S(12) à ce plan sont respectivement 0,001, 0,020 et −1,476 Å. L'angle entre les cycles I et II est nul. Les cycles benzo (II) et phényle (III) font entre eux un angle de 124° et les plans des deux cycles phényle (III) et (IV) font un angle de 84°.

Discussion. Les particularités suivantes nous ont semblé plus spécifiques de cette structure.

(*a*) Comme il a été dit précédemment, le cycle oxazole est plan, les cycles benzo et oxazole sont coplanaires.

(*b*) L'atome d'azote N(3) est plan: la distance de N(3) au plan des atomes P(11), C(2) et C(4) est de 0,001 Å. La longueur de la liaison N(3)—P(11) est de 1,711 (2) Å soit 50% de caractère double [le taux de double liaison a été calculé à partir des valeurs des rayons de covalence d'aprés Pauling (1960)]. Ce résultat est en accord avec une formation d'une liaison hybride supplémentaire ($p\pi$–$d\pi$) entre l'atome d'azote (hybridé sp^2) et de phosphore (recouvrement latéral partiel de l'orbitales p de l'atome d'azote et d de l'atome de phosphore avec retour d'une paire électronique de l'azote vers le phosphore).

(*c*) La liaison P(11)—S(12) de 1,889 (1) Å possède 85% de caractère de triple liaison.

(*d*) Les liaisons entre atomes qui forment le cycle oxazole I présentent toutes un caractère de double liaison:

O(1)—C(12)	49%
C(2)—N(3)	47%
N(3)—C(4)	30%
C(4)—C(5)	83%
C(5)—O(1)	35%

nous sommes donc en présence d'un cycle fortement conjugué avec le cycle benzo.

(*e*) Les atomes S(10), C(2), N(3) et P(11) sont coplanaires [d_{max} = 0,001 Å pour N(3)—plan moyen] et la distance entre P(11) et S(10) est de 3,331 (2) Å. Cette distance est à comparer à celle d'un homologue acyclique de la littérature (Mikolajczyk, Kielbasinski & Basinsky, 1984). Il s'agit d'une thiourée substituée analogue à (1) dont la distance S···P est de 3,73 Å et pour laquelle la migration du groupe phosphoryle, de l'atome d'azote à l'atome de soufre, est réversible. Ceci permet de supposer que pour le composé décrit dans cet article cette tautomérisation est aussi possible, la structure obtenue dans le cristal étant la forme thermodynamiquement la plus stable. Cette dernière remarque est donc compatible avec le premier mécanisme proposé au début de l'article (schéma 2).

Références

BLONSKI, C., BELGHITH, H., KLAÉBÉ, A. & PÉRIÉ, J. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. 1369–1374.

BLONSKI, C., GASC, M. B., HEGARTY, A. F., KLAÉBÉ, A. & PÉRIÉ, J. (1984). *J. Am. Chem. Soc.* **106**, 7525–7529.

CHEN, C. S., PARTHASARATHY, R. & DE TITA, G. (1976). *J. Am. Chem. Soc.* **98**, 4983–4990.

International Tables for X-ray Crystallography (1974). Tome IV, pp. 99–149. Birmingham: Kynoch Press. (Distributeur actuel Kluwer Academic Publishers, Dordrecht.)

KLUGER, R. & ADAWADKAR, P. D. (1976). *J. Am. Chem. Soc.* **98**, 3741–3742.

KLUGER, R., DAVIS, P. P. & ADAWADKAR, P. D. (1979). *J. Am. Chem. Soc.* **101**, 5995–6000.

KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.

LYNEN, L. (1967). *Biochem. J.* **102**, 381–400.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. de York, Angleterre, et de Louvain, Belgique.

MIKOLAJCZYK, M., KIELBASINSKI, P. & BASINSKY, W. (1984). *J. Org. Chem.* **49**, 899–908.

MOTHERWELL, S. & CLEGG, B. (1978). *PLUTO.* Programme pour le dessin des structures moléculaires et cristallines. Univ. de Cambridge, Angleterre.

