

Table 2. Selected geometric parameters (Å, °)

Si—N(1)	1.745 (5)	C(1)—C(2)	1.37 (1)
Si—C(6)	1.84 (1)	C(1)—C(5)	1.50 (1)
N(1)—N(2)	1.402 (7)	C(2)—C(3)	1.40 (1)
N(1)—C(1)	1.373 (9)	C(3)—C(4)	1.50 (1)
N(2)—C(3)	1.328 (8)		
N(1)—Si—C(6)	111.3 (2)	N(1)—C(1)—C(5)	123.4 (7)
N(1)—Si—N(1')	107.5 (2)	C(2)—C(1)—C(5)	129.3 (8)
Si—N(1)—N(2)	111.8 (4)	C(1)—C(2)—C(3)	105.3 (7)
Si—N(1)—C(1)	137.6 (5)	N(2)—C(3)—C(2)	112.9 (6)
N(2)—N(1)—C(1)	110.5 (6)	N(2)—C(3)—C(4)	119.2 (8)
N(1)—N(2)—C(3)	104.0 (5)	C(2)—C(3)—C(4)	127.8 (9)
N(1)—C(1)—C(2)	107.3 (7)		
C(6)—Si—N(1)—N(2)	-46.2 (4)	C(6)—Si—N(1)—C(1)	139.2 (6)

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An Oxazolidinone Derivative of D-Furanose

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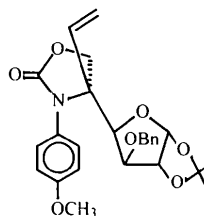
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Abstract

In the title compound, 4-(3-*O*-benzyl-1,2-*O*-isopropylidene- α -D-xylofuranos-5-yl)-3-(4-methoxyphenyl)-4-vinyloxazolidin-2-one, C₂₆H₂₉NO₇, the benzyloxy side chain extends axially with a *gauche-trans-gauche* conformation. The furanoid ring adopts a C(4)-*endo* envelope conformation. The isopropylidene ring has a half-chair conformation and the oxazolidinone ring has a minor O(5)-*exo* envelope conformation. Possible C—H \cdots O interactions are observed.

Comment

The crystal structure analysis of the title compound (I) was undertaken as part of our research programme on the synthesis of a novel immunosuppressant ISP-1 (Rama Rao, Gurjar, Rama Devi & Ravikumar, 1993). Bond lengths and angles (Table 2) are generally close to normal (Allen *et al.*, 1987), except those involving the disordered C(7) atom. The benzyloxy side chain extends axially with a *gauche-trans-gauche* conformation [C(2)—C(3)—O(3)—C(20) 84.7 (5), C(3)—O(3)—C(20)—C(21) -179.4 (4), O(3)—C(20)—C(21)—C(22) 100.4 (6) $^\circ$]. This side chain takes an *anti* orientation with respect to the isopropylidene ring [O(2)—C(2)—C(3)—O(3) -157.0 (4) $^\circ$].



(I)

The furanoid ring adopts a C(4)-*endo* envelope conformation with asymmetry parameter $\Delta C_s[C(4)] = 4.63^\circ$ (Duax & Norton, 1975). The C(4) atom

The refinement was by full-matrix least squares on *F*. Some H atoms were located from difference Fourier maps; others were placed at calculated positions (C—H 0.95 Å). H-atom displacement parameters were set at $B = 1.3(B_{eq}$ of the parent atom). H-atom parameters were not refined.

The structure was solved by direct methods (MULTAN11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier techniques. Molecular graphics were obtained using ORTEPII (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BK1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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deviates by -0.55 \AA from the plane through the other four atoms [C(1), C(2), C(3) and O(4)] of the ring. The dihedral angle between the plane defined by atoms C(1), C(2), C(3) and O(4) and the plane through C(1), C(2), O(2) and O(1) is $115.5(2)^\circ$. The isopropylidene ring has a half-chair conformation with asymmetry parameter $\Delta C_2[C(8), O(2)] = 1.07^\circ$. The oxazolidinone ring takes a minor O(5)-*exo* envelope conformation $\{\Delta C_5[O(5)] = 1.05^\circ\}$ with a deviation of 0.11 \AA from the plane through the other four atoms. There are possible C—H...O intra- and intermolecular interactions (Table 2) which, from geometrical considerations, may be attributed to C—H...O hydrogen bonds (Taylor & Kennard, 1982).

