Momany & Scheraga, 1973) and closely resembles conformation IV of *N*-acetyl-*N'*-methylglycine amide for which an unconstrained *ab initio* geometry on the 4-21G level is published (Schäfer, Van Alsenoy & Scarsdale, 1982). The comparison (Table 2) between our experimental results and the calculated structure is remarkably good, keeping in mind the inherent distinction between X-ray and 4-21G *ab initio* distances. It reveals the existence of differences in primary parameters (bond distances and angles) that might have gone unnoticed and are, indeed, often neglected in standard geometries frequently used in peptide stereochemistry. For example the peptide N(2)–C(7) bond is about 0·03 Å shorter than the peptide N(1)–C(5) bond, while C(3)–C(7), *i.e.* the C–C(=O) inside the backbone, is about 0·04 Å longer than C(5)–C(6), *i.e.* the terminating C–C(=O). These observations, which are in agreement with the calculations, suggest a larger contribution from charged resonances of the type $^{\ominus}$O–C=N$^{\oplus}$–C to the structure of the *N'*-methyl amide side in comparison with the *N*-acetyl side. Furthermore, valence angles for which the experimental value is over 120° are matched, with only one exception, by calculated values larger than 120°. The same holds for angles under 120°. It shows that the large value (~124°) for O(3)–C(7)–N(2) is inherent to the *N'*-methylglycine amide fragment. The fact that the latter is part of a ring system explains the deviation of the C(5)–N(1)–C(3)–C(7) torsion angle from the calculated value. This, together with the aforementioned differences of the N(1)–C(3)–C(7) valence and the C(3)–C(7) torsion angles between the *cis* and *trans* derivatives, shows that the backbone of a peptide can be chemically manipulated to a certain extent.

Aside from this conclusion, the internal consistency of the experimental values and, moreover, the excellent agreement with the calculations, precisely at points where conformationally induced geometry variations were expected, strongly underline the conclusion of Schäfer *et al.* (1982) about the importance of local geometries for peptide conformations.

FV thanks the Belgian Organization IWONL for a predoctoral grant. CVdA thanks the Belgian Science Foundation NFWO for financial assistance. Part of the work was supported by NATO, Research Grant No. 0314/82.

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Structure of the 3^1_{10}-Helical Pentapeptide Boc-L-Pro-Aib-L-Ala-Aib-L-Ala-OH

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(*Received* 15 *March* 1985; *accepted* 15 *August* 1985)

Abstract. $C_{24}H_{41}N_5O_8$, $M_r = 527{\cdot}62$, orthorhombic, $P2_12_12$, $a = 18{\cdot}839$ (3), $b = 18{\cdot}776$ (7), $c = 9{\cdot}179$ (1) Å, $V = 3247{\cdot}0$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}079$ Mg m^{-3} (disordered hydrocarbon solvent not included), λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}601$ mm^{-1}, $F(000) = 1136$, $T = 293$ K, final $R = 0{\cdot}056$ for 2429 unique observed reflections. The pentapeptide (1) represents the N-terminal sequence 2–6 of the membrane-modifying icosapeptide antibiotic alamethicin F30. Pentapeptide (1) adopts a left-handed 3^1_{10}-helical structure of one type II followed by two type III consecutive β-turns with 4→1 hydrogen bonds. Despite

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0108-2701/85/121821-05$01.50

their different end groups, pentapeptide (1) and *p*Cl-Bz-L-Pro-Aib-L-Ala-Aib-L-Ala-OMe (2) [Cameron, Hanson & Taylor (1982). *Cryst. Struct. Commun.* **11**, 321–330] are the first L-amino-acid pentapeptides, with left-handed 3_{10}-helical conformations in the solid state. However, this obviously preferred backbone changes to a right-handed α-helix upon incorporation into alamethicin.

