 (5)	0·3938 (4)	0·1598 (3)	4·59 (7)
H(21)	−0·464 (5)	−0·235 (5)	0·248 (3)	6·5
H(22)	−0·548 (4)	−0·272 (5)	0·378 (3)	6·5
H(23)	−0·633 (5)	−0·181 (5)	0·307 (3)	6·5
H(31)	−0·082 (5)	0·423 (5)	0·417 (2)	5·4
H(32)	−0·308 (4)	0·319 (5)	0·390 (2)	5·4
H(5)	0·126 (5)	0·545 (5)	0·242 (3)	6·4
H(6)	0·078 (5)	0·675 (6)	0·031 (3)	7·7
H(7)	−0·266 (6)	0·604 (6)	−0·074 (3)	8·6
H(8)	−0·555 (6)	0·443 (5)	−0·010 (4)	7·2
H(9)	−0·497 (4)	0·332 (4)	0·194 (2)	5·7

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44444 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1·811 (6), 1·827(6) Å; Rout, Seshasayee, Subrahmanyan & Aravamudan, 1983] and dibenzyl disulfide [S—S, 2·015 (3); S—C, 1·844 (12) Å; van Dijk & Visser, 1971] while the C(1)—S(1)—S(2)—C(3) dihedral angle [−80·5 (1)°] lies near the lower end of the range reported (78·6–101°; Rout *et al.*, 1983) for several similar compounds. The acetyl and disulfide groups are coplanar to within ±0·01 Å. As a consequence, there is a short intramolecular S(2)···O contact [3·078 (2) Å] similar to those reported for dibenzoyldisulfane.

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Table 2. *Selected bond lengths* (Å) *and angles* (°)

Numbers in parentheses are estimated standard deviations in the least-significant digits.

S(1)–S(2)	2·024 (1)	C(4)–C(5)	1·380 (5)
S(1)–C(1)	1·802 (3)	C(4)–C(9)	1·376 (4)
S(2)–C(3)	1·825 (4)	C(5)–C(6)	1·369 (6)
O–C(1)	1·191 (4)	C(6)–C(7)	1·383 (8)
C(1)–C(2)	1·476 (5)	C(7)–C(8)	1·352 (8)
C(3)–C(4)	1·491 (4)	C(8)–C(9)	1·375 (5)
S(2)–S(1)–C(1)	103·6 (1)	C(3)–C(4)–C(9)	120·4 (3)
S(1)–S(2)–C(3)	102·8 (1)	C(5)–C(4)–C(9)	118·6 (3)
S(1)–C(1)–O	122·8 (3)	C(4)–C(5)–C(6)	120·9 (4)
S(1)–C(1)–C(2)	111·6 (2)	C(5)–C(6)–C(7)	119·2 (4)
O–C(1)–C(2)	125·7 (3)	C(6)–C(7)–C(8)	120·7 (4)
S(2)–C(3)–C(4)	114·9 (2)	C(7)–C(8)–C(9)	119·9 (4)
C(3)–C(4)–C(5)	121·1 (3)	C(4)–C(9)–C(8)	120·8 (4)

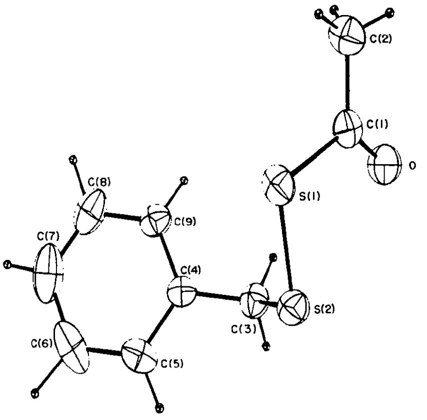

Fig. 1. View of the title compound showing the atom-numbering scheme.

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4-Methyl-3-{(*Z*)-methyl[(*E*)-dimethyl(phenyl)silyl]methylidene}azetidin-2-one: an α-Alkylidene-β-lactam

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Abstract. $C_{14}H_{19}NOSi$, $M_r = 245{\cdot}43$, orthorhombic, *Pbca*, $a = 8{\cdot}215\ (3)$, $b = 10{\cdot}551\ (4)$, $c = 32{\cdot}795\ (14)$ Å, $V = 2842\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}15$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 1{\cdot}14$ cm^{-1}, $F(000) = 1056$, $T = 295$ K, final $R = 0{\cdot}056$ for 1502 observed reflections. The β-lactam ring and alkylidene moiety are nearly coplanar. The methyl group is in the axial conformation with respect to the β-lactam ring.

Experimental. The title compound (I) was prepared by the procedure described previously (Buynak, Rao,

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