

Fig. 2. The crystal structure of fluocinonide. Circles represent C atoms (open), O (solid) and F (dotted). The arrow points to the H bond.

and (II) involve the isopropylidenedioxy group bridging C(16) to C(17) on the *D* ring, and the 21-acetoxy group. These appear to have little effect on the conformation of the *D* ring which has the (13 β ,14 α) half-chair form in both molecules. The additional five-membered ring has a flattened O(17)-envelope conformation. In (I), the C(17) side chain as far as O(21) has an extended conformation similar to the side chain in (II) and cortisone acetate (Declercq, Germain & Van Meerssche, 1972*b*). The terminal acetoxy group is planar. Torsion angles for the C(21)–O(21) bond are

also similar in (I) and cortisone acetate, -77.1 (5) versus -82 (1) $^\circ$.

In the crystal structure of (I) (Fig. 2), molecules are H bonded in discrete pairs through the interaction O(11)–H \cdots O(3), where the O(11)–H bond length is 0.92 (6) Å, the O(11) \cdots O(3) and H \cdots O(3) distances are 2.79, 1.96 Å and the angles C(11)–O(11)–H, O–H \cdots O are 110 (4) and 150 $^\circ$ respectively. The shortest intermolecular distances involving fluorine atoms are F(6) \cdots O(3), 3.55 Å; F(9) \cdots C(6), 3.55 Å; F(9) \cdots C(7), 3.60 Å; F(6) \cdots F(9), 3.79 Å.

S-IC is grateful to the Korean Science and Engineering Foundation for a research fellowship. This work was supported in part by NIH Grant HL-20350.

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Acta Cryst. (1987). **C43**, 919–922

Structure and Stereochemistry of a Carbapenam Derivative

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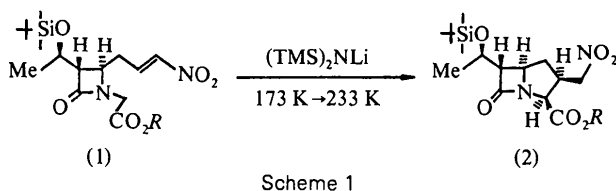
(Received 6 June 1986; accepted 26 November 1986)

Abstract. *p*-Nitrobenzyl 6-(1-*tert*-butyldimethylsiloxyethyl)-3-nitromethyl-7-oxo-1-azabicyclo[3.2.0]heptane-2-carboxylate, C₂₃H₃₃N₃O₈Si, *M_r* = 507.62, orthorhombic, *P*2₁2₁2₁, *a* = 7.7817 (12), *b* = 10.5608 (15), *c* = 33.062 (7) Å, *V* = 2717.1 Å³, *Z* = 4, *D_x* = 1.241 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, μ = 1.16 mm⁻¹, *F*(000) = 1080, *T* = 293 K, *R* = 0.070 for 2139 observed reflections. The absolute configuration was confirmed. In the β-lactam system, the five-membered ring has an envelope conformation and the four-membered ring is nearly planar. The *tert*-butyldimethylsilyl group shows disorder, and in the *p*-

nitrophenyl system the nitro group is tilted 17 $^\circ$ out of the plane of the aromatic ring.

Introduction. An operationally novel entry into the medically important carbapenam nucleus (Ratcliffe & Albers-Schönberg, 1982) was realized by an internal Michael-type ring closure of (1) to give the carbapenam structure (2) as shown in Scheme 1 (Hanessian & Désilets, 1987; Désilets, 1986). Given the bicyclic ring structure of the product, it was deemed of interest to ascertain the stereochemical course of the event, since a related cyclization in the penam series had most

surprisingly given the thermodynamically less stable (apparently the more hindered) product (Bedeschi, Bélanger-Gariépy, Hanessian & Brisse, 1986). The crystal-structure determination reported here is that of (2), where $R = -CH_2-C_6H_4-NO_2$.