Introduction. The sterically hindered amino acid α-aminoisobutyric acid (Aib) is a constituent of many peptide antibiotics (peptaibols) and is of interest for the design of new analogues of biologically active peptides, *e.g.* of angiotensin II as shown already by Marshall, Eilers & Vine (1972). During the total synthesis of the icosapeptide antibiotic alamethicin F30 (Schmitt & Jung, 1985), many of the intermediate segments crystallized readily. Their structure determinations turned out to be most fascinating, because we found tripeptides with new β-turns (Jung, Brückner, Bosch, Winter, Schaal & Strähle, 1983; Bosch, Jung, Voges & Winter, 1984), a pentapeptide with a 3^r_{10}-helix (Bosch, Jung & Winter, 1983), a nonapeptide with an $\alpha/3_{10}$-helix (Bosch, Jung, Schmitt, Sheldrick & Winter, 1984; Bosch, Jung, Schmitt & Winter, 1985*a*) and an undecapeptide with an α-helix (Butters, Hütter, Jung, Pauls, Schmitt, Sheldrick & Winter, 1981; Schmitt, Winter, Bosch & Jung, 1982; Bosch, Jung, Schmitt & Winter, 1985*b*).

Continuing this systematic series we describe in the following a pentapeptide with a *left*-handed 3_{10}-helix consisting of Aib and L-configurated residues. The sequence of (1) is identical with that of the positions 2–6 of alamethicin and with that of *p*Cl-Bz-L-Pro-Aib-L-Ala-Aib-L-Ala-OMe (2) (Cameron, Hanson & Taylor, 1982), which also exhibits a left-handed 3_{10}-helix. We refer also to recent structure determinations by other groups working in this field (Francis, Iqbal, Balaram & Vijayan, 1983; Toniolo, Bonora, Benedetti, Bavoso, Di Blasio, Pavone & Pedone, 1983).

Experimental. Single crystal 0·25 × 0·25 × 0·2 mm from ethyl acetate/light petroleum, Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation with graphite monochromator, 25 reflections used to measure lattice parameters, 2777 reflections ($|F| > 0$), 2434 unique, ω scan, $\theta = 5$–$60°$, $0 \le h \le 10$, $-21 \le k \le 0$, $-21 \le l \le 0$, two standard reflections ($40\bar{1}$ and $\bar{3}\bar{4}\bar{4}$) with constant intensity; Lp correction, absorption ignored; direct methods (*MULTAN*80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refinement on F with *SHELX*76 (Sheldrick, 1976), non-H atoms anisotropic, H atoms isotropic as rigid groups with fixed distance of 0·96 Å, (O)–H atom from difference Fourier map allowed to refine freely, common temperature factor for all H atoms; final $R = 0{\cdot}056$, $wR = 0{\cdot}056$ for 2429 reflections, unit weights (five extinction-damaged reflections excluded),* max. Δ/σ 0·7, av. Δ/σ 0·1, no electron density $> \pm 0{\cdot}25$ e Å^{-3} in last difference Fourier maps; scattering factors from Cromer & Mann (1968), f' and f'' from Cromer & Liberman (1970).

Discussion. Fig. 1 shows a perspective view of the pentapeptide together with the atomic numbering. Tables 1 and 2 contain atomic coordinates with equivalent isotropic temperature parameters and bond lengths and angles respectively. The title compound (1) adopts a 3_{10}-helical secondary structure, consisting of three consecutive β-turns (types II, III′ and III′; Venkatachalam, 1968) with three intramolecular 4→1 hydrogen bonds [N(3)···O(2) = 2·906 (9), N(4)···O(3) = 3·000 (9) and N(5)···O(4) = 3·073 (9) Å]. 3_{10}-Helices are often observed in Aib-containing oligopeptides (Bosch *et al.*, 1983). The octapeptide *p*Br-Bz-$(Aib)_8$-OBu^t (Toniolo, Bonora, Bavoso, Benedetti, Di Blasio, Pavone & Pedone, 1984) and even a decapeptide (Francis *et al.*, 1983) crystallized in 3_{10}-helical structures. In contrast to most other pentapeptides with L-configurated amino acids, which adopt regular right-handed 3_{10}-helices, (1) folds into a *left*-handed conformation in the solid state. The torsional angles are summarized in Table 3. As observed in other cases (Bosch, Jung, Voges & Winter, 1984; Jung *et al.*, 1983), the influence of the different protecting groups on the backbone conformations of (1) and (2) is also a very small one. Furthermore the methyl ester of (1) exhibits almost the same CD and NMR spectra as the pentapeptide acid (1) in methanolic solution. On the other hand the related tetrapeptide Boc-L-Pro-Aib-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42441 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

