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1,1,3′-Trimethyl-3′-(trimethylsilyl)perhydroazetidino[1,2-*c*][1,3]oxazine-5-spiro-2′-oxiran-6-one, a Novel β-Lactam

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Abstract. $C_{13}H_{23}NO_3Si$, $M_r = 269{\cdot}41$, triclinic, $P\bar{1}$, $a = 10{\cdot}056\ (6)$, $b = 10{\cdot}597\ (5)$, $c = 7{\cdot}585\ (5)$ Å, $\alpha = 102{\cdot}73\ (5)$, $\beta = 89{\cdot}18\ (5)$, $\gamma = 102{\cdot}01\ (4)°$, $V = 770{\cdot}7\ (8)$ Å^3, $Z = 2$, $D_x = 1{\cdot}16$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 1·56 cm^{-1}, $F(000) = 292$, $T = 295$ K. Final $R = 0{\cdot}050$ for 1780 observed reflections. The X-ray structure shows the approximate planarity of the β-lactam ring, the chair conformation of the six-membered ring and the addition of perbenzoic acid oxygen from the least hindered side. The dihedral angle between the β-lactam plane and the three-membered ring is 88·4 (2)°.

Experimental. The title compound (I) was prepared by *m*-chloroperoxybenzoic acid oxidation of the corresponding alkene. Crystals of (I) are colorless rhombohedral plates; crystal dimensions 0·40 × 0·60 × 0·24 mm; unit-cell parameters obtained by least-squares refinement of 15 reflections in the range $10 < 2\theta < 25°$, cell checked for higher symmetry alternatives using *XCELL* (Sheldrick, 1988), 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), 2287 measured reflections, 2026 independent reflections in the range $3 < 2\theta < 45°$, $R_{int} = 0{\cdot}005$, *hkl* range h −10→10, k −11→11, l 0→8, 1780 observed reflections remeasured after every 100 reflections did not

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

	x	y	z	U_{eq}(Å^2)†
Si	0·2790 (1)	0·2254 (1)	0·3681 (1)	0·0481 (2)
C(2)	0·2570 (3)	0·1319 (3)	0·1233 (3)	0·0481 (6)
C(3)	0·2969 (2)	0·0040 (2)	0·0580 (3)	0·0412 (5)
C(4)	0·3201 (2)	−0·1042 (2)	0·1517 (3)	0·0400 (5)
C(5)	0·2290 (3)	−0·1383 (3)	0·3023 (3)	0·0480 (6)
C(6)	0·2056 (3)	−0·2851 (3)	0·2902 (4)	0·0577 (7)
O(7)	0·1461 (2)	−0·3595 (2)	0·1178 (3)	0·0577 (5)
C(8)	0·2246 (3)	−0·3392 (3)	−0·0370 (4)	0·0511 (6)
N(9)	0·2693 (2)	−0·1978 (2)	−0·0196 (3)	0·0434 (5)
C(10)	0·2437 (3)	−0·1065 (3)	−0·1066 (3)	0·0511 (7)
C(11)	0·1215 (3)	0·1819 (3)	0·4900 (4)	0·0671 (8)
C(12)	0·3093 (4)	0·4041 (3)	0·3615 (5)	0·0733 (9)
C(13)	0·4282 (3)	0·1953 (3)	0·4790 (5)	0·0739 (9)
C(14)	0·1607 (4)	0·1691 (3)	0·0005 (4)	0·0712 (8)
O(15)	0·3916 (2)	0·1192 (2)	0·0420 (3)	0·0596 (5)
C(16)	0·3490 (3)	−0·4022 (3)	−0·0453 (5)	0·0745 (9)
C(17)	0·1291 (3)	−0·4020 (3)	−0·1984 (4)	0·0683 (9)
O(18)	0·1987 (3)	−0·1126 (2)	−0·2578 (3)	0·0804 (7)

† The standard deviations of the U_{eq}'s were calculated according to Schomaker & Marsh (1983).

