| | | | | |
|---|---|---|---|---|
| O(34) | −0.0612 (4) | 0.2726 (3) | 0.20315 (9) | 4.3 (2) |
| F(35) | −0.1661 (4) | 0.0704 (3) | 0.25983 (8) | 5.7 (2) |
| O(36) | −0.1613 (5) | −0.4648 (4) | 0.1548 (1) | 5.3 (2) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| N(1)—C(2) | 1.402 (5) | N(16)—C(17) | 1.375 (4) |
| N(1)—C(6) | 1.412 (5) | N(16)—C(25) | 1.463 (5) |
| N(1)—C(10) | 1.466 (7) | C(17)—C(18) | 1.357 (5) |
| C(2)—N(3) | 1.375 (5) | C(17)—N(22) | 1.358 (5) |
| C(2)—O(11) | 1.220 (5) | C(18)—C(19) | 1.414 (5) |
| N(3)—C(4) | 1.376 (5) | C(18)—N(20) | 1.374 (5) |
| N(3)—C(12) | 1.457 (7) | C(19)—O(26) | 1.227 (4) |
| C(4)—C(5) | 1.364 (5) | N(20)—C(21) | 1.329 (5) |
| C(4)—N(9) | 1.359 (5) | C(21)—N(22) | 1.337 (5) |
| C(5)—C(6) | 1.417 (6) | N(27)—C(28) | 1.349 (5) |
| C(5)—N(7) | 1.375 (5) | N(27)—C(32) | 1.370 (5) |
| C(6)—O(13) | 1.218 (5) | C(28)—N(29) | 1.374 (5) |
| N(7)—C(8) | 1.339 (6) | C(28)—O(33) | 1.227 (4) |
| C(8)—N(9) | 1.322 (5) | N(29)—C(30) | 1.385 (5) |
| N(14)—C(15) | 1.394 (5) | C(30)—C(31) | 1.434 (6) |
| N(14)—C(19) | 1.399 (4) | C(30)—O(34) | 1.219 (5) |
| N(14)—C(23) | 1.468 (5) | C(31)—C(32) | 1.328 (6) |
| C(15)—N(16) | 1.374 (5) | C(31)—F(35) | 1.346 (5) |
| C(15)—O(24) | 1.223 (4) | | |
| C(2)—N(1)—C(6) | 126.7 (3) | C(15)—N(16)—C(17) | 119.1 (3) |
| C(2)—N(1)—C(10) | 116.9 (4) | C(15)—N(16)—C(25) | 120.8 (3) |
| C(6)—N(1)—C(10) | 116.3 (4) | C(17)—N(16)—C(25) | 120.1 (3) |
| N(1)—C(2)—N(3) | 116.9 (3) | N(16)—C(17)—C(18) | 121.7 (3) |
| N(1)—C(2)—O(11) | 121.4 (4) | N(16)—C(17)—N(22) | 126.3 (3) |
| N(3)—C(2)—O(11) | 121.8 (4) | C(18)—C(17)—N(22) | 111.9 (3) |
| C(2)—N(3)—C(4) | 119.7 (3) | C(17)—C(18)—C(19) | 123.2 (3) |
| C(2)—N(3)—C(12) | 119.6 (4) | C(17)—C(18)—N(20) | 105.2 (3) |
| C(4)—N(3)—C(12) | 120.7 (4) | C(19)—C(18)—N(20) | 131.4 (3) |
| N(3)—C(4)—C(5) | 121.8 (3) | N(14)—C(19)—C(18) | 112.1 (3) |
| N(3)—C(4)—N(9) | 126.2 (3) | N(14)—C(19)—O(26) | 120.7 (3) |
| C(5)—C(4)—N(9) | 112.0 (3) | C(18)—C(19)—O(26) | 127.2 (3) |
| C(4)—C(5)—C(6) | 123.4 (3) | C(18)—N(20)—C(21) | 106.5 (3) |
| C(4)—C(5)—N(7) | 104.9 (3) | N(20)—C(21)—N(22) | 113.3 (4) |
| C(6)—C(5)—N(7) | 131.7 (3) | C(17)—N(22)—C(21) | 103.0 (3) |
| N(1)—C(6)—C(5) | 111.5 (3) | C(28)—N(27)—C(32) | 123.4 (3) |
| N(1)—C(6)—O(13) | 121.3 (4) | N(27)—C(28)—N(29) | 115.2 (3) |
| C(5)—C(6)—O(13) | 127.2 (3) | N(27)—C(28)—O(33) | 122.6 (3) |
| C(5)—N(7)—C(8) | 106.3 (3) | N(29)—C(28)—O(33) | 122.2 (3) |
| N(7)—C(8)—N(9) | 113.6 (4) | C(28)—N(29)—C(30) | 126.7 (3) |
| C(4)—N(9)—C(8) | 103.2 (3) | N(29)—C(30)—C(31) | 112.5 (4) |
| C(15)—N(14)—C(19) | 126.1 (3) | N(29)—C(30)—O(34) | 121.7 (4) |
| C(15)—N(14)—C(23) | 115.9 (3) | C(31)—C(30)—O(34) | 125.9 (3) |
| C(19)—N(14)—C(23) | 118.0 (3) | C(30)—C(31)—C(32) | 122.9 (3) |
| N(14)—C(15)—N(16) | 117.5 (3) | C(30)—C(31)—F(35) | 116.0 (4) |
| N(14)—C(15)—O(24) | 120.6 (3) | C(32)—C(31)—F(35) | 121.1 (4) |
| N(16)—C(15)—O(24) | 121.8 (3) | N(27)—C(32)—C(31) | 119.4 (4) |