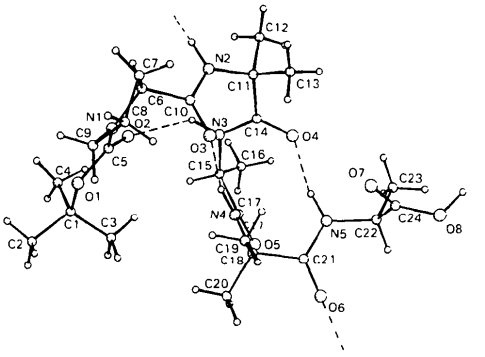

Fig. 1. Perspective view of the pentapeptide Boc-L-Pro-Aib-L-Ala-Aib-L-Ala-OH (1) with atomic numbering.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$ (Å^2).

	x	y	z	U_{eq}
N(1)	0·1086 (2)	0·3807 (2)	0·4083 (5)	0·050 (3)
N(2)	0·2721 (2)	0·3626 (2)	0·5660 (5)	0·061 (3)
N(3)	0·3008 (2)	0·2468 (2)	0·3922 (5)	0·056 (3)
N(4)	0·2010 (2)	0·1462 (2)	0·4615 (4)	0·047 (2)
N(5)	0·2524 (3)	0·0669 (2)	0·6922 (4)	0·055 (3)
O(1)	0·0736 (2)	0·3293 (2)	0·2056 (4)	0·066 (2)
O(2)	0·1917 (2)	0·3346 (2)	0·2587 (4)	0·058 (2)
O(3)	0·1845 (2)	0·2897 (2)	0·6008 (4)	0·056 (2)
O(4)	0·3494 (2)	0·1860 (2)	0·5784 (5)	0·072 (3)
O(5)	0·2595 (2)	0·0619 (2)	0·3318 (4)	0·061 (2)
O(6)	0·1761 (2)	−0·0205 (2)	0·6377 (5)	0·069 (3)
O(7)	0·2342 (3)	0·1091 (3)	0·9749 (5)	0·105 (4)
O(8)	0·2815 (3)	0·0096 (2)	1·0624 (4)	0·079 (3)
C(1)	0·0815 (3)	0·2864 (4)	0·0720 (6)	0·061 (4)
C(2)	0·0070 (4)	0·2866 (5)	0·0092 (9)	0·113 (6)
C(3)	0·1027 (5)	0·2129 (4)	0·114 (1)	0·118 (7)
C(4)	0·1325 (6)	0·3228 (7)	−0·0276 (8)	0·120 (7)
C(5)	0·1302 (3)	0·3465 (3)	0·2883 (6)	0·052 (4)
C(6)	0·1619 (3)	0·4073 (3)	0·5095 (6)	0·051 (3)
C(7)	0·1163 (3)	0·4396 (3)	0·6325 (7)	0·067 (4)
C(8)	0·0463 (3)	0·4016 (4)	0·6205 (7)	0·077 (5)
C(9)	0·0357 (3)	0·3888 (4)	0·4606 (7)	0·066 (4)
C(10)	0·2087 (3)	0·3478 (3)	0·5636 (6)	0·049 (3)
C(11)	0·3313 (3)	0·3113 (3)	0·6145 (7)	0·070 (4)
C(12)	0·4049 (3)	0·3434 (4)	0·584 (1)	0·100 (6)
C(13)	0·3222 (5)	0·2957 (4)	0·7771 (8)	0·098 (6)
C(14)	0·3270 (3)	0·2418 (3)	0·5273 (7)	0·059 (3)
C(15)	0·2959 (3)	0·1845 (3)	0·2994 (6)	0·061 (4)
C(16)	0·3691 (4)	0·1584 (4)	0·2531 (9)	0·097 (6)
C(17)	0·2512 (3)	0·1251 (3)	0·3667 (5)	0·051 (3)
C(18)	0·1536 (3)	0·0945 (3)	0·5329 (6)	0·054 (3)
C(19)	0·1072 (3)	0·1378 (4)	0·6389 (8)	0·079 (5)
C(20)	0·1059 (4)	0·0559 (4)	0·4229 (8)	0·081 (5)
C(21)	0·1959 (3)	0·0413 (3)	0·6251 (6)	0·052 (3)
C(22)	0·2898 (3)	0·0284 (3)	0·8090 (6)	0·053 (3)
C(23)	0·3700 (3)	0·0393 (4)	0·7941 (8)	0·074 (4)
C(24)	0·2656 (3)	0·0538 (3)	0·9568 (5)	0·066 (4)