PAULING, L. (1960). *The Nature of the Chemical Bond*, 3$^{\text{ième}}$ éd., p. 224. Cornell Univ. Press.

SHELDRICK, G. (1976). *SHELX76.* Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.

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Structure of 5-Ethyl-3-phenylperhydropyrrolo[2,1-*b*]oxazolidine-5-carbonitrile

BY A. CHIARONI, S. ARSENIYADIS, C. KAN AND C. RICHE

Institut de Chimie des Substances Naturelles, CNRS, 91190 – Gif sur Yvette, France

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Abstract. $C_{15}H_{18}N_2O$, $M_r = 242{\cdot}32$, monoclinic, $C2$, $a = 15{\cdot}039$ (6), $b = 9{\cdot}535$ (4), $c = 19{\cdot}050$ (6) Å, $\beta = 104{\cdot}66$ (2)°, $V = 2642{\cdot}7$ Å^3, $Z = 8$, $D_x = 1{\cdot}218$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $F(000) = 1040$, $\mu = 5{\cdot}33$ cm^{-1}, final $R = 0{\cdot}047$ for 2264 observed reflections. The relative stereochemistry was determined and the absolute configuration fixed by the known absolute configuration of the starting material. C(3) and

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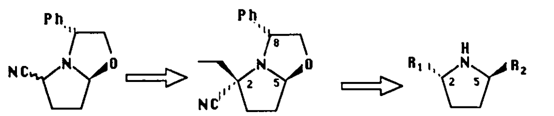
C(4) are 0·37 and −0·23 Å out of the plane of the pyrrolidine ring; O(6) lies 0·55 Å out of the plane of the oxazolidine ring. Crystal packing is by van der Waals forces.

Introduction. The title compound (2) has been prepared as part of an investigation on the asymmetric synthesis of *trans*-2,5-dialkylpyrrolidines (3), constituents of the venom of the South African ant species (Jones, Blum & Fales, 1982), starting from a chiral pool-derived synthon (1) (Arseniyadis, Huang, Piveteau & Husson, 1988).



(1) (2) (3)

R_1, R_2 } Alkyl groups

The stereochemical outcome of the reaction scheme left some ambiguities, owing especially to the lack of mutual relaxation of H(8) and H(5) protons through their dipolar interaction (1D NOE DIFF, NOESY techniques used). An X-ray diffraction analysis to determine the ring junction was therefore crucial in rationalizing synthetic achievements and assigning relative configuration at C(2) and C(5) atoms. Since the absolute configuration at C(8) is established, based on the starting amino acid (*R*)-phenylglycine, this study allowed an adequate assignment of the absolute configuration at the C(2) and C(5) centres.

Experimental. Colourless crystals (m.p. 328 K) were obtained from evaporation of an acetone–hexane mixture. Data collected on a Philips PW 1100 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Crystal size 0·5 × 0·4 × 0·3 mm. Unit-cell parameters from setting angles of 25 accurately selected reflections. From 2413 intensities obtained by the θ–2θ scan technique up to $\theta = 64°$ ($-17 \le h \le 16$, k: 0→11, l: 0→22), 2342 unique reflections of which 2264 observed with $I \ge 3\sigma(I)$, $\sigma(I)$ from counting statistics. Three standard reflections monitored every 2 h showed no significant changes in intensity. Corrections for Lorentz–polarization effects, not for absorption.

Structure solved by direct methods with program *DEVIN* (Riche, 1982) and refined anisotropically by full-matrix least squares minimizing the function $\sum w(|F_o| - |F_c|)^2$. All H atoms located on difference Fourier maps, included in calculations at theoretical positions [d(C—H) = 1·08 Å] and assigned the equivalent isotropic thermal factor of the bonded C atom. Refinement converged at $R = 0{\cdot}047$ and $wR = 0{\cdot}054$, weighting scheme $w = 1/[\sigma^2(F_o) + 0{\cdot}00005F_o^2]$; $(\Delta/\sigma)_{max} = 1{\cdot}1$. On the final difference Fourier map, $(\Delta\rho)_{max} = 0{\cdot}21$ and $(\Delta\rho)_{min} = -0{\cdot}26$ e Å^{-3}. Calculations performed with program *SHELX*76 (Sheldrick, 1976). Atomic scattering factors those of *SHELX*. Final atomic coordinates, bond distances and angles, and torsion angles are given in Tables 1, 2 and 3 respectively.*

