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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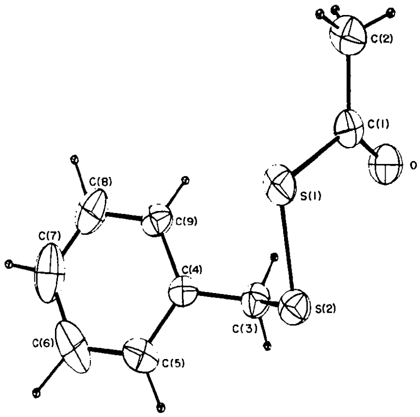

Fig. 1. View of the title compound showing the atom-numbering scheme.

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)

References

Bohme, H. & Clement, M. (1952). *Ann. Chem.* **576**, 61–69.
Bonner, W. A. (1950). *J. Am. Chem. Soc.* **72**, 4270–4271.
Dijk, B. van & Visser, G. J. (1971). *Acta Cryst.* B**27**, 846.
Enraf–Nonius (1983). *Structure Determination Package*. Enraf–Nonius, Delft, Holland.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
John, E., Bharadwaj, P. K., Krogh-Jespersen, K., Potenza, J. A. & Schugar, H. J. (1986). *J. Am. Chem. Soc.* **108**, 5015–5017.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Rout, G. C., Seshasayee, M., Subrahmanyan, T. & Aravamudan, G. (1983). *Acta Cryst.* C**39**, 1387–1389.
Tsurugi, J. & Nakabayashi, T. (1959). *J. Org. Chem.* **24**, 807–810.

Acta Cryst. (1988). C**44**, 391–393

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}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 1·14 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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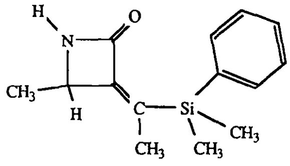
Chandrasekaran & Haley, 1985). Colorless cubic crystals of (I) were grown by the slow evaporation of a solution containing hexane and dichloromethane. A single crystal ($0{\cdot}40 \times 0{\cdot}60 \times 0{\cdot}24$ mm) was mounted on a goniometer head with an epoxy resin; unit-cell parameters by least-squares fit of 15 reflections in the range $10 < 2\theta < 25°$, space group *Pbca* (space group No. 61) from systematic absences; automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, variable scan rate (3·0–14·7° min^{-1}, depending on intensity), 3026 measured reflections, 2521 independent reflections in the range $3 < 2\theta < 50°$, $R_{\text{int}} = 0{\cdot}005$, *hkl* range h 0→9, k 0→12, l 0→38, 1502 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; 3 standard reflections remeasured after every 100 reflections did not show any significant change in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic; H atoms located in difference Fourier maps; H-atom isotropic temperature factors were included in the refinement and H-atom positional parameters were constrained to maintain the expected geometries for $-CH_3$, aliphatic $-CH$ and aromatic $-CH$ groups. $w = 1/(\sigma^2F + 0{\cdot}003621F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}056$ and $wR = 0{\cdot}055$ for 1502 observed reflections, $S = 2{\cdot}12$, $(\Delta/\sigma)_{\max} = 0{\cdot}02$, $\Delta\rho_{(\max,\min)} = 0{\cdot}31$, $-0{\cdot}21$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N, O and Si, and real and imaginary parts of the dispersion corrections for Si were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1.* Selected bond lengths, angles and torsion angles are given in Table 2. Si is nearly coplanar with the β-lactam ring. The atoms bonding to Si show a tetrahedral configuration. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1; the packing of the molecule is shown in the unit cell in Fig. 2.