Ala-Aib-OBzl (Smith, Pletnev, Duax, Balasubramanian, Bosshard, Czerwinski, Kendrick, Mathews & Marshall, 1981) adopts a regular *right*-handed conformation ($\varphi/\psi \approx -60/-30°$). Obviously the L-Pro residue cannot be responsible for the unusual handedness of (1). Because the torsional angles of Aib in the tripeptides Boc-L-Ala-Aib-L-Ala-OMe (Bosch, Jung, Voges & Winter, 1984) and Ac-L-Ala-Aib-L-Ala-OMe (Jung *et al.*, 1983) are also 'left-handed' ($\varphi/\psi \approx +60/+30°$), we suppose that the structural segment -Ala-Aib-Ala- imparts the major influence upon secondary structure of (1) and (2). It should also be noted that in Piv-Pro-Aib-NHMe and the disulfide Boc-Cys-Pro-Aib-Cys-NHMe the adoption of type II and III turns has been found for the -Pro-Aib- sequence (Prasad, Balaram & Balaram, 1982; Ravi, Prasad & Balaram, 1983).

Recently the preference of the Boc-Pro-urethane group for *cis* configuration has been shown (Benedetti, Pedone, Toniolo, Némethy, Pottle & Scheraga, 1980) in a survey of 31 Boc-peptides. Although we could confirm this preference in other cases (Bosch, Schmitt, Jung & Winter, 1984), the pentapeptides (1) and (2) have a *trans*-urethane bond. Model considerations show that the formation of the 3^1_{10}-helix with the corresponding *cis* configuration would be strongly hindered on steric reasons.

Proline is well known to restrict the conformational freedom of a peptide backbone due to the rigid pyrrolidine ring system. As a result the φ torsional angles are usually situated close to −60°, whereas for the ψ angles three main regions are observable: about 150° (polyproline structure), about 70° (γ-turn) and about −40° (3_{10}- or α-helical area) (Flippen & Karle, 1976; Madison, 1977; Prasad & Balaram, 1982). Both pentapeptides (1) and (2) adopt a conformation related to polyproline (*e.g.* ~150°), according to a type II β-turn. Together with two consecutive type III′ β-turns (Table 3), the 3^1_{10}-helix finally results. The pyrrolidine ring adopts the usual envelope conformation with C(8) (≡ C$^\gamma$) being situated 0·21 Å above the least-squares plane through the ring atoms.