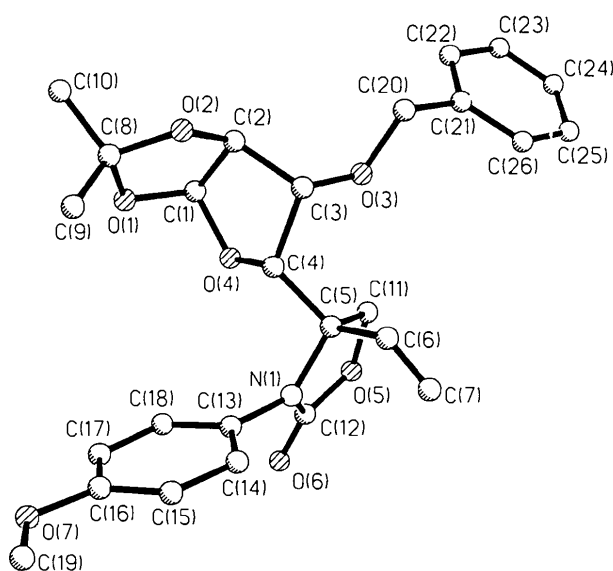


Fig. 1. A perspective view of the title molecule with the atom-labelling scheme.

Experimental

Crystal data

$C_{26}H_{29}NO_7$

$M_r = 467.5$

Orthorhombic

$P2_12_12_1$

$a = 6.937(2) \text{ \AA}$

$b = 15.320(2) \text{ \AA}$

$c = 23.423(3) \text{ \AA}$

$V = 2489.3(8) \text{ \AA}^3$

$Z = 4$

$D_x = 1.242 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.7107 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 9-22^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Needle

$0.28 \times 0.19 \times 0.12 \text{ mm}$

Pale white

Crystal source: aqueous solution

Data collection

Siemens R3m/V diffractometer

$\theta_{\max} = 22.5^\circ$

$h = 0 \rightarrow 7$

$\omega-2\theta$ scans

Absorption correction:

none

1918 measured reflections

1880 independent reflections

1415 observed reflections

$[I \geq 3\sigma(I)]$

$k = 0 \rightarrow 16$

$l = 0 \rightarrow 25$

2 standard reflections

monitored every 98

reflections

intensity decay: $< 1\%$

Refinement

Refinement on F

$R = 0.049$

$wR = 0.056$

$S = 0.923$

1415 reflections

316 parameters

$w = 1/[\sigma^2(F) + 0.0047F^2]$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.231 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.180 \text{ e \AA}^{-3}$