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal factors* (×10^3 Å^2)

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

	x	y	z	U_{eq}
N1	8013 (1)	10475 (2)	10792 (1)	46 (1)
C2	8925 (1)	9841 (1)	11149 (1)	45 (2)
C3	8924 (2)	9746 (3)	11956 (1)	64 (2)
C4	8352 (2)	11010 (3)	12056 (1)	72 (3)
C5	7623 (2)	11074 (3)	11361 (1)	63 (2)
O6	6858 (1)	10184 (2)	11381 (1)	81 (2)
C7	6465 (1)	9835 (4)	10642 (1)	74 (3)
C8	7294 (1)	9592 (2)	10324 (1)	51 (2)
C9	9612 (1)	10928 (3)	11074 (1)	54 (2)
N10	10141 (1)	11760 (3)	11023 (2)	86 (3)
C11	9155 (1)	8486 (3)	10822 (1)	52 (2)
C12	10149 (2)	8033 (3)	11118 (1)	70 (3)
C13	7111 (1)	9945 (2)	9532 (1)	49 (2)
C14	7754 (1)	10622 (3)	9244 (1)	54 (2)
C15	7579 (2)	10907 (4)	8507 (1)	68 (2)
C16	6754 (2)	10513 (4)	8047 (2)	78 (3)
C17	6107 (2)	9846 (4)	8320 (2)	76 (3)
C18	6275 (2)	9551 (3)	9053 (1)	64 (2)
N1′	7922 (1)	7740 (2)	5666 (1)	43 (1)
C2′	8830 (1)	7150 (2)	6056 (1)	43 (2)
C3′	8792 (2)	7057 (3)	6851 (1)	60 (2)
C4′	8186 (2)	8296 (3)	6927 (1)	67 (2)
C5′	7481 (1)	8306 (3)	6211 (1)	57 (2)
O6′	6735 (1)	7383 (2)	6212 (1)	68 (2)
C7′	6382 (1)	7031 (3)	5472 (1)	62 (2)
C8′	7235 (1)	6861 (2)	5169 (1)	45 (2)
C9′	9523 (1)	8235 (3)	5995 (1)	56 (2)
N10′	10055 (2)	9063 (3)	5959 (2)	89 (3)
C11′	9096 (1)	5771 (3)	5756 (1)	50 (2)
C12′	10077 (2)	5310 (3)	6117 (1)	68 (3)
C13′	7083 (1)	7287 (2)	4388 (1)	47 (2)
C14′	7752 (1)	7995 (3)	4138 (1)	56 (2)
C15′	7600 (2)	8393 (3)	3420 (1)	72 (3)
C16′	6786 (2)	8084 (3)	2932 (1)	79 (3)
C17′	6117 (2)	7372 (3)	3160 (1)	74 (3)
C18′	6254 (2)	6969 (3)	3882 (1)	63 (2)

Discussion. One of the two molecules of the asymmetric unit (unprimed) is shown in Fig. 1 with the atomic labelling, according to the known C(8) *R* absolute configuration. The pyrrolidine and oxazolidine rings appear *cis*-fused and thus H(5) and H(8) are *trans* to each other. The absolute configuration is *R* at C(2), the ethyl chain being in the β position, and *S* at C(5). Values reported in Tables 2 and 3 show both molecules of the asymmetric unit strictly identical. The average dihedral angle between the two five-membered rings is 122·8°.

