Refinement

Refinement on F
Final $R = 0.039$
$wR = 0.034$
891 reflections
155 parameters
All H-atom parameters refined, U(H) with a common value
$w = 2.0811/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) (P, O) and Cromer & Mann (1968) (C, H, N)

Data collection: Stoe *DIF4*. Cell refinement: Stoe *DIF4*. Data reduction: Stoe *REDU4*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985), option *TREF*. Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976).

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

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

	x	y	z	$U_{\rm eq}$
P	0.7918 (2)	0.2453 (1)	0.1453 (1)	0.0177 (6)
O1	0.7896 (5)	0.1255 (2)	0.1767 (2)	0.026 (2)
O2	0.9947 (5)	0.3039 (2)	0.2396 (3)	0.035 (2)
O3	0.7778 (4)	0.2604 (2)	0.0197 (2)	0.029 (2)
O4	0.5664 (5)	0.2987 (3)	0.1454 (3)	0.037 (3)
O5	0.4182 (5)	0.2209 (3)	0.3018 (3)	0.043 (3)
N1	0.9090 (6)	0.0815 (3)	0.4095 (3)	0.022 (3)
N2	0.9839 (7)	0.9607 (3)	0.1062 (3)	0.028 (3)
C1	0.8225 (8)	0.9225 (4)	−0.0127 (4)	0.028 (3)
C2	0.7699 (8)	−0.0141 (4)	0.4130 (4)	0.025 (4)
C3	1.2060 (9)	0.9927 (4)	0.1062 (4)	0.028 (3)
C4	1.1552 (7)	0.0542 (4)	0.4615 (4)	0.024 (3)

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

P—O3	1.511 (2)	N2—C3	1.479 (7)
P—O2	1.519 (2)	C1—C3$^{\rm ii}$	1.50 (1)
P—O1	1.524 (2)	O1—H2$^{\rm iii}$	1.89 (4)
P—O4	1.589 (3)	O1—H11	1.92 (6)
N1—C2	1.487 (7)	O2—H13$^{\rm iv}$	1.72 (4)
N1—C4	1.479 (6)	O2—H8$^{\rm v}$	1.79 (4)
C2—C4$^{\rm i}$	1.489 (5)	O3—H14$^{\rm vi}$	1.80 (3)
N2—C1	1.471 (9)	O3—H5$^{\rm vi}$	1.89 (6)
O2—P—O3	112.70 (3)	O1—P—O2	112.20 (3)
O1—P—O3	111.80 (3)	O4—P—O2	107.50 (3)
O4—P—O3	106.00 (3)	O1—P—O4	107.30 (3)

Symmetry code: (i) $2-x, -y, 1-z$; (ii) $2-x, 2-y, -z$; (iii) $x, y-1, z$; (iv) $1+x, y, z$; (v) $2-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vi) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

In order to synthesize a fluoro-aluminophosphate with a microporous framework, $(N_2C_4H_{12}).HPO_4.H_2O$ was prepared hydrothermally from a 1:2:2:5:80 mixture of Al_2O_3, P_2O_5, NH_4F, piperazine and water. The resultant product was heated at 443 K for 3 d in an autoclave under autogenous pressure then filtered, washed and finally dried in air. A fragment of a colorless crystal without definite faces was isolated and its quality tested with Laue photographs.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71053 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1011]

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(4*S*,5*S*)-4-Methyl-5-phenyl-1,3-oxazolidin-2-one

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Abstract

Ring closure of methyl (2*S*,3*R*)-*N*-[(3-hydroxy-3-phenyl)-2-propyl]carbamate by treatment with *p*-toluenesulfonyl chloride and pyridine leads to the title compound in which the oxazolidinone ring is puckered with C5 out of the plane by 21.56°.