Atomic scattering factors

from *SHELXTL-Plus*

(Sheldrick, 1991)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	0.1386 (7)	0.8682 (3)	0.3663 (2)	0.126 (3)
O(2)	0.2737 (6)	0.9759 (2)	0.3140 (2)	0.080 (2)
O(3)	0.1773 (6)	0.8651 (2)	0.1800 (1)	0.060 (1)
O(4)	0.1674 (6)	0.7748 (2)	0.2907 (2)	0.062 (1)
O(5)	0.2029 (6)	0.5948 (2)	0.2046 (2)	0.072 (2)
O(6)	0.3057 (6)	0.5073 (3)	0.2750 (2)	0.082 (2)
O(7)	0.8442 (5)	0.6677 (3)	0.4653 (2)	0.074 (2)
N(1)	0.4350 (7)	0.6442 (3)	0.2602 (2)	0.049 (1)
C(1)	0.0781 (8)	0.8517 (3)	0.3104 (2)	0.070 (3)
C(2)	0.1563 (7)	0.9272 (3)	0.2752 (2)	0.064 (2)
C(3)	0.2836 (7)	0.8839 (3)	0.2297 (2)	0.052 (2)
C(4)	0.3387 (8)	0.7986 (3)	0.2601 (2)	0.049 (2)
C(5)	0.3964 (8)	0.7208 (3)	0.2226 (2)	0.047 (2)
C(6)	0.5692 (8)	0.7435 (3)	0.1859 (3)	0.087 (3)
C(7')†	0.6916 (10)	0.7940 (10)	0.1802 (7)	0.067 (6)
C(7'')‡	0.6968 (12)	0.7043 (11)	0.1627 (6)	0.097 (6)
C(8)	0.2329 (7)	0.9493 (3)	0.3712 (3)	0.089 (3)
C(9)	0.4189 (8)	0.9352 (6)	0.4012 (3)	0.149 (6)
C(10)	0.1107 (7)	1.0131 (6)	0.4011 (3)	0.122 (4)
C(11)	0.2246 (8)	0.6838 (4)	0.1874 (3)	0.064 (2)
C(12)	0.3168 (8)	0.5763 (4)	0.2493 (3)	0.058 (2)
C(13)	0.5452 (7)	0.6483 (3)	0.3114 (2)	0.043 (2)
C(14)	0.7423 (8)	0.6621 (4)	0.3110 (3)	0.052 (2)
C(15)	0.8462 (7)	0.6696 (4)	0.3614 (2)	0.054 (2)
C(16)	0.7548 (7)	0.6615 (3)	0.4132 (3)	0.054 (2)
C(17)	0.5590 (7)	0.6437 (4)	0.4133 (3)	0.074 (3)
C(18)	0.4565 (8)	0.6395 (4)	0.3638 (3)	0.068 (2)
C(19)	1.0449 (10)	0.6850 (6)	0.4664 (3)	0.084 (3)
C(20)	0.1641 (8)	0.9371 (4)	0.1419 (3)	0.102 (3)
C(21)	0.0472 (7)	0.9118 (4)	0.0906 (3)	0.074 (3)
C(22)	-0.1400 (7)	0.9366 (3)	0.0874 (3)	0.083 (4)
C(23)	-0.2486 (8)	0.9151 (4)	0.0393 (4)	0.063 (5)
C(24)	-0.1768 (8)	0.8680 (4)	-0.0014 (4)	0.066 (6)
C(25)	0.0069 (8)	0.8409 (3)	0.0003 (4)	0.077 (5)
C(26)	0.1203 (7)	0.8624 (4)	0.0470 (3)	0.080 (4)

† Atom included with occupancy factor 0.65.

‡ Atom included with occupancy factor 0.35.

Table 2. Selected geometric parameters (\AA , $^\circ$)

O(1)—C(1)	1.398 (7)	O(1)—C(8)	1.409 (7)
O(2)—C(2)	1.430 (7)	O(2)—C(8)	1.428 (8)
O(3)—C(3)	1.407 (6)	O(3)—C(20)	1.421 (7)
O(4)—C(1)	1.408 (7)	O(4)—C(4)	1.434 (7)
O(5)—C(11)	1.431 (7)	O(5)—C(12)	1.342 (7)
O(6)—C(12)	1.218 (7)	O(7)—C(16)	1.373 (7)
O(7)—C(19)	1.417 (8)	N(1)—C(5)	1.492 (7)
N(1)—C(12)	1.348 (7)	N(1)—C(13)	1.423 (7)