The pyrrolidine ring is nearly in a half-chair conformation with atoms C(3) and C(4) out of the plane of the other three atoms by 0·37 and −0·23 Å respectively, while the oxazolidine ring adopts an

* Lists of structure factors, anisotropic thermal factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51070 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and bond angles* (°)

N1–C2	1·496 (3)	N1′–C2′	1·490 (3)
N1–C5	1·473 (3)	N1′–C5′	1·469 (3)
N1–C8	1·478 (3)	N1′–C8′	1·472 (3)
C2–C3	1·540 (3)	C2′–C3′	1·532 (3)
C2–C9	1·496 (3)	C2′–C9′	1·494 (3)
C2–C11	1·511 (3)	C2′–C11′	1·527 (3)
C3–C4	1·520 (4)	C3′–C4′	1·522 (4)
C4–C5	1·491 (3)	C4′–C5′	1·501 (3)
C5–O6	1·438 (3)	C5′–O6′	1·427 (3)
O6–C7	1·421 (3)	O6′–C7′	1·415 (3)
C7–C8	1·537 (3)	C7′–C8′	1·544 (3)
C8–C13	1·501 (3)	C8′–C13′	1·503 (3)
C9–N10	1·145 (4)	C9′–N10′	1·138 (4)
C11–C12	1·521 (4)	C11′–C12′	1·527 (3)
C13–C14	1·387 (3)	C13′–C14′	1·392 (3)
C13–C18	1·405 (3)	C13′–C18′	1·403 (3)
C14–C15	1·387 (4)	C14′–C15′	1·382 (4)
C15–C16	1·378 (4)	C15′–C16′	1·368 (4)
C16–C17	1·370 (4)	C16′–C17′	1·371 (4)
C17–C18	1·384 (4)	C17′–C18′	1·393 (4)
C2–N1–C5	108·2 (2)	C2′–N1′–C5′	107·8 (2)
C2–N1–C8	119·4 (2)	C2′–N1′–C8′	120·7 (2)
C5–N1–C8	107·4 (2)	C5′–N1′–C8′	107·1 (2)
N1–C2–C3	104·2 (2)	N1′–C2′–C3′	104·9 (2)
N1–C2–C9	104·7 (2)	N1′–C2′–C9′	105·6 (2)
N1–C2–C11	115·9 (2)	N1′–C2′–C11′	115·6 (2)
C3–C2–C9	108·1 (2)	C3′–C2′–C9′	108·6 (2)
C3–C2–C11	114·7 (2)	C3′–C2′–C11′	113·5 (2)
C9–C2–C11	108·5 (2)	C9′–C2′–C11′	108·2 (2)
C2–C3–C4	102·8 (2)	C2′–C3′–C4′	102·8 (2)
C3–C4–C5	103·5 (2)	C3′–C4′–C5′	102·8 (2)
N1–C5–C4	107·3 (2)	N1′–C5′–C4′	107·4 (2)
N1–C5–O6	105·1 (2)	N1′–C5′–O6′	105·7 (2)
C4–C5–O6	111·5 (2)	C4′–C5′–O6′	111·7 (2)
C5–O6–C7	104·3 (2)	C5′–O6′–C7′	104·0 (2)
O6–C7–C8	104·5 (2)	O6′–C7′–C8′	105·0 (2)
N1–C8–C7	102·7 (2)	N1′–C8′–C7′	102·4 (2)
N1–C8–C13	113·5 (2)	N1′–C8′–C13′	112·9 (2)
C7–C8–C13	113·7 (2)	C7′–C8′–C13′	114·2 (2)
C2–C9–N10	179·5 (3)	C2′–C9′–N10′	179·1 (3)
C2–C11–C12	113·3 (2)	C2′–C11′–C12′	113·2 (2)
C8–C13–C14	122·2 (2)	C8′–C13′–C14′	121·8 (2)
C8–C13–C18	120·0 (2)	C8′–C13′–C18′	120·5 (2)
C14–C13–C18	117·8 (2)	C14′–C13′–C18′	117·6 (2)
C13–C14–C15	121·3 (2)	C13′ C14′ C15′	121·2 (2)
C14–C15–C16	120·0 (3)	C14′–C15′–C16′	120·5 (3)
C15–C16–C17	119·7 (3)	C15′–C16′–C17′	119·6 (3)
C16–C17–C18	120·8 (3)	C16′–C17′–C18′	120·8 (3)
C13–C18–C17	120·4 (3)	C13′–C18′–C17′	120·2 (2)

envelope conformation with O(6) out of the plane of the four other atoms by 0·55 Å. In the crystal only normal van der Waals contacts are observed.

