methods (*MULTAN*11/82; Main *et al.*, 1982) and refined by full-matrix least-squares analysis. H atoms were generated in idealized positions and included in the structure-factor calculations, but not refined. All calculations were performed using *SDP* (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank the Hong Kong Research Grants Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(3*R*,4*S*,5*S*)-5-Benzyl-4-hydroxy-3-[1(*S*)-(1,1-dimethylethoxycarbonylamino)-2-phenylethyl]pyrrolidin-2-one

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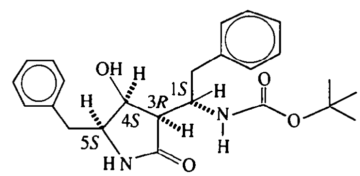
Abstract

The title compound, *tert*-butyl {1(*S*)-[(3*R*,4*S*,5*S*)-5-benzyl-4-hydroxy-2-oxo-3-pyrrolidinyl]-2-phenylethyl}-carbamate, $C_{24}H_{30}N_2O_4$, is an example of an all-*cis* trisubstituted pyrrolidin-2-one. The absolute configurations of the C atoms in the lactam ring were assigned to be 3*R*, 4*S* and 5*S* relative to the known chirality, 1*S*, of the C^{α} atom of the L-phenylalanine fragment in the compound. There is one intramolecular hydrogen bond between the pyrrolidin-2-one carbonyl O atom and the N—H group of the L-phenylalanine fragment [N—H···O 2.936 (5) Å].

Comment

The aldol addition of dilithiated methyl 3(*S*)-(1,1-dimethylethyloxycarbonylamino)-4-phenylbutanoate to *N*,*N*-dibenzyl-L-phenylalaninol resulted in the formation of two diastereomeric aldol products. *N*,*N*-Dibenzyl deprotection of the minor product gave the title compound, (I), as the sole product (Ettmayer, Hübner & Gstach, 1994).



(I)

The purpose of the structure analysis was the determination of the absolute configuration of the chiral centres in the pyrrolidinone ring relative to the known configuration of the C^{α} atom of the phenylalanine fragment

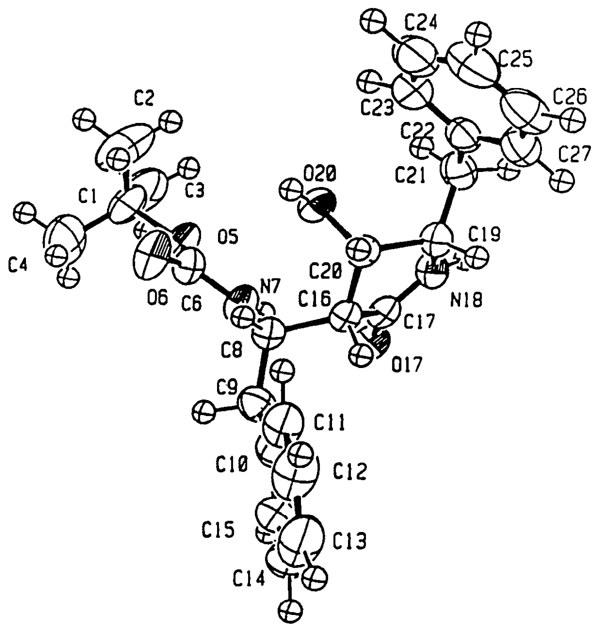


Fig. 1. *ORTEP* (Johnson, 1965) view of the title compound showing the atom-numbering scheme. Displacement ellipsoids of the non-H atoms are plotted at the 50% probability level; H atoms are represented by spheres of 0.15 Å radii.

in the synthesized compound. This stereochemical assignment demonstrates that aldol addition of β-aminobutanoates to aldehydes is governed by *ul*-1,2-induction (see Seebach & Estermann, 1987, 1988).

An *ORTEP* (Johnson, 1965) drawing of the molecular conformation showing the atomic numbering scheme is presented in Fig. 1. The pyrrolidinone ring has an envelope (*cs*) conformation with atom C(20) on the flap. The hydroxy group is in an axial position on the ring, the other two substituents being in pseudo-equatorial positions.

In the crystal there are one intramolecular and two intermolecular hydrogen bonds (Table 2 and Fig. 2). There are no other unusually close intermolecular contacts.