Table 3. *Hydrogen-bonding geometry* (Å, °)

| *D*—H···*A* | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|
| N(7)—H(N7)···N(22) | 2.05 (4) | 2.903 (5) | 162 (2) |
| N(20)—H(N20)···O(33) | 1.87 (4) | 2.772 (4) | 170 (2) |
| N(27)—H(N27)···O(36) | 1.83 (4) | 2.702 (5) | 174 (2) |
| N(29)—H(N29)···O(26) | 1.90 (4) | 2.785 (4) | 172 (2) |
| O(36)—H(O36A)···O(34$^{i}$) | 1.99 (5) | 2.829 (4) | 168 (4) |
| O(36)—H(O36B)···N(9$^{ii}$) | 2.21 (5) | 2.980 (5) | 163 (6) |

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, -1-y, -z$.

All H atoms were located from a difference Fourier map. Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Program(s) used to solve structure: *MULTAN*88 (Main *et al.*, 1988). Program(s) used to refine structure: *KPPXRAY* (Taga, Masuda, Higashi & Iizuka, 1991) including a modified version of *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *KPPXRAY*. Software used to prepare material for publication: *EDCIF-J* (Osaki & Taga, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms, and torsion angles have been deposited with the IUCr (Reference: AS1172). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). C**51**, 1859–1861

# (2*R*,3*S*,4*S*)-3-Hydroxy-4-methyl-1-[(1*S*)-1-phenylethyl]pyrrolidine-2-methanol

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(*Received* 11 *October* 1994; *accepted* 26 *May* 1994)

## Abstract

The crystal structure determination of the title compound, $C_{14}H_{21}NO_2$, established the absolute configuration of the *N*-phenylethyl chain. The pyrrolidine ring

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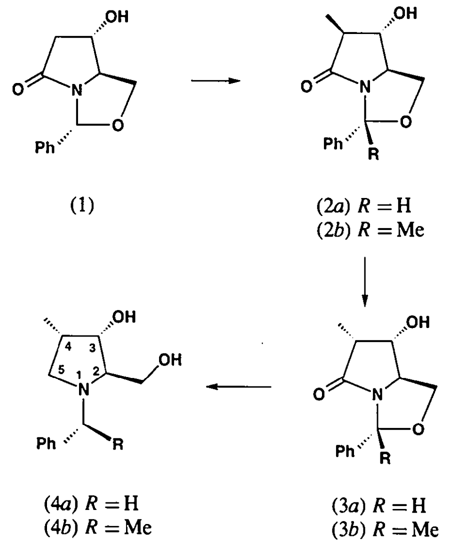
adopts an envelope conformation. The 3-hydroxy and 2-hydroxymethyl groups are in pseudo-axial positions, while the the *N*-(1-phenylethyl) and 4-methyl groups are pseudo-equatorial.

## Comment

Alkylation of bicyclic (2*R*,5*R*,6*R*)-6-hydroxy-2-phenyl-3-oxa-1-azabicyclo[3.3.0]octan-8-one, (1) (Herdeis & Hubmann, 1994), by methyl iodide, using a large excess of LDA-HMPA-MeI, gave rise not only to the monomethylated compound (2*a*), but also to a dimethylated compound, (2*b*)†. The 6,7-*cis* diastereoisomers (3*a*) and 3(*b*), obtained by deprotonation–protonation of (2), were reduced by $BH_3Me_2S$ to give the *N*-benzyl and *N*-(1-phenylethyl) pyrrolidines (4*a*) and (4*b*). The structure of (4*b*), the title compound, was established by X-ray analysis, which, in addition, allowed the determination of the absolute configuration of the *N*-(1-phenylethyl) substituent. The absolute configuration of (1) is known from that of the (*S*)-5-(hydroxymethyl)-2-pyrrolidinone precursor.