Table 3. *Comparison of selected torsion angles* (°) *of the two molecules*

C5–N1–C2–C3	14·9 (2)	14·1 (2)
N1–C2–C3–C4	−32·1 (2)	−32·1 (2)
C2–C3–C4–C5	37·4 (2)	37·6 (2)
C3–C4–C5–N1	−28·9 (2)	−29·9 (2)
C4–C5–N1–C2	8·6 (2)	9·9 (2)
N1–C5–O6–C7	−37·6 (2)	−38·2 (2)
C4–C5–O6–C7	−153·5 (3)	−154·7 (3)
C5–O6–C7–C8	40·0 (2)	39·0 (2)
O6–C7–C8–N1	−26·8 (2)	−24·9 (2)
C7–C8–N1–C5	4·0 (2)	1·7 (2)
C8–N1–C5–O6	19·9 (2)	21·9 (2)
N1–C8–C13–C14	22·7 (2)	24·6 (2)
N1–C8–C13–C18	−159·2 (3)	−155·8 (3)
C7–C8–C13–C14	139·5 (3)	141·1 (3)
C7–C8–C13–C18	−42·4 (2)	−39·4 (2)
C8–N1–C2–C9	138·4 (2)	136·1 (2)
C8–N1–C2–C11	18·9 (2)	16·5 (2)
C5–N1–C2–C11	142·0 (2)	139·9 (2)
N1–C2–C11–C12	169·7 (3)	172·9 (3)
C3–C2–C9–N10	9·8 (3)	5·3 (3)

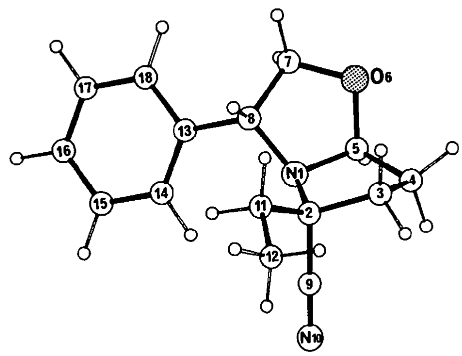


Fig. 1. Molecular structure and atomic numbering.

References

ARSENIYADIS, S., HUANG, P. Q., PIVETEAU, D. & HUSSON, H. P. (1988). *Tetrahedron*, **44**, 2457–2470.

JONES, T. H., BLUM, M. S. & FALES, H. M. (1982). *Tetrahedron*, **38**, 1949–1958.

RICHE, C. (1982). *DEVIN*. Abstr. Seventh Eur. Crystallogr. Meet., Jerusalem, Aug. 29–Sept. 3, p. 25.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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1,6-Diazabicyclo[4.4.0]decane Hydrate

BY J. M. WHITE, R. W. ALDER AND A. G. ORPEN

Departments of Inorganic and Organic Chemistry, The University, Bristol BS8 1TS, England

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Abstract. $C_8H_{16}N_2.H_2O$, $M_r = 158{\cdot}25$, monoclinic, $C2/c$, $a = 12{\cdot}139$ (8), $b = 7{\cdot}106$ (4), $c = 11{\cdot}295$ (5) Å, $\beta = 105{\cdot}79$ (4)°, $V = 937{\cdot}5$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}11$ g cm^{-3}, $\bar{\lambda}$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}70$ cm^{-1}, $F(000) = 352$, $R = 0{\cdot}041$ for 592 unique observed reflections. The title compound adopts a *trans*-decalin conformation in the solid state. The molecules are linked together by water molecules in an extended hydrogen-bonded network in the direction of the z axis; the N(1)···O(1) distance is 3·019 (3) Å and the N(1)···H(1)–O(1) angle is 170 (2)°. The N(1)–N(1′) bond length, corrected for rigid-body motion, is 1·486 (3) Å.

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