(I)

Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-hydrogen atoms (e.s.d.'s in parentheses)*

	x	*y*	*z*	U_{eq}/U (Å^2)
Si	0·51477 (12)	0·20628 (10)	0·13138 (3)	0·0469 (2)
O	0·5838 (3)	0·4171 (3)	0·04958 (8)	0·0582 (7)
N	0·3396 (4)	0·3970 (3)	0·01250 (9)	0·0658 (10)
C(1)	0·4480 (5)	0·3743 (4)	0·04287 (11)	0·0507 (10)
C(2)	0·2158 (4)	0·3068 (4)	0·02719 (12)	0·0601 (11)
C(3)	0·3348 (4)	0·2806 (4)	0·06224 (11)	0·0453 (9)
C(4)	0·3348 (4)	0·2082 (3)	0·09502 (11)	0·0453 (8)
C(5)	0·4714 (4)	0·0831 (4)	0·17105 (11)	0·0520 (10)
C(6)	0·3889 (6)	0·1125 (5)	0·20670 (13)	0·0730 (13)
C(7)	0·3507 (7)	0·0200 (5)	0·23534 (14)	0·0891 (16)
C(8)	0·3983 (7)	−0·1020 (6)	0·22924 (16)	0·0867 (16)
C(9)	0·4806 (6)	−0·1340 (5)	0·19487 (15)	0·0848 (15)
C(10)	0·5161 (5)	−0·0424 (4)	0·16567 (13)	0·0671 (11)
C(11)	0·0544 (5)	0·3665 (5)	0·03790 (14)	0·0799 (14)
C(12)	0·1897 (5)	0·1246 (4)	0·10458 (14)	0·0620 (11)
C(13)	0·5340 (5)	0·3638 (4)	0·15548 (12)	0·0667 (12)
C(14)	0·7040 (5)	0·1636 (5)	0·10324 (14)	0·0712 (13)

* The standard deviations of U_{eq} values were calculated according to Schomaker & Marsh (1983).

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

Si–C(4)	1·901 (3)	N–C(2)	1·474 (5)
Si–C(5)	1·874 (4)	C(1)–C(3)	1·498 (5)
Si–C(13)	1·847 (4)	C(2)–C(3)	1·533 (5)
Si–C(14)	1·864 (4)	C(2)–C(11)	1·509 (6)
O–C(1)	1·223 (5)	C(3)–C(4)	1·319 (5)
N–C(1)	1·358 (5)	C(4)–C(12)	1·516 (5)
C(1)–N–C(2)	95·7 (3)	C(3)–C(2)–C(11)	117·5 (3)
O–C(1)–N	131·7 (4)	C(1)–C(3)–C(2)	87·7 (3)
O–C(1)–C(3)	137·2 (4)	C(1)–C(3)–C(4)	136·6 (3)
N–C(1)–C(3)	91·1 (3)	C(2)–C(3)–C(4)	135·6 (3)
N–C(2)–C(3)	85·5 (3)	Si–C(4)–C(3)	121·2 (3)
N–C(2)–C(11)	114·4 (3)	Si–C(4)–C(12)	118·4 (2)
		C(3)–C(4)–C(12)	120·4 (3)
C(3)–C(4)–Si–C(5)	−175·8 (3)		
C(3)–C(4)–Si–C(13)	65·1 (3)		
C(3)–C(4)–Si–C(14)	−56·8 (3)		
C(12)–C(4)–Si–C(5)	4·5 (3)		
C(12)–C(4)–Si–C(13)	−114·7 (3)		
C(12)–C(4)–Si–C(14)	123·5 (3)		

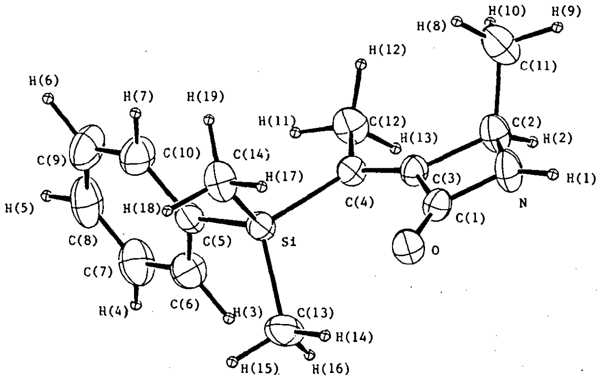


Fig. 1. *ORTEP* drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

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* Anisotropic temperature factors, H parameters, deviations from a plane through the β-lactam ring and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44460 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