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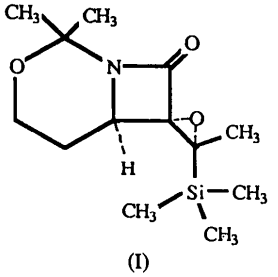
show any significant change (≃3%) in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Direct methods *SHELXS*86 (Sheldrick, 1986), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), non-hydrogen atoms anisotropic; H atoms located in difference Fourier maps, H with fixed isotropic thermal parameters and geometric constraints as primary, secondary, and tertiary hydrogens; $w = 1/(\sigma^2F + 0{\cdot}001054F^2)$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}050$, $wR = 0{\cdot}066$ and $S = 1{\cdot}95$ for 1780 observed reflections and 163 parameters, $(\Delta/\sigma)_{max} = 0{\cdot}14$, $\Delta\rho_{(max, min)} = 0{\cdot}27$, $-0{\cdot}26$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N, O, Si and real and imaginary parts of dispersion corrections for Si used were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters of the non-H atoms are given in Table 1.*



(I)

Related literature. The identification of the atoms and the configuration of the title compound are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Selected bond lengths, bond angles, and torsion angles with their standard deviations are given in Table 2. The β-lactam ring is approximately planar with atoms forming the least-squares plane not deviating more than 0·011 (3) Å. The six-membered ring has a chair conformation and the puckering parameters (Cremer & Pople, 1975) are $q_2 = 0{\cdot}125$ (3) Å, $q_3 = -0{\cdot}482$ (3) Å, $Q = 0{\cdot}498$ (3) Å, $\varphi_2 = 287$ (1)°, and $\theta = 14{\cdot}5$ (3)°. Furthermore, the six-membered ring is fused to the β-lactam in a *syn* conformation. As anticipated, the epoxidation has occurred from the least hindered, convex side of the molecule. Few examples of α-spiroepoxy-β-lactams could be found in the literature and no structural data are available. The crystal structure of a related α-alkylidene-β-lactam has been determined (Siriwardane, Chu & Buynak, 1988). Other related literature includes the preparation of β-lactams using different routes (Jephcote, John, Edwards, Luk & Williams, 1984; Sebti & Foucaud, 1984) and similar spiropenicillin derivatives have been found to be modest penicillinase inhibitors (Sheehan, Chacko, Commons, Lo, Ponzi & Schwabacher, 1984).

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

Si–C(2)	1·894 (3)	C(2)–Si–C(11)	110·1 (1)
Si–C(11)	1·849 (3)	C(2)–Si–C(12)	105·5 (1)
Si–C(12)	1·866 (4)	C(2)–Si–C(13)	111·3 (1)
Si–C(13)	1·848 (3)	C(11)–Si–C(12)	109·2 (2)
C(2)–C(3)	1·472 (4)	C(11)–Si–C(13)	111·7 (1)
C(2)–C(14)	1·519 (4)	C(12)–Si–C(13)	108·8 (2)
C(2)–O(15)	1·499 (4)	Si–C(2)–C(3)	123·1 (2)
C(3)–C(4)	1·533 (3)	Si–C(2)–C(14)	118·2 (2)
C(3)–C(10)	1·527 (4)	Si–C(2)–O(15)	111·2 (2)
C(3)–O(15)	1·411 (3)	C(3)–C(2)–O(15)	56·7 (2)
C(4)–C(5)	1·513 (4)	C(2)–C(3)–O(15)	62·6 (2)
C(4)–N(9)	1·474 (3)	C(6)–O(7)–C(8)	116·0 (2)
C(5)–C(6)	1·506 (4)	O(7)–C(8)–N(9)	107·7 (2)
C(6)–O(7)	1·436 (4)	C(4)–N(9)–C(8)	123·8 (2)
O(7)–C(8)	1·436 (4)	C(4)–N(9)–C(10)	96·4 (2)
C(8)–N(9)	1·449 (4)	C(8)–N(9)–C(10)	136·5 (2)
N(9)–C(10)	1·352 (4)	N(9)–C(10)–O(18)	133·7 (3)
C(10)–O(18)	1·222 (4)	C(2)–O(15)–C(3)	60·7 (2)