Experimental. Crystal bounded by $\{001\}$, $\{010\}$, $\{100\}$, dimensions $0.05 \times 0.21 \times 0.48$ mm. Unit-cell dimensions from 25 well centered reflections in the range $40 \leq 2\theta \leq 60^\circ$. Nonius CAD-4 diffractometer, graphite-monochromatized $Cu K\alpha$ radiation, ω - 2θ scan, $\Delta\omega = (1.00 + 0.14 \tan\theta)^\circ$, $2\theta_{max} = 140.0^\circ$, $0 \leq h \leq 9$, $0 \leq k \leq 12$, $0 \leq l \leq 40$. Orientation monitored every 100 measurements, intensity checked every hour using seven standard reflections, largest intensity fluctuation of standards: 2.1%. No absorption correction. 2953 measured reflections of which 2139 with $I \geq 1.96\sigma(I)$ were retained for structure determination and refinement. Direct methods with *MULTAN80** (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Block-diagonal least-squares refinement based on F , anisotropic for all atoms but H. H atoms found on difference Fourier syntheses isotropically refined. Methyl H atoms were calculated (sp^3 hybridization, $C-H = 0.95 \text{ \AA}$) to give optimum fit with residual peaks observed in difference Fourier syntheses. H-atom coordinates of the methyl groups were recalculated after each cycle of refinement and given a $B_{iso} = 11.0 \text{ \AA}^2$.

The thermal motion was found to be very high for the *tert*-butyldimethylsilyl group. A substantial fraction of the residual electron density concentrated in this region suggested that this group could be disordered among two or more sites. Attempts to resolve this disorder could not yield coherent sets of coordinates. Furthermore there was no significant improvement in R , wR or S . Function minimized $\sum w(|F_o| - |F_c|)^2$. The real and imaginary parts of the anomalous dispersion of Si were included in the structure-factor calculations. Final† $R = 0.070$, $wR = 0.062$ and $S = 2.452$. Weights derived from the counting statistics, $w = 1/\{\sigma(I)^2 +$

Table 1. Final atomic coordinates and their e.s.d.'s ($\times 10^5$ for Si, $\times 10^4$ for O, N, C) and U_{eq} (\AA^2 , $\times 10^3$) for $C_{23}H_{33}N_3O_8Si$

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Si(1)	43800 (31)	95322 (21)	55395 (6)	85
O(1)	2019 (6)	8985 (4)	7290 (1)	77
O(2)	4132 (5)	9933 (3)	6012 (1)	62
O(3)	4945 (5)	7207 (3)	8104 (1)	54
O(4)	5429 (6)	9001 (3)	7767 (1)	80
O(5)	11144 (5)	6131 (4)	7641 (1)	89
O(6)	10289 (6)	6230 (5)	7024 (1)	117
O(7)	-2271 (6)	10375 (5)	8814 (2)	119
O(8)	-655 (7)	11784 (4)	9059 (2)	108
N(2)	10081 (7)	6389 (5)	7383 (3)	74
N(3)	-873 (6)	10780 (5)	8899 (1)	77
N(4)	4204 (6)	7587 (4)	7068 (1)	47
C(1)	7043 (8)	8051 (5)	6891 (2)	59
C(2)	7003 (8)	6945 (5)	7203 (2)	53
C(3)	5174 (7)	7032 (4)	7395 (1)	46
C(5)	5236 (7)	8007 (5)	6722 (1)	49
C(6)	4062 (7)	9164 (5)	6687 (1)	51
C(7)	3154 (8)	8641 (5)	7062 (2)	53
C(8)	3035 (8)	9377 (5)	6306 (2)	61
C(9)	1481 (9)	10220 (7)	6371 (2)	87
C(10)	5011 (21)	7888 (9)	5498 (3)	258
C(11)	2382 (14)	9506 (17)	5268 (3)	276
C(12)	6046 (16)	10561 (8)	5340 (2)	167
C(13)	5141 (18)	11937 (10)	5346 (3)	228
C(14)	6454 (16)	10258 (9)	4897 (2)	186
C(15)	7586 (11)	10659 (18)	5587 (3)	268
C(16)	5061 (8)	7911 (6)	8485 (2)	67
C(17)	3465 (8)	8637 (5)	8583 (1)	51
C(18)	1904 (9)	8297 (5)	8441 (2)	71
C(19)	447 (8)	8981 (7)	8542 (2)	84
C(20)	629 (7)	10004 (5)	8798 (2)	56
C(21)	2154 (8)	10338 (5)	8941 (2)	69
C(22)	3583 (8)	9650 (6)	8834 (2)	69
C(24)	8428 (8)	6981 (5)	7521 (2)	58
C(25)	5182 (7)	7891 (5)	7767 (2)	51