Comment

In our studies of the stereoselective conversion of (-)-(1*R*,2*S*)-norephedrine into (+)-(1*S*,2*S*)-2-amino-1-phenylpropanol, we found that (2*S*,3*R*)-*N*-[(3-hydroxy-

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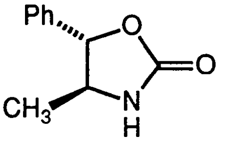
3-phenyl)-2-propyl]carbamate, derived from carbamoylation of (−)-norephedrine with methyl chloroformate and triethylamine, underwent ring closure when refluxed with *p*-toluenesulfonyl chloride and pyridine in methylene chloride to give the title compound (I); m.p. 388-392 K (recrystallized from methylene chloride), $[\alpha]_D^{22^\circ\mathrm{C}}$ = +8° (*c* = 0.375 mol dm^{-3}, $CHCl_3$) {literature m.p. 390.5-392 K, $[\alpha]_D^{26^\circ\mathrm{C}}$ = +25.9° (*c* = 0.34 mol dm^{-3}, $CHCl_3$) (Fodor, Stefanovsky & Kurtev, 1967)}. Thin-layer chromatography and ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR indicated the product to be a single pure compound. While our NMR data agreed with the reported values (Spassov, Stefanovsky, Kurtev & Fodor, 1972) and our melting point agreed with that reported by Fodor, Stefanovsky & Kurtev (1967), our specific rotation differed considerably from that reported by the latter. Moreover, two different pathways available for ring closure could have provided diastereomerically different products. It was, therefore, desirable to determine the structure of our product unequivocally by X-ray diffraction.



(I)

In either ring-closure mechanism, the stereochemistry at C2 would be unaltered, while cyclization involving attack by OH on the carbonyl with displacement of methanol would leave the stereochemistry at C5 similarly unaltered. This study proves that the reaction was accompanied by inversion at C5. Apparently, tosylation of the hydroxyl group is followed by intramolecular displacement of the tosylate by the carbonyl O atom, a process similar to that reported by Kano, Yokomatsu, Iwasawa & Shibuya (1987).

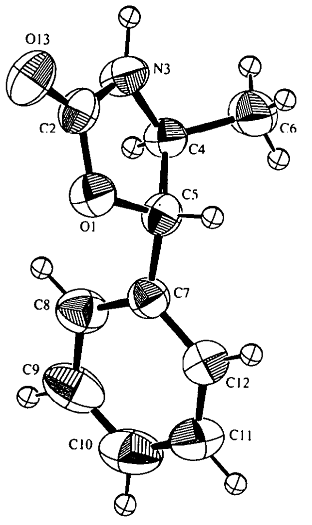


Fig. 1. Molecular configuration and atom-numbering scheme with thermal ellipsoids at the 50% probability level. H atoms are shown as unlabeled isotropic spheres of arbitrary radii.

The oxazolidinone ring is puckered, with C5 out of the O1—C2—N3—C4 plane by 21.56° (the dihedral angle between the least-squares planes formed by O1—C2—N3—C4 and O1—C5—C4). The N3—C4—C5 angle is somewhat smaller [100.0 (2)°] than the normal bond angle for cyclopentane [102-106° (Adams, Geise & Bartell, 1970)]. The torsion angle C6—C4—C5—C7 is −93.8 (3)°. A dihedral angle of 87.80° is formed by the phenyl-ring plane and the planar moiety of the oxazolidinone ring (O1, C2, N3 and C4).

Experimental

Crystal data

$C_{10}H_{11}NO_2$
M_r = 177.20
Monoclinic
$P2_1$
a = 5.950 (2) Å
b = 8.238 (1) Å
c = 9.952 (2) Å
β = 102.82 (2)°
V = 475.7 (2) Å^3
Z = 2
D_x = 1.237 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10.03-12.39°
μ = 0.081 mm^{-1}
T = 296 K
Equant
0.29 × 0.26 × 0.16 mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
988 measured reflections
899 independent reflections
746 observed reflections [$I > 3.0\sigma(I)$]

R_{int} = 0.010
$\theta_{\max}$ = 25°
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 9$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 100 reflections intensity variation: 0.3%

Refinement

Refinement on F
Final R = 0.029
wR = 0.036
S = 1.51
746 reflections
118 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max}$ = 0.0005
$\Delta\rho_{\max}$ = 0.10 e Å^{-3}
$\Delta\rho_{\min}$ = −0.11 e Å^{-3}

Extinction correction: Zachariasen type 2 Gaussian isotropic
Extinction coefficient: 0.47 (16) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985).