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

C(1)–C(2)	1·517 (10)	C(14)–O(4)	1·222 (7)
C(1)–C(3)	1·486 (10)	C(14)–N(3)	1·338 (8)
C(1)–C(4)	1·493 (12)	N(3)–C(15)	1·450 (7)
C(1)–O(1)	1·475 (7)	C(15)–C(16)	1·523 (9)
O(1)–C(5)	1·348 (7)	C(15)–C(17)	1·529 (8)
C(5)–O(2)	1·211 (7)	C(17)–O(5)	1·240 (7)
C(5)–N(1)	1·338 (7)	C(17)–N(4)	1·344 (6)
N(1)–C(6)	1·456 (7)	N(4)–C(18)	1·473 (7)
N(1)–C(9)	1·463 (7)	C(18)–C(19)	1·540 (9)
C(6)–C(7)	1·543 (8)	C(18)–C(20)	1·534 (9)
C(7)–C(8)	1·502 (9)	C(18)–C(21)	1·534 (8)
C(8)–C(9)	1·500 (9)	C(21)–O(6)	1·224 (6)
C(6)–C(10)	1·508 (7)	C(21)–N(5)	1·319 (7)
C(10)–O(3)	1·230 (6)	N(5)–C(22)	1·473 (7)
C(10)–N(2)	1·336 (7)	C(22)–C(23)	1·532 (9)
N(2)–C(11)	1·460 (8)	C(22)–C(24)	1·509 (7)
C(11)–C(12)	1·536 (9)	C(24)–O(7)	1·205 (8)
C(11)–C(13)	1·530 (10)	C(24)–O(8)	1·311 (7)
C(11)–C(14)	1·534 (9)		

O(1)–C(1)–C(2)	102·8 (5)	C(12)–C(11)–C(14)	106·7 (5)
O(1)–C(1)–C(3)	108·6 (5)	C(13)–C(11)–C(14)	109·9 (5)
O(1)–C(1)–C(4)	108·9 (6)	C(11)–C(14)–O(4)	120·7 (5)
C(2)–C(1)–C(3)	110·4 (7)	C(11)–C(14)–N(3)	116·4 (5)
C(3)–C(1)–C(4)	114·2 (7)	C(14)–N(3)–C(15)	120·7 (5)
C(2)–C(1)–C(4)	111·2 (6)	N(3)–C(15)–C(16)	111·5 (5)
C(1)–O(1)–C(5)	121·4 (4)	N(3)–C(15)–C(17)	112·7 (5)
O(1)–C(5)–O(2)	125·9 (5)	C(16)–C(15)–C(17)	112·2 (5)
O(1)–C(5)–N(1)	109·8 (5)	C(15)–C(17)–O(5)	121·6 (5)
C(5)–N(1)–C(6)	118·6 (4)	C(15)–C(17)–N(4)	115·8 (5)
C(5)–N(1)–C(9)	127·3 (5)	C(17)–N(4)–C(18)	121·3 (4)
C(6)–N(1)–C(9)	113·7 (4)	N(4)–C(18)–C(19)	106·0 (4)
N(1)–C(6)–C(7)	102·6 (4)	N(4)–C(18)–C(20)	111·9 (5)
N(1)–C(6)–C(10)	111·1 (4)	C(19)–C(18)–C(20)	109·5 (5)
C(6)–C(7)–C(8)	104·4 (5)	C(19)–C(18)–C(21)	106·9 (5)
C(7)–C(8)–C(9)	105·4 (5)	C(20)–C(18)–C(21)	111·2 (5)
C(8)–C(9)–N(1)	102·3 (4)	C(18)–C(21)–O(6)	120·7 (5)
C(7)–C(6)–C(10)	112·1 (4)	C(18)–C(21)–N(5)	116·1 (5)
C(6)–C(10)–O(3)	122·0 (5)	C(21)–N(5)–C(22)	123·1 (5)
C(6)–C(10)–N(2)	115·1 (4)	N(5)–C(22)–C(23)	109·9 (5)
C(10)–N(2)–C(11)	122·7 (5)	N(5)–C(22)–C(24)	110·7 (4)
N(2)–C(11)–C(12)	107·8 (5)	C(23)–C(22)–C(24)	109·6 (5)
N(2)–C(11)–C(13)	110·2 (5)	C(22)–C(24)–O(7)	123·0 (5)
C(12)–C(11)–C(13)	110·8 (6)	C(22)–C(24)–O(8)	113·2 (5)
N(2)–C(11)–C(14)	111·4 (5)	O(7)–C(24)–O(8)	123·8 (5)