$\{0.02(I_{net})\}^2$. The refinement was concluded when $(\Delta/\sigma)_{max} = 0.30$, $(\Delta/\sigma)_{av} = 0.06$ and the residual electron density on final difference Fourier synthesis = $-0.22, +0.30 e \text{ \AA}^{-3}$. The set of atomic coordinates presented in Table 1 is in agreement with the known absolute configuration. For the other enantiomer, $R = 0.071$, $wR = 0.062$ and $S = 2.480$. The scattering curves for the non-H atoms were taken from Cromer & Mann (1968) and those for the H atoms from Stewart, Davidson & Simpson (1965). The anomalous-dispersion coefficients f' and f'' for Si were those given by Cromer & Liberman (1970).

Discussion. A stereopair showing the molecular conformation and the atomic numbering is given in Fig. 1. The bond distances, angles and torsion angles calculated from the final refined coordinates are given in Table 2.

The β -lactam ring system

The five-membered ring has the envelope conformation folded along C(2)–C(5). The fold angle, *i.e.* the dihedral angle between plane (1) [C(1), C(2), C(5)] and plane (2) [C(2), C(3), N(4), C(5)], is 36.4° . In *p*-nitrobenzyl 6-(1-*tert*-butyldimethylsiloxyethyl)-3-methylthio-3-nitromethyl-7-oxo-4-thia-1-azabicyclo-[3.2.0]heptane-2-carboxylate, which differs from the present compound by the replacement of S in the ring

* The programs used in this work are the same as those that were used in Bedeschi *et al.* (1986).

† Lists of structure factors, H-atom coordinates and anisotropic temperature factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43592 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

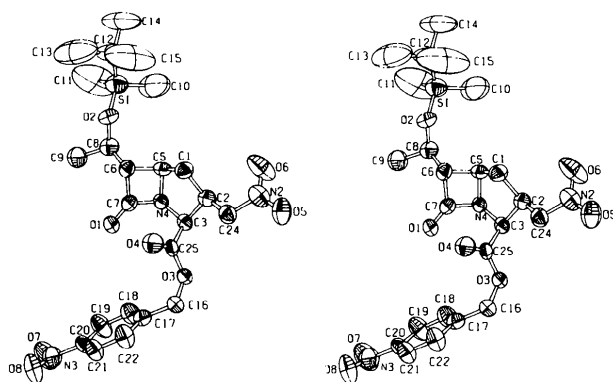


Fig. 1. Stereoview of the molecule including the atomic numbering.

Table 2. Bond distances (Å), bond angles (°) and torsion angles (°) with their *e.s.d.*'s for $C_{23}H_{33}N_3O_8Si$