Si–C(2)–C(3)–C(4)	21·5 (4)
Si–C(2)–C(3)–C(10)	155·1 (2)
Si–C(2)–C(3)–O(15)	−95·1 (2)
Si–C(2)–O(15)–C(3)	116·4 (2)
C(2)–C(3)–C(4)–N(9)	147·4 (3)
C(2)–C(3)–C(10)–N(9)	−149·6 (3)
C(3)–C(4)–C(5)–C(6)	143·7 (2)
C(3)–C(4)–N(9)–C(8)	−163·9 (2)
C(3)–C(4)–N(9)–C(10)	−1·5 (2)
C(3)–C(10)–N(9)–C(4)	1·5 (2)
C(3)–C(10)–N(9)–C(8)	160·1 (3)
C(3)–O(15)–C(2)–C(14)	−107·3 (3)
C(4)–C(3)–C(10)–N(9)	−1·4 (2)
C(4)–C(3)–C(10)–O(18)	179·9 (4)

* Anisotropic temperature factors, bond lengths, bond angles, torsion angles, hydrogen parameters, least-squares planes, and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51493 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

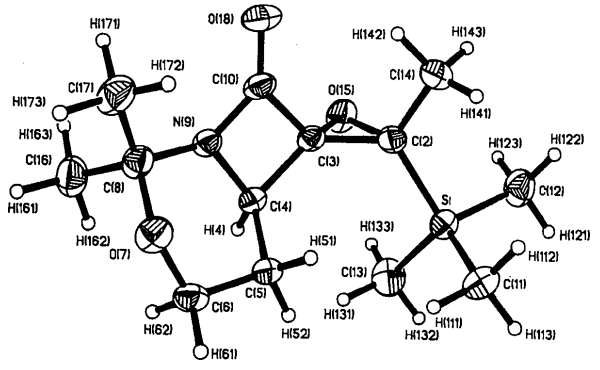

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. Hydrogen atoms are represented as spheres of arbitrary radii.

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Structure of a Substituted 2-Furylethylene Derivative

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Abstract. Methyl 2-cyano-3-(5-dimethylamino-2-furyl)-acrylate, $C_{11}H_{12}N_2O_3$, $M_r = 220{\cdot}2$, monoclinic, $P2_1/c$, $a = 10{\cdot}012\ (2)$, $b = 13{\cdot}820\ (3)$, $c = 8{\cdot}293\ (1)$ Å, $\beta = 106{\cdot}39\ (1)°$, $V = 1100{\cdot}8$ Å^3, $Z = 4$, $D_m = 1{\cdot}31$, $D_x = 1{\cdot}33$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}82$ mm^{-1}, $F(000) = 464$, $T = 293$ K. Final $R = 0{\cdot}065$ for 802 observed reflections. It is proved that in the reaction of 5-dimethylamino-2-furaldehyde with the methyl ester of cyanoacetic acid only the *E* isomer is produced. The molecules are nearly planar with dihedral angles between furan, methyl ester and dimethylamine planes ranging from 11·2 (2) to 23·6 (5)°. They are aligned to form stacks perpendicular to the *c* axis.

Experimental. Orange prismatic crystal with dimensions 0·43 × 0·09 × 0·08 mm; D_m by flotation in *n*-octane/CCl_4; monoclinic space group $P2_1/c$ (No. 14); lattice parameters and Bravais translation lattice found by program *UB* (Sivý, Sivý & Koreň, 1987). Intensities collected with Syntex $P2_1$ diffractometer, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{max} = 110°$; time per reflection *ca* 60 s, two standard reflections, variation 2·2%; 25 reflections with $11{\cdot}2 < 2\theta < 26{\cdot}6°$ used for refinement of lattice parameters; min. and max. transmission factors are the same and equal 0·8475 (absorption correction not applied); index range $0 \le h \le 8$, $0 \le k \le 14$, $-10 \le l \le 10$; 1621 reflections measured, 1393 unique, $R_{int} = 0{\cdot}03$ (238 reflections used), 802 reflections observed with $I > 1{\cdot}5\sigma(I)$, 591 unobserved. Because of strong secondary extinction the following reflections were excluded: 200, 102, 1,13,2, 294, 494, 594, 694, 794, and $\bar{3}66$. Data reduction carried out with program *XP*21 (Pavelčík, 1987). A furan ring, entered as a randomly oriented and randomly positioned molecular group to program *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), gave for $E > 1{\cdot}5$ (174 reflections used) the full structure except for the C(16) atom, which was found from a difference Fourier map.

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