(1)

(2*a*) *R* = H
(2*b*) *R* = Me

(4*a*) *R* = H
(4*b*) *R* = Me

(3*a*) *R* = H
(3*b*) *R* = Me

The structure of (4*b*) is shown in Fig. 1. The pyrrolidine ring adopts an envelope conformation with atom C4 out of the plane of the other four atoms by 0.624 (2) Å. The hydroxy group O8—H and the hydroxymethyl group C6—O7—H are in pseudo-axial positions, while the *N*-(1-phenylethyl) and C9-methyl groups are pseudo-equatorial. In the crystal, two hydrogen bonds are formed between neighbouring molecules: O7—H···O8($x-1$, $y$, $z$) [O···O 2.726 (2), H···O 1.86 (2) Å, O—H···O 168 (2)°] and O8—H···O7($\frac{1}{2}+x$, $\frac{3}{2}-y$, $-z$) [O···O 2.742 (2), H···O 1.77 (2) Å, O—H···O 174 (2)°].

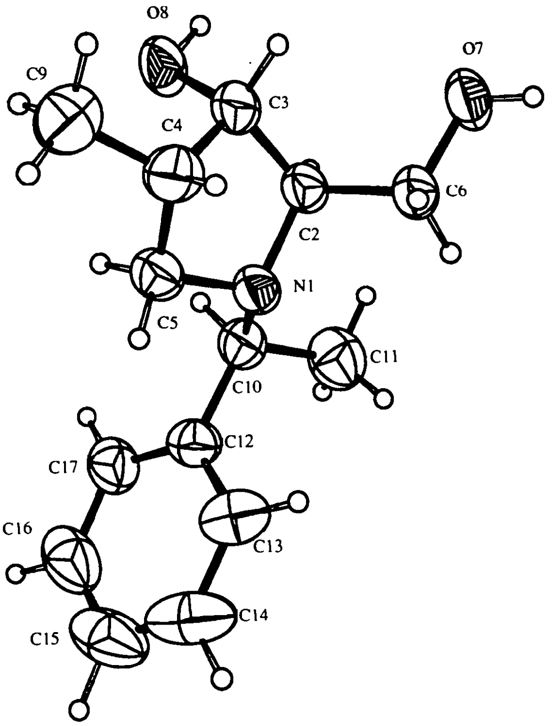


Fig. 1. Perspective view of the title compound showing 50% probability displacement ellipsoids.

## Experimental

Crystals of compound (4*a*) (m.p. 427–429 K) were obtained by recrystallization from dichloromethane.

*Crystal data*

$C_{14}H_{21}NO_2$
$M_r = 235.33$
Orthorhombic
$P2_12_12_1$
$a = 6.593\ (4)$ Å
$b = 10.237\ (5)$ Å
$c = 20.219\ (10)$ Å
$V = 1364.6\ (12)$ Å$^3$
$Z = 4$
$D_x = 1.15$ Mg m$^{-3}$

Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 14.9–20.0°$
$\mu = 0.57$ mm$^{-1}$
$T = 293$ K
Prism
0.40 × 0.23 × 0.16 mm
Colourless

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4724 measured reflections
2368 independent reflections
2244 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.027$
$\theta_{max} = 65.84°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 23$
3 standard reflections frequency: 60 min intensity decay: none

*Refinement*

Refinement on *F*
$R = 0.036$
$wR = 0.049$

$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.23$ e Å$^{-3}$

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† This observation differs from results previously described (Brunet-Griffart, 1995).