Si(1) O(2)	1.630 (4)	C(6)–C(7)	1.530 (7)	N(3)–O(8)	1.197 (7)
Si(1) C(10)	1.810 (10)	C(6)–C(8)	1.509 (8)	N(3)–C(20)	1.466 (8)
Si(1) C(11)	1.796 (11)	C(7)–O(1)	1.217 (7)	C(16)–C(17)	1.495 (9)
Si(1) C(12)	1.816 (11)	C(8)–O(2)	1.421 (7)	C(17)–C(18)	1.351 (9)
C(1) C(2)	1.559 (7)	C(8)–C(9)	1.517 (9)	C(18)–C(19)	1.385 (9)
C(1) C(5)	1.514 (8)	C(12)–C(13)	1.615 (15)	C(19)–C(20)	1.380 (9)
C(2)–C(3)	1.561 (8)	C(12)–C(14)	1.532 (12)	C(20)–C(21)	1.325 (8)
C(2) C(24)	1.529 (8)	C(12)–C(15)	1.454 (14)	C(21)–C(22)	1.375 (8)
C(3) N(4)	1.443 (7)	N(2)–O(5)	1.219 (7)	C(22)–C(17)	1.357 (8)
C(3) C(25)	1.528 (7)	N(2)–O(6)	1.210 (7)	C(25)–O(3)	1.341 (6)
N(4) C(5)	1.466 (7)	N(2)–C(24)	1.501 (8)	C(25)–O(4)	1.188 (6)
N(4) C(7)	1.381 (7)	N(3)–O(7)	1.202 (7)	O(3)–C(16)	1.465 (6)
C(5) C(6)	1.530 (7)				
C(2) C(1)–C(5)	101.7 (4)	C(13)–C(12)–C(15)	106.8 (9)		
C(1) C(2)–C(3)	104.1 (4)	C(14)–C(12)–C(15)	112.4 (9)		
C(2)–C(3)–N(4)	101.3 (4)	C(1)–C(2)–C(24)	115.0 (5)		
C(3)–N(4)–C(5)	114.9 (4)	C(3)–C(2)–C(24)	112.4 (4)		
N(4) C(5)–C(1)	103.3 (4)	C(2)–C(24)–N(2)	113.7 (5)		
N(4) C(5)–C(6)	88.5 (4)	C(24)–N(2)–O(5)	117.5 (5)		
C(5) C(6)–C(7)	85.8 (4)	C(24)–N(2)–O(6)	118.1 (5)		
C(6)–C(7)–N(4)	91.7 (4)	O(5)–N(2)–O(6)	124.4 (5)		
C(7) N(4)–C(5)	94.0 (4)	C(2)–C(3)–C(25)	111.0 (4)		
C(3) N(4)–C(7)	130.4 (4)	N(4)–C(3)–C(25)	111.3 (4)		
C(1) C(5)–C(6)	123.9 (4)	C(3)–C(25)–O(3)	110.4 (4)		
N(4) C(7)–O(1)	131.4 (5)	C(3)–C(25)–O(4)	125.9 (5)		
C(6) C(7)–O(1)	136.8 (5)	O(3)–C(25)–O(4)	123.6 (5)		
C(5) C(6)–C(8)	119.9 (4)	C(25)–O(3)–C(16)	115.6 (4)		
C(7) C(6)–C(8)	119.1 (5)	O(3)–C(16)–C(17)	113.3 (5)		
C(6) C(8)–O(2)	108.3 (4)	C(16)–C(17)–C(18)	122.4 (5)		