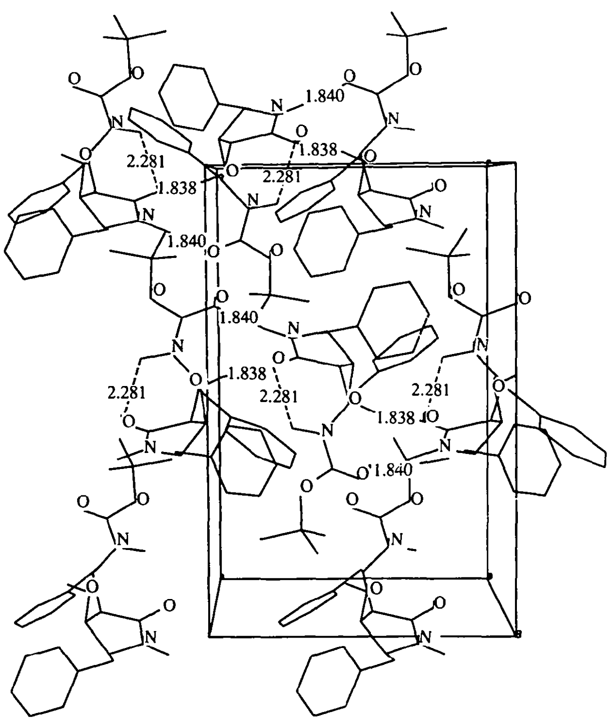


Fig. 2. *ORTEP* (Johnson, 1965) view of the crystal packing of the title compound. Hydrogen bonds are indicated by dashed lines.

Experimental

Crystals of the title compound were grown from an ethyl acetate solution.

Crystal data

$C_{24}H_{30}N_2O_4$
$M_r = 410.51$
Orthorhombic
$P2_12_12_1$
$a = 10.293$ (12) Å
$b = 14.207$ (32) Å
$c = 15.601$ (28) Å
$V = 2281.5$ Å^3
$Z = 4$
$D_x = 1.195$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 12 reflections
$\theta = 17$–$24°$
$\mu = 0.621$ mm^{-1}
$T = 295$ K
Prismatic
$0.3 \times 0.2 \times 0.1$ mm
Colourless

Data collection

Enraf–Nonius CAD-4*F* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (Gould & Smith, 1986)
$T_{min} = 0.85$, $T_{max} = 0.94$
2552 measured reflections
2462 independent reflections
2302 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.032$
$\theta_{max} = 70°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 6.3%

Refinement

Refinement on *F*
$R = 0.068$
$wR = 0.069$
$S = 3.113$
2302 reflections
271 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0007F^2]$
$(\Delta/\sigma)_{max} = 0.44$
$\Delta\rho_{max} = 0.241$ e Å^{-3}
$\Delta\rho_{min} = -0.349$ e Å^{-3}
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	0.3044 (4)	0.2614 (3)	0.2987 (2)	0.064 (2)
C(2)	0.3825 (7)	0.3506 (3)	0.3005 (4)	0.123 (5)
C(3)	0.1633 (5)	0.2791 (5)	0.3167 (4)	0.10 (3)
C(4)	0.3582 (7)	0.1893 (6)	0.3591 (3)	0.114 (4)
O(5)	0.3009 (2)	0.2212 (2)	0.21238 (16)	0.0580 (13)
C(6)	0.4089 (3)	0.1957 (3)	0.1698 (2)	0.0493 (16)
O(6)	0.5181 (2)	0.2084 (3)	0.19522 (17)	0.0702 (16)
N(7)	0.3757 (3)	0.1541 (2)	0.09622 (18)	0.0517 (14)
C(8)	0.4694 (3)	0.1279 (2)	0.03054 (19)	0.0441 (14)
C(9)	0.4725 (4)	0.0208 (2)	0.0235 (3)	0.0575 (19)
C(10)	0.5767 (4)	−0.0225 (3)	−0.0316 (2)	0.0555 (18)
C(11)	0.6880 (4)	0.0269 (3)	−0.0601 (3)	0.065 (2)
C(12)	0.7836 (5)	−0.0205 (5)	−0.1077 (3)	0.095 (3)
C(13)	0.7669 (7)	−0.1146 (5)	−0.1311 (4)	0.112 (4)
C(14)	0.6607 (7)	−0.1603 (4)	−0.1020 (4)	0.111 (4)
C(15)	0.5622 (6)	−0.1173 (3)	−0.0524 (3)	0.078 (3)
C(16)	0.4367 (3)	0.1759 (2)	−0.05582 (18)	0.0409 (14)
C(17)	0.2927 (3)	0.1828 (2)	−0.07686 (19)	0.0435 (14)
O(17)	0.2094 (2)	0.12480 (18)	−0.05431 (16)	0.0547 (12)
N(18)	0.2738 (3)	0.2596 (2)	−0.12219 (18)	0.0493 (14)
C(19)	0.3932 (3)	0.3138 (2)	−0.1388 (2)	0.0476 (16)
C(20)	0.4825 (3)	0.2790 (2)	−0.06659 (19)	0.0423 (14)
O(20)	0.4609 (2)	0.33052 (16)	0.01039 (16)	0.0515 (12)
C(21)	0.3684 (4)	0.4199 (3)	−0.1397 (3)	0.0580 (18)
C(22)	0.4898 (4)	0.4751 (2)	−0.1556 (2)	0.0535 (17)
C(23)	0.5505 (5)	0.5238 (3)	−0.0905 (3)	0.068 (2)
C(24)	0.6631 (6)	0.5730 (3)	−0.1060 (4)	0.089 (3)
C(25)	0.7149 (5)	0.5773 (3)	−0.1857 (4)	0.088 (3)
C(26)	0.6561 (5)	0.5292 (3)	−0.2509 (4)	0.087 (3)
C(27)	0.5425 (4)	0.4778 (3)	−0.2376 (3)	0.066 (2)