Table 3. *Torsion angles* (°) *of pentapeptide Boc-L-Pro-Aib-L-Ala-Aib-L-Ala*-OH *as defined by IUPAC–IUB Commission on Biochemical Nomenclature* (1970)

	φ	ψ	ω
Pro^1	−57·9	+135·6	180·0
Aib^2	+56·3	+26·8	+178·5
Ala^3	+58·1	+26·9	180·0
Aib^4	+60·9	+35·9	+166·2
Ala^5	−97·2	+162·4	—

The valency-angle geometry around C^{α} (Aib) is asymmetric. Our results (110·2 and 109·9° in Aib[2] and 111·9 and 111·2° in Aib[4], respectively 107·8 and 106·7° in Aib[2] and 106·0 and 106·9° in Aib[4]) are in agreement with the observations of Paterson, Rumsey, Benedetti, Némethy & Scheraga (1981).

The unit cell contains four undefined and disordered hydrocarbon solvent molecules of weak electron densities, which are not considered in this discussion. The only intermolecular hydrogen bond connects N(2) with O(6) of a related pentapeptide molecule [N(2)···O(6)′ = 3·010 (9) Å; symmetry code: $0{\cdot}5 - x, 0{\cdot}5 + y, 1 - z$]. The resulting linear antiparallel chainlike head-to-tail arrangement parallel to [010] is illustrated in Fig. 2.

A similar head-to-tail connexion of 3_{10}-helices was observed in the related pentapeptide Boc-Aib-L-Ala-Aib-L-Ala-Aib-OMe (Bosch *et al.*, 1983) but, in contrast to (1), this peptide showed a different packing: the right-handed 3_{10}-helical single molecules formed parallel, left-handed 'superhelices' along [001]. In both cases there are only hydrophobic contacts between the resulting one-dimensional helical rods. With respect to the valency angles around C^{α}(Aib), intramolecular or intermolecular hydrogen bonding and packing, the pentapeptides (1) and (2) are very similar.

The conformation of the Boc-Pro head group of (1) is of considerable interest with respect to the peculiar pore-forming properties of the synthetic alamethicin precursor Boc-alamethicin(2–20)-OBzl, which has exactly the same N-terminal sequence as (1). The Boc-Pro group of each molecule (1) is situated like a voluminous side chain perpendicular to the helix cylinders, which are formed by the -Aib-Ala-Aib-Ala-OH segments along the *b* axis (Fig. 2). If a similar situation is maintained also for the head group of the nonadecapeptide, one could possibly explain the particular formation of only 1–2 high ionic-conductance levels (Jung, Becker, Schmitt, Voges, Boheim & Griesbach, 1984) by steric effects. The Boc-Pro[2] part and the Gln[7] side chains of Boc-alamethicin(2–20)-OBzl would be situated opposite to each other along the α-helix axis of this nonadecapeptide, which would allow only a limited number of molecules within a stable conducting aggregate.