C(6) C(8)–C(9)	113.1 (5)	C(17)–C(18)–C(19)	120.9 (6)		
O(2) C(8)–C(9)	109.5 (5)	C(18)–C(19)–C(20)	118.2 (6)		
Si(1) O(2)–C(8)	128.3 (3)	C(19)–C(20)–C(21)	121.3 (6)		
O(2) Si(1)–C(10)	110.7 (4)	C(20)–C(21)–C(22)	119.5 (6)		
O(2) Si(1)–C(11)	112.4 (5)	C(21)–C(22)–C(17)	121.3 (6)		
O(2) Si(1)–C(12)	106.1 (4)	C(22)–C(17)–C(18)	118.9 (5)		
C(10) Si(1)–C(11)	100.5 (6)	C(22)–C(17)–C(16)	118.7 (5)		
C(10) Si(1)–C(12)	110.7 (5)	N(3)–C(20)–C(19)	119.7 (5)		
C(11) Si(1)–C(12)	116.5 (5)	N(3)–C(20)–C(21)	118.9 (5)		
Si(1) C(12)–C(13)	102.9 (7)	O(7)–N(3)–C(20)	118.0 (5)		
Si(1) C(12)–C(14)	111.7 (7)	O(8)–N(3)–C(20)	118.9 (5)		
Si(1) C(12)–C(15)	115.3 (8)	O(7)–N(3)–O(8)	123.1 (5)		
C(13) C(12)–C(14)	106.9 (8)				
C(1)–C(2) C(3)–N(4)	28.3 (5)	C(8)–C(6)–C(7)–N(4)	119.6 (5)		
C(2) C(3)–N(4)–C(5)	–7.8 (5)	C(24)–C(2)–C(3)–C(25)	35.0 (6)		
C(3)–N(4)–C(5)–C(1)	–16.4 (5)	C(5)–C(6)–C(8)–O(2)	–80.6 (6)		
N(4) C(5)–C(1)–C(2)	32.9 (5)	Si(1)–O(2)–C(8)–C(6)	132.6 (4)		
C(5) C(1)–C(2)–C(3)	–38.4 (5)	Si(1)–O(2)–C(8)–C(9)	–103.8 (5)		
N(4) C(5)–C(6)–C(7)	2.3 (4)	O(2)–Si(1)–C(12)–C(13)	–67.5 (7)		
C(5) C(6)–C(7)–N(4)	–2.5 (4)	O(2)–Si(1)–C(12)–C(14)	178.2 (6)		
C(5) N(4)–C(7)–C(6)	2.6 (4)	O(2)–Si(1)–C(12)–C(15)	48.3 (9)		
C(7) N(4)–C(5)–C(6)	–2.6 (4)	C(12)–Si(1)–O(2)–C(8)	–177.0 (5)		
C(5) C(1)–C(2)–C(24)	–161.7 (5)	O(4)–C(25)–O(3)–C(16)	–1.8 (7)		
C(24) C(2)–C(3)–N(4)	153.3 (4)	N(4)–C(3)–C(25)–O(3)	137.4 (4)		
C(1) C(2)–C(3)–C(25)	–90.0 (5)	N(4)–C(3)–C(25)–O(4)	–45.4 (7)		
C(5) N(4)–C(3)–C(25)	110.3 (5)	C(2)–C(3)–C(25)–O(3)	–110.5 (5)		
C(3)–N(4)–C(7)–O(1)	–46.8 (7)	C(2)–C(3)–C(25)–O(4)	66.7 (7)		
C(5) C(6)–C(7)–O(1)	174.4 (7)	C(5)–C(6)–C(8)–C(9)	157.9 (5)		
C(1) C(5)–C(6)–C(8)	136.0 (5)				