$S = 0.64$
2242 reflections
162 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0003F^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| N1 | 0.7732 (2) | 0.5662 (1) | 0.1626 (1) | 0.045 (1) |
| C2 | 0.6869 (2) | 0.6186 (2) | 0.1002 (1) | 0.047 (1) |
| C3 | 0.7931 (2) | 0.5466 (2) | 0.0444 (1) | 0.052 (1) |
| C4 | 0.8657 (2) | 0.4207 (2) | 0.0770 (1) | 0.053 (1) |
| C5 | 0.9303 (2) | 0.4708 (2) | 0.1441 (1) | 0.049 (1) |
| C6 | 0.4591 (2) | 0.5961 (2) | 0.1014 (1) | 0.053 (1) |
| O7 | 0.3676 (2) | 0.6608 (1) | 0.0468 (1) | 0.060 (1) |
| O8 | 0.9675 (2) | 0.6166 (1) | 0.0212 (1) | 0.062 (1) |
| C9 | 1.0233 (3) | 0.3422 (2) | 0.0396 (1) | 0.070 (2) |
| C10 | 0.8455 (2) | 0.6648 (2) | 0.2098 (1) | 0.049 (1) |
| C11 | 0.6774 (3) | 0.7594 (2) | 0.2301 (1) | 0.072 (2) |
| C12 | 0.9293 (2) | 0.5995 (1) | 0.2711 (1) | 0.050 (1) |
| C13 | 0.8126 (3) | 0.5117 (2) | 0.3070 (1) | 0.068 (2) |
| C14 | 0.8872 (5) | 0.4572 (2) | 0.3648 (1) | 0.089 (3) |
| C15 | 1.0751 (5) | 0.4897 (2) | 0.3873 (1) | 0.094 (3) |
| C16 | 1.1954 (4) | 0.5758 (3) | 0.3519 (1) | 0.084 (2) |
| C17 | 1.1210 (3) | 0.6302 (2) | 0.2936 (1) | 0.061 (2) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| N1—C2 | 1.484 (2) | C3—O8 | 1.434 (2) |
| N1—C5 | 1.471 (2) | C4—C5 | 1.513 (2) |
| N1—C10 | 1.468 (2) | C4—C9 | 1.515 (3) |
| C2—C3 | 1.519 (2) | C6—O7 | 1.421 (2) |
| C2—C6 | 1.520 (2) | C10—C11 | 1.528 (3) |
| C3—C4 | 1.525 (2) | C10—C12 | 1.513 (2) |
| C2—N1—C5 | 107.1 (1) | C3—C4—C9 | 116.6 (1) |
| C2—N1—C10 | 115.3 (1) | C5—C4—C9 | 115.8 (1) |
| C5—N1—C10 | 113.1 (1) | N1—C5—C4 | 104.8 (1) |
| N1—C2—C3 | 106.2 (1) | C2—C6—O7 | 109.7 (1) |
| N1—C2—C6 | 108.1 (1) | N1—C10—C11 | 112.0 (1) |
| C3—C2—C6 | 113.1 (1) | N1—C10—C12 | 110.3 (1) |
| C2—C3—C4 | 103.5 (1) | C11—C10—C12 | 108.9 (1) |
| C2—C3—O8 | 111.7 (1) | C10—C12—C13 | 120.8 (1) |
| C4—C3—O8 | 108.2 (1) | C10—C12—C17 | 120.3 (1) |
| C3—C4—C5 | 100.9 (1) | | |
| C5—N1—C2—C3 | 2.3 (1) | C4—C5—N1—C2 | −27.1 (1) |
| N1—C2—C3—C4 | 23.1 (1) | C2—N1—C10—C12 | 179.9 (2) |
| C2—C3—C4—C5 | −38.6 (1) | N1—C10—C12—C13 | −54.9 (2) |
| C3—C4—C5—N1 | 40.7 (1) | N1—C2—C6—O7 | 172.0 (2) |

The parameters for the H atoms bonded to the hydroxy groups O7 and O8 were refined. The isotropic displacement factors of H atoms bonded to C atoms were set equal to $1.1U_{\rm eq}$ of the bonded atom. Two reflections, 340 and 002, were omitted from the refinement.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1987). Data reduction: *NONIUS* (Riche, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *R3M* (Riche, 1983) and *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *ACTACIF* (Riche, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1230). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 1861–1864

# 9,9′-Thiobis(1,2,3,4,7,8-hexahydro-7-methyl-6*H*-pyrimido[1,6-*a*]pyrimidine-6,8-dione)

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## Abstract

The structure determination of $C_{16}H_{20}N_6O_4S$, has shown that the molecule consists of two fused-ring systems joined by an S atom. Each ring system is nearly planar except for one $sp^3$ C atom. The mean S—C distance [1.730 (3) Å] represents a bond order of 1.5. The bonding pattern of the pyrimidine-2,6-dione moiety is analysed in detail and compared with that of analogous compounds. The molecule is stabilized by two intramolecular hydrogen bonds, while the intermolecular packing is governed by van der Waals forces.

## Comment

As a part of our studies on purine and pyrimidine derivatives, we attempted to convert, by treatment with thionylchloride, 6-(γ-hydroxypropylamino)-