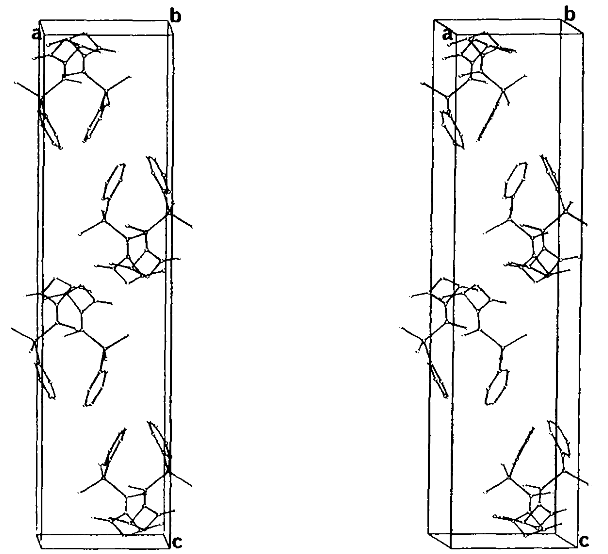

Fig. 2. Stereoscopic drawing showing the packing of molecules in the unit cell. H atoms are removed for clarity.

Related literature. α-Alkylidene-β-lactams have recently assumed importance as β-lactamase inhibitors (Chen, Chang, Hedberg, Guarino, Welch, Kiessling, Retsema, Haskell, Anderson, Manousos & Barrett, 1978). β-Silylenones are valuable synthetic intermediates for the synthesis of α,β-unsaturated ketones (Fleming & Perry, 1981).

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References

Buynak, J. D., Rao, M. N., Chandrasekaran, R. Y. & Haley, E. (1985). *Tetrahedron Lett.* **26**, 5001–5004.

Chen, Y. L., Chang, C.-W., Hedberg, K., Guarino, K., Welch, W. M., Kiessling, L., Retsema, J. A., Haskell, S. L., Anderson, M., Manousos, M. & Barrett, J. F. (1978). *J. Antibiot.* **40**, 1–20.

Fleming, I. & Perry, D. A. (1981). *Tetrahedron,* **37**, 4027–4034.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Schomaker, V. & Marsh, R. E. (1983). *Acta Cryst.* A**39**, 819–820.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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7-Chloro-6-oxobicyclo[3.2.0]hept-2-en-5-yl 2,4,6-Trimethylbenzoate

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Abstract. $C_{17}H_{17}ClO_3$ (1), $M_r = 304\cdot78$, monoclinic, $P2_1/n$, $a = 11\cdot795$ (2), $b = 7\cdot649$ (1), $c = 17\cdot827$ (2) Å, $\beta = 104\cdot57$ (1)°, $V = 1556\cdot6$ (3) Å^3, $Z = 4$, $D_x = 1\cdot300$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 2\cdot49$ cm^{-1}, $F(000) = 640$, $T = 293$ K, $R = 0\cdot0521$ for 1883 unique reflections. The cyclobutane ring is folded by 3·6 (3)° along C(1)—C(6). A least-squares plane (r.m.s.d. 0·012 Å) through this ring makes an angle of 64·5 (6)° with a plane through the cyclopentene ring (r.m.s.d. 0·012 Å). Torsion angles around the five-membered ring range from −3·0 (6) to 3·2 (4)°. The plane of the phenyl ring (r.m.s.d. 0·003 Å) and the C(8)C(9)O(2)O(3) plane (r.m.s.d. 0·003 Å) make an angle of 81·4 (6)°.

Experimental. Colorless, transparent crystals of (1) from chloroform, 0·68 × 0·53 × 0·18 mm; Nicolet $R3m/\mu$ update of $P2_1$ diffractometer; data collected Wyckoff mode ($3 \leq 2\theta \leq 50°$; 2θ fixed, ω varied), scan rate 4 – 29·3° min $^{-1}$, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($25\cdot01 \leq 2\theta \leq 40\cdot42°$), angles measured by a centering routine; systematic absences ($h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$) consistent with space group $P2_1/n$, Laue symmetry $2/m$, monitored reflections ($12\bar{9}$ and $13\bar{1}$) showed only

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