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å²)

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

	x	y	z	U_{eq}
O1	0.7028 (3)	0.1345	0.2978 (2)	0.0505 (1)
C2	0.8586 (4)	0.0572 (4)	0.3971 (3)	0.0473 (1)
N3	0.7758 (3)	−0.0860 (3)	0.4239 (2)	0.0540 (1)
C4	0.5611 (4)	−0.1265 (4)	0.3273 (2)	0.0457 (1)
C5	0.4877 (4)	0.0418 (4)	0.2706 (2)	0.0448 (1)
C6	0.3890 (4)	−0.2107 (5)	0.3927 (3)	0.0660 (1)
C7	0.3696 (4)	0.0488 (4)	0.1208 (2)	0.0461 (1)
C8	0.4690 (5)	−0.0169 (4)	0.0202 (3)	0.0613 (1)
C9	0.3508 (6)	−0.0141 (5)	−0.1163 (3)	0.0744 (1)
C10	0.1370 (7)	0.0537 (6)	−0.1525 (3)	0.0804 (1)
C11	0.0393 (5)	0.1204 (6)	−0.0538 (4)	0.0858 (1)
C12	0.1537 (4)	0.1188 (5)	0.0825 (3)	0.0645 (1)
O13	1.0419 (3)	0.1189 (3)	0.4493 (2)	0.0645 (1)

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.355 (3)	C5—C7	1.502 (3)
O1—C5	1.463 (3)	C7—C8	1.381 (4)
C2—N3	1.328 (4)	C7—C12	1.383 (4)
C2—O13	1.211 (3)	C8—C9	1.385 (4)
N3—C4	1.457 (3)	C9—C10	1.362 (5)
C4—C5	1.524 (4)	C10—C11	1.363 (5)
C4—C6	1.499 (4)	C11—C12	1.376 (4)
C2—O1—C5	108.6 (2)	C4—C5—C7	115.8 (2)
O1—C2—N3	109.9 (2)	C5—C7—C8	121.6 (2)
O1—C2—O13	121.4 (2)	C5—C7—C12	119.4 (2)
N3—C2—O13	128.7 (3)	C8—C7—C12	119.0 (2)
C2—N3—C4	112.4 (2)	C7—C8—C9	119.9 (3)
N3—C4—C5	100.0 (2)	C8—C9—C10	120.5 (3)
N3—C4—C6	113.8 (2)	C9—C10—C11	119.8 (3)
C5—C4—C6	114.5 (2)	C10—C11—C12	120.6 (3)
O1—C5—C4	103.9 (2)	C7—C12—C11	120.1 (3)
O1—C5—C7	111.2 (2)		

The scan rate was 3° min^{-1} (in ω). Weak reflections [$I < 10\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to improve accuracy. H atoms were placed at assumed positions (C,N—H = 0.95 Å, $U = 1.2 \times U_{eq}$ of the associated atom) and fixed. The correct enantiomer was chosen on the basis of the known configuration of the starting material [(−)-(1*R*,2*S*)-norephedrine].

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55982 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1047]

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Structure of Vicogenin

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Abstract

Vicogenin is a new 28-nortriterpenoid. Its structure has been elucidated as 2α,3β,16α,17β,23-pentahydroxy-28-norolean-12-ene. In the crystal there are two water molecules per vicogenin molecule. Hydrogen bonds link the vicogenin molecules to each other and to the water molecules.

Comment

The title compound (I) has been isolated for the first time from the chloroform extract of the plant *Vicoa indica* DC(composital). The plant is said to exhibit antifertility activity when administered during the post-partum period in albino rats (Gandhi,

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