C(1)—O(1)—C(8)	112.0 (5)	C(2)—O(2)—C(8)	109.4 (4)
C(3)—O(3)—C(20)	113.3 (4)	C(1)—O(4)—C(4)	108.4 (4)
C(11)—O(5)—C(12)	111.0 (4)	C(16)—O(7)—C(19)	118.2 (5)
C(12)—N(1)—C(13)	121.3 (5)	C(5)—N(1)—C(13)	123.9 (4)
C(5)—N(1)—C(12)	112.7 (4)	O(1)—C(1)—O(4)	109.0 (4)
O(4)—C(1)—C(2)	107.6 (4)	O(1)—C(1)—C(2)	105.2 (4)
O(2)—C(2)—C(1)	104.9 (4)	O(2)—C(2)—C(3)	109.8 (4)
O(3)—C(3)—C(2)	111.2 (4)	O(3)—C(3)—C(4)	109.9 (4)
O(4)—C(4)—C(3)	103.9 (4)	O(4)—C(4)—C(5)	107.8 (4)
N(1)—C(5)—C(4)	108.6 (4)	N(1)—C(5)—C(11)	99.4 (4)
N(1)—C(5)—C(6)	111.9 (4)	O(1)—C(8)—O(2)	105.6 (4)
O(2)—C(8)—C(10)	111.8 (5)	O(2)—C(8)—C(9)	108.2 (5)
O(1)—C(8)—C(10)	110.9 (4)	O(1)—C(8)—C(9)	108.2 (5)
O(5)—C(11)—C(5)	106.2 (4)	O(6)—C(12)—N(1)	127.9 (6)
O(5)—C(12)—N(1)	110.0 (5)	O(5)—C(12)—O(6)	122.0 (5)
N(1)—C(13)—C(18)	120.4 (5)	N(1)—C(13)—C(14)	122.1 (5)
O(7)—C(16)—C(15)	124.8 (5)	O(7)—C(16)—C(17)	117.0 (5)
O(3)—C(20)—C(21)	109.8 (5)		

Furanoid ring

C(1)—C(2)—C(3)—C(4)	-25.5 (5)
C(2)—C(3)—C(4)—O(4)	37.2 (5)
C(1)—O(4)—C(4)—C(3)	-36.0 (5)
C(4)—O(4)—C(1)—C(2)	19.1 (5)
O(4)—C(1)—C(2)—C(3)	5.5 (5)

Isopropylidene ring

C(8)—O(2)—C(2)—C(1)	-14.7 (5)
C(2)—O(2)—C(8)—O(1)	17.7 (6)
C(1)—O(1)—C(8)—O(2)	-13.8 (6)
C(8)—O(1)—C(1)—C(2)	4.8 (6)
O(1)—C(1)—C(2)—O(2)	6.1 (5)

Oxazolidinone ring

N(1)—C(5)—C(11)—O(5)	-5.2 (5)
C(12)—O(5)—C(11)—C(5)	8.0 (6)
C(11)—O(5)—C(12)—N(1)	-7.5 (6)
C(5)—N(1)—C(12)—O(5)	3.8 (6)
C(12)—N(1)—C(5)—C(11)	1.1 (6)

Intra- and intermolecular hydrogen-bond geometry

D—H...A	H...A	D...A	D—H...A
C(11)—H...O(3)	2.381 (7)	2.802 (7)	106.1 (5)
C(11)—H...O(4)	2.394 (8)	2.821 (8)	106.5 (5)
C(18)—H...O(6)	2.795 (8)	3.086 (8)	98.4 (5)
C(3)—H...O(6 ^b)	2.484 (6)	3.421 (6)	165.3 (5)
C(15)—H...O(4 ^b)	2.509 (7)	3.211 (7)	129.9 (5)

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 + x, y, z$.

The vinyl side chain is disordered, resulting in two sites for the terminal C(7') and C(7'') atoms with site occupancies (constrained to unity) refining to 0.65 (1) and 0.35 (1), respectively. A $\Delta\rho$ map showed the positions of all H atoms except those attached to the atoms C(6) and C(7). They were included in the refinement at calculated positions riding on their bonded atoms with fixed isotropic displacement parameters. *SHELXTL-Plus* (Sheldrick, 1991) was used for data processing, structure solution and refinement. *PARST* (Nardelli, 1983) was used for the final interpretation of molecular geometry and crystal packing. All calculations were performed on a VAX 3100 workstation.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: VJ1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,5-Dinitro-4-(4-trifluoromethylphenoxy)-toluene, Conformationally Related to Thyroid Hormones*

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Abstract

The title compound, C₁₄H₉F₃N₂O₅, was synthesized as a crystalline intermediate in the synthetic route to the diiodo compound which is a potential thyroid-hormone analog. Although all the bond distances

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