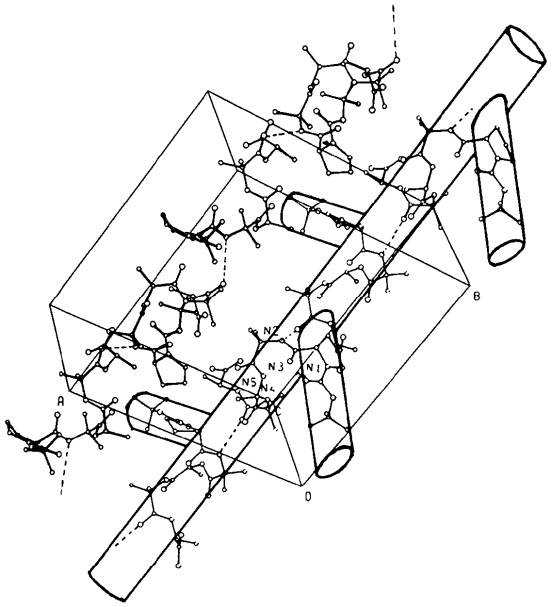

Fig. 2. Perspective view of the molecular packing (hydrogen bonds are indicated as dashed lines) and illustration of the linear helical rods together with the voluminous Boc-Pro 'side chains'.

We are grateful to the Deutsche Forschungsgemeinschaft (SFB 76) and the Fonds der Chemischen Industrie for financial support.

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Structure of the Antimicrobial Agent Cinoxacin

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(*Received* 22 *April* 1985; *accepted* 3 *September* 1985)

Abstract. 1-Ethyl-1,4-dihydro-4-oxo[1,3]dioxolo[4,5-*g*]cinnoline-3-carboxylic acid, $C_{12}H_{10}N_2O_5$, M_r = 262·2, triclinic, $P\bar{1}$, a = 6·946 (1), b = 9·119 (2), c = 9·129 (2) Å, α = 80·42 (2), β = 83·46 (2), γ = 80·66 (2)°, V = 560·4 Å^3, Z = 2, D_x = 1·552 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 10·04 cm^{-1}, $F(000)$ = 272, T = 293 K, R = 0·055 for 1345 independent reflections. A nearly planar three-ring system is observed (maximum angle between the two end rings 1·8°). The ethyl group is approximately perpendicular to the plane [N–N–C–C torsion angle 97·0 (2)°]. A hydrogen bond is formed between the carboxyl and ketone groups (O···H 1·732 Å).

Introduction. Cinoxacin is an antimicrobial agent related to oxolinic acid. It is also active, both *in vitro* (Giamarellon & Jackson, 1975) and *in vivo* (Greenwood & O'Grady, 1978), against a large variety of Gram-negative bacteria, especially against some often found in infections of the urinary tract. (*Escherichia coli, Klebsiella* sp., *Enterobacter* sp., *etc.*). It has been suggested that this is *via* interaction of the compound with the DNA-gyrase, probably a metalloenzyme (Timmers & Sternglanz, 1978).

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0108-2701/85/121825-02$01.50

Experimental. Colorless prismatic crystals grown by slow evaporation of chloroform solution. Crystal 0·10 × 0·26 × 0·14 mm. Unit cell from 15 reflections (3° < 2θ < 20°). Intensity data collected on a Nicolet *R*3*m* four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. ω-scan, 3 < 2θ < 115° (h: 0 to 7, k: −9 to 9, l: −9 to 9), variable scan rate from 4 to 30° min^{-1}, scan width 1·0°. Two monitor reflections ($1\bar{1}1$ and $01\bar{2}$) with constant intensity (variation <3%). 1345 observed reflections with $I > 2\sigma(I)$ used in structure analysis, 166 unobserved. Lorentz and polarization corrections; no correction for absorption or extinction. R_{merge} = 0·0142 from merging equivalent reflections. Structure solved by direct methods and Fourier difference methods using *SHELXTL* (Sheldrick, 1981). Refinement on F by full-matrix least-squares method; anisotropic thermal parameters for all non-hydrogen atoms. H atoms in idealized positions, fixed U = 0·06 Å^2. Weighting scheme $[\sigma^2(F_o) + G(F_o^2)]^{-1}$, where σ is the estimated standard deviation based on counting statistics and G an adjustable variable; final G = 0·0086. Max. electron density in final map <0·5 e Å^{-3}. $(\Delta/\sigma)_{max}$ = 0·122. Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed on a Nova 4S computer. Final R = 0·055 and wR = 0·073.