X-Ray Molecular Structure and Absolute Configuration of the Ring Nitrogen Atoms in (−)-1*R*,2*S*-1-(*S*)-α-Phenylethylcarbamoyl-2-methyl-3,3-pentamethylenediaziridine

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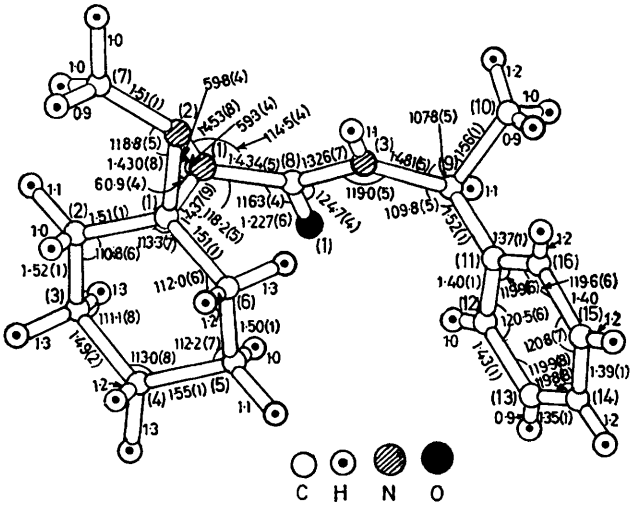
Summary The ring nitrogen atoms in (−)-1*R*,2*S*-1-(*S*)-α-phenylethylcarbamoyl-2-methyl-3,3-pentamethylenediaziridine (I) were found to have the 1*R*,2*S* configuration; the pyramidal geometry of an amide nitrogen, N(1) in the ring is shown to be essentially identical with that of N(2).

RECENTLY a series of previously unreported chiral diaziridines have been prepared.[1] We have now established the absolute configuration of (I) and confirmed an amide conjugation relaxation effect on nitrogen inclusion into a three-membered ring.[2]

The preparation of compound (I) has already been described,[1] m.p. 92—93 °C, $[\alpha]_D = -62{\cdot}4°$ (*c* 1·2 in EtOH). *Crystal data:* $C_{16}N_3OH_{23}$, $M = 273{\cdot}38$, monoclinic, $a = 8{\cdot}650$ (3), $b = 11{\cdot}200(4)$, $c = 9{\cdot}584$ (3) Å, $\gamma = 119°\ 39'$ (3), $U = 806{\cdot}9$ Å^3, $D_c = 1{\cdot}12$ g cm^{-3}, $Z = 2$, space group $P2_1$. Intensities of 957 independent non-zero reflections *hk*0—*hk*8 were measured on a DAR-UM automatic *X*-ray diffractometer with graphite-monochromated Cu-K_α radiation by the layer registration technique. Absorption corrections were not applied.

The structure was solved by the multivariant phase method[3] using the tangent formula of Karle and Hauptman, and refined by full-matrix least-squares with anisotropic temperature factors to an *R* value of 0·074. The absolute configuration and structural parameters are shown in the Figure. It is of interest that the pyramidal geometry of the ring amide nitrogen N(1) is practically analogous to that of N(2); the C(8)–N(1) bond forms an angle of 59·0° with the plane of the N(1)–N(2)–C(1) ring, while the corresponding angle of the C(7)–N(2) bond is 60·3°. The amide fragment of the substituent N(1)–C(8)–O–N(3)–C(9) is practically planar forming angles of 116·4° and 135·8° with the N(1)–N(2)–C(1) and C(11)–C(12) . . . C(16) planes, resepctively, and 31·4 with the C(9)–C(10) bond. The 1*R*,2*S*-configuration of the cyclic nitrogen atoms in (I) was established with respect to a known *S*-configuration of the C(9) centre (Figure). Hence the 1*R*,2*R* configuration can be assigned to the nitrogen atoms in (+)-1-methyl-3,3-pentamethylenediaziridine.



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[1] R. G. Kostyanovskii, A. E. Polyakov, and V. I. Markov, *Izvest. Akad. Nauk S.S.S.R., Ser. Khim.*, 1974, 1847; 1975, 198, *Dokl. Akad. Nauk S.S.S.R.*, 1974, **219**, 873.

[2] T. Z. Papoyan, I. I. Chervin, and R. G. Kostyanovskii, *Izvest. Akad. Nauk S.S.S.R., Ser. Khim.*, 1968, 1530; R. G. Kostyanovskii, K. S. Zakharov, M. Zaripova, and V. F. Rudchenko, *ibid.*, 1975, 875; *Tetrahedron Letters*, 1974, 4207.

[3] V. I. Andrianov, Z.Sh. Safina, and B. L. Tarnopol'skii, *Zhur. strukt. Khim.*, 1974, **15**, 911.