Table 2. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
N(7)—H(7)···O(17)	2.281 (5)	2.936 (5)	117.2 (3)
O(20)—H(20A)···O(17^{i})	1.838 (3)	2.722 (4)	166.9 (2)
N(18)—H(18)···O(6^{ii})	1.840 (4)	2.904 (5)	167.3 (3)

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

Programs used to solve and refine the structure were *SHELXS*86 (Sheldrick, 1985) and *SHELX*76 (Sheldrick, 1976), respectively.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1122). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Triacetyl Derivation of a Pyrano[3,2-*g*]-pteridine

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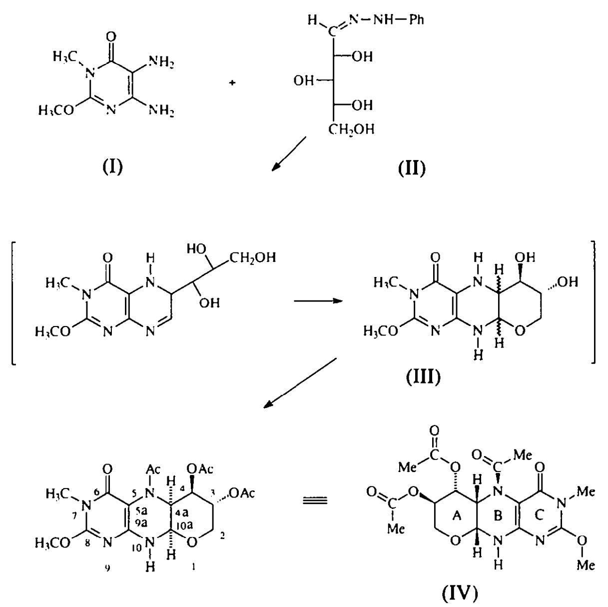
Abstract

The structure and stereochemistry of the title compound, (3*R*,4*R*,4a*S*,10a*R*)-5-acetyl-3,4,4a,5,6,7,10,10a-octahydro-8-methoxy-7-methyl-6-oxo-2*H*-pyrano[3,2-*g*]pteridin-3,4-diyl diacetate, $C_{17}H_{22}N_4O_8$, is established, with *cis* H atoms at the *A*/*B* ring fusion. Molecules are linked by N—H···O hydrogen bonds [N···O 2.816 (5) Å] to form infinite spirals about 2_1 screw axes.

Comment

The pteridine ring system has been the subject of many synthetic efforts because of its ubiquitous occurrence in nature (Pfleiderer, 1992). Among the most interesting naturally occurring pteridine systems are the 6-polyhydroxyalkyl derivatives such as biopterine and neopterine. The initial synthesis of this class of compounds was achieved by the reaction between a carbohydrate derivative and a conveniently substituted 5,6-diaminopyrimidine (Viscontini, Provenzale, Ohlgart & Mallevialle, 1970; Schircks, Bieri & Viscontini, 1985); a drawback was that these reactions usually yielded a complex mixture of many reaction products. Soyka, Pfleiderer & Prewo (1990) investigated this reaction type in more detail and found that under a nitrogen atmosphere at 333 K in acidic media, condensation between the diaminopyrimidine and the phenylhydrazone of the corresponding aldose led cleanly to pyrano[2,3-*g*]pteridine derivatives.

Using the conditions described by Soyka, Pfleiderer & Prewo (1990), we reacted 5,6-diamino-2-methoxy-3-methylpyrimidin-4(3*H*)-one, (I), with D-xylose phenylhydrazone, (II). The pteridine (III) so obtained was converted to its crystalline triacetyl derivative; this yielded crystals suitable for X-ray study, which identified the derivative as the title compound (IV), shown in Fig. 1 with the numbering scheme.



The bond lengths and angles are entirely in accord with those expected for this type of molecule (Orpen *et al.*, 1994) and serve to establish the structure