system by C(1), the corresponding dihedral angle is 38.6° (Bedeschi *et al.*, 1986).

Nakayama, Kimura, Tanabe, Mizoguchi, Watanabe, Mori, Miyahara & Kawasaki (1981) evaluate the distortion of the β -lactam ring system by measuring the sum (ΣN) of the three N-atom bond angles and the distance (D) of the N atom to the plane of the three attached C atoms. In the present case the values $\Sigma N = 339.3^\circ$ and $D = 0.37 \text{ \AA}$ correspond well to those of the penam derivative of Bedeschi *et al.* (1986) where $\Sigma N = 338.3^\circ$ and $D = 0.39 \text{ \AA}$. These, however, differ significantly from the corresponding values in carbapenems such as carpetimycin (Nakayama *et al.*, 1981) or thienamycin (Albers-Schönberg, Arison, Hensens, Hirshfield, Hoogsteen, Kaczka, Rhodes, Kahan, Kahan, Ratcliffe, Walton, Ruswinkle, Morin & Christensen, 1978).

The four-membered ring is observed here to be less distorted, *i.e.* more nearly planar than in the other penam derivative. The four torsion angles describing the ring conformation have values around $\pm 2.5^\circ$ while they were $\pm 10^\circ$ in Bedeschi *et al.* (1986). In a least-squares-plane calculation including N(4), C(5), C(6) and C(7), one finds the deviations to be less than $0.020(5) \text{ \AA}$. Atom O(1) is $0.106(4) \text{ \AA}$ away from the above plane of atoms.

The *tert*-butyldimethylsilyl and *p*-nitrobenzyl groups

The *tert*-butyldimethylsilyl moiety makes an α junction at C(6) of the β -lactam. As indicated earlier, the refinement of this group of atoms led to very high thermal parameters indicative of a disordered orientation. A view of the unit-cell content (Fig. 2) reveals that these groups form slabs parallel to ab at $z = 0, \frac{1}{2}, \text{ etc.}$ The disorder may be explained by the fact that there are no contacts between methyl groups shorter than 4.00 \AA . A search, in the Cambridge Structural Database (CSD) (1986), for structures with the *tert*-butyldimethylsilyl fragment revealed 11 entries. In two of these, the *tert*-butyl group of atoms was disordered and could not be refined at all (Ferguson, Siew, Whalley & Yeates, 1982) or, if so, led to unrealistic values of the bond distances and angles (Wovkulich, Barcelos, Batcho, Sereno, Baggolini, Hennessy & Uskokovic, 1984). The Si–O bond distance and the Si–O–C bond angle agree well with the averages calculated from the nine reliable entries in

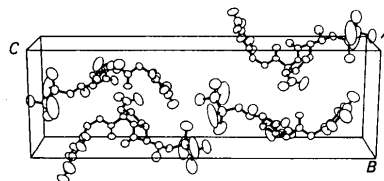


Fig. 2. Packing of the molecule within a unit cell.

the CSD. For Si—O one finds a range of 1.630 to 1.693 Å, and an average of 1.650 Å, while for the Si—O—C bond angle these quantities are 122.5 to 137.9°, with an average of 127.7°.

In this compound, the six ring atoms of the *p*-nitrobenzyl group are coplanar (plane 3). The plane of the NO₂ substituent is tilted about C(20)—N(3) by 17° with respect to plane (3). One may note (Fig. 1) a tendency for the principal axes of the thermal ellipsoids of C(18), C(19), C(21) and C(22) to be normal to plane (3) while C(17) and C(20) are slightly more isotropic. This suggests a slight oscillation of the ring about the C(17)—C(20) axis.

Stereochemistry

As in the case of the penam cyclization (Bedeschi *et al.*, 1986) kinetically controlled intramolecular Michael cyclization of (1) gives the *more* congested bicyclic product (2). While the precise reasons for this behavior are not clear, the outcome can be explained based on butane-type interactions in the transition state leading to (2), particularly since kinetic control was exercised as illustrated in Scheme 1.

Financial support of this research by NSERC and FCAR is gratefully acknowledged. We are also thankful to Mr M. J. Olivier for the X-ray intensity measurements.

Acta Cryst. (1987). C43, 922–926

Structures of Two Related Bufadienolides: Gamabufotalin (3β,11α,14-Trihydroxy-5β,14β-bufa-20,22-dienolide) and Arenobufagin (3β,11α,14-Trihydroxy-12-oxo-5β,14β-bufa-20,22-dienolide)

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(Received 9 September 1986; accepted 3 December 1986)

Abstract. Gamabufotalin, (1), C₂₄H₃₄O₅, *M_r* = 402.54, orthorhombic, *P*2₁2₁2₁, *a* = 7.850 (1), *b* = 14.766 (1),

c = 17.836 (1) Å, *V* = 2067.5 (3) Å³, *Z* = 4, *D_x* = 1.293 Mg m⁻³, λ(Cu Kα) = 1.54184 Å, μ = 0.68 mm⁻¹, *F*(000) = 872, *T* = 295 (1) K, *R* = 0.037 for 2284 unique observed reflections. The conformation

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