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

Cl(1)—C(6)	1.734 (2)	C(5)—C(10)	1.391 (4)
S(1)—O(2)	1.426 (2)	C(6)—C(7)	1.380 (4)
S(1)—O(3)	1.419 (2)	C(7)—C(8)	1.390 (4)
S(1)—N(1)	1.703 (2)	C(8)—C(9)	1.377 (4)
S(1)—C(11)	1.755 (2)	C(9)—C(10)	1.378 (4)
O(1)—C(1)	1.209 (4)	C(11)—C(12)	1.382 (4)
N(1)—C(1)	1.414 (4)	C(11)—C(16)	1.379 (4)
N(1)—C(5)	1.432 (3)	C(12)—C(13)	1.383 (4)
C(3)—C(2)	1.333 (4)	C(13)—C(14)	1.372 (4)
C(1)—C(2)	1.497 (4)	C(14)—C(15)	1.372 (4)
C(2)—C(4)	1.481 (4)	C(15)—C(16)	1.390 (4)
C(5)—C(6)	1.393 (3)		
O(2)—S(1)—O(3)	119.9 (1)	C(6)—C(5)—C(10)	118.4 (2)
O(2)—S(1)—N(1)	104.3 (1)	Cl(1)—C(6)—C(5)	120.3 (2)
O(2)—S(1)—C(11)	108.3 (1)	Cl(1)—C(6)—C(7)	118.7 (2)
O(3)—S(1)—N(1)	106.8 (1)	C(5)—C(6)—C(7)	121.0 (2)
O(3)—S(1)—C(11)	110.4 (1)	C(6)—C(7)—C(8)	119.6 (3)
S(1)—N(1)—C(1)	117.8 (2)	C(7)—C(8)—C(9)	119.8 (3)
S(1)—N(1)—C(5)	118.4 (2)	C(8)—C(9)—C(10)	120.5 (3)
C(1)—N(1)—C(5)	121.6 (2)	C(5)—C(10)—C(9)	120.6 (3)
C(1)—C(2)—C(3)	121.5 (3)	S(1)—C(11)—C(12)	120.3 (2)
C(3)—C(2)—C(4)	123.5 (3)	S(1)—C(11)—C(16)	118.4 (2)
C(1)—C(2)—C(4)	114.7 (3)	C(12)—C(11)—C(16)	121.4 (2)
O(1)—C(1)—N(1)	120.3 (3)	C(11)—C(12)—C(13)	118.7 (3)
O(1)—C(1)—C(2)	121.3 (3)	C(12)—C(13)—C(14)	120.5 (3)
N(1)—C(1)—C(2)	118.4 (3)	C(13)—C(14)—C(15)	120.7 (3)
N(1)—C(5)—C(6)	121.5 (2)	C(14)—C(15)—C(16)	119.8 (3)
N(1)—C(5)—C(10)	120.1 (2)	C(11)—C(16)—C(15)	119.1 (3)

The structure was solved by a direct method using *MULTAN*84 (Main, Germain & Woolfson, 1984). Refinements were made by block-diagonal least-squares using *HBLS*-V (Ashida, 1973). Software used to prepare material for publication included *MOLCON* (Fujii, 1979) and *ORTEP*II (Johnson, 1976). Computations were carried out at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center.

The authors thank the Research Center for Protein Engineering, Institute for Protein Research, Osaka University, for the use of the facility.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ashida, T. (1973). *HBLS*-V. *The Universal Crystallographic Computing System, Osaka*. The Computation Center, Osaka Univ., Japan.

Fujii, S. (1979). *MOLCON. The Universal Crystallographic Computing System, Osaka*. The Computation Center, Osaka Univ., Japan.

James, H. B. & Ciotti, C. J. Jr (1955). *J. Am. Chem. Soc.* **77**, 6214–6215.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kálmán, A., Czugler, M. & Argay, G. (1981). *Acta Cryst.* B**37**, 868–877.

Kashino, S., Iwamoto, T., Yamamoto, E. & Shiraga, T. (1994). *Bull. Chem. Soc. Jpn*, **67**, 1226–1231.

Main, P., Germain, G. & Woolfson, M. M. (1984). *MULTAN*84. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

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6-Chloro-1-ethyl-1,4-dihydro-4-oxo-7-(4-methyl-1-piperazinyl)-1,8-naphthyridine-3-carboxylic Acid, $C_{16}H_{19}ClN_4O_3$

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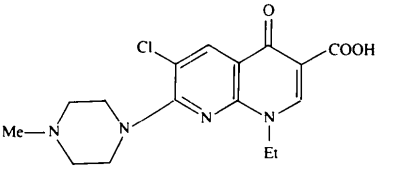
Abstract

The title compound has antibacterial properties. The piperazine fragment, possessing a chair conformation, is almost fully extended with respect to the naphthyridine ring plane, the dihedral angle between these two planes being 27.9 (3)°.

Comment

Nalidixic acid is bactericidal to most of the common gram-negative bacteria responsible for urinary tract infection (Harvey, 1975). It specifically inhibits DNA synthesis in susceptible bacterial cells (Matsumoto *et al.*, 1984). The title compound is 6,7-disubstituted nalidixic acid. It has been found that the introduction of a chloro group at the C6 position markedly influences the antibacterial activity. Also, with respect to *N*-methyl piperazinyl derivatives, introduction of the C6 substituent tends to enhance the activity against both gram-positive and gram-negative organisms (Matsumoto *et al.*, 1984). The structure determination of the title compound, (I), was undertaken to obtain a better understanding of the effect of structural and conformational change on biological activity.



(I)

Fig. 1 shows an *ORTEP*II diagram (Johnson, 1976) of the molecule with the atomic numbering scheme. The bond lengths and angles in the naphthyridine ring are normal and comparable to those in the structure of nalidixic acid (Huber, Sake Gowda & Acharya, 1980).

Each pyridine ring is planar within the limits of experimental error, but the ring fusion induces slight buckling of the ten-membered naphthyridine ring, presumably because of lone-pair repulsion. The plane of the *N*-ethyl group is almost at right angles to the naphthyridine ring system, the torsion angle C2—N1—C11—C12 being 89.2 (8)°.

The piperazine fragment in the present structure has a chair conformation characterized by the puckering parameters $Q = -0.589$ Å, $\theta = 178.8(7)°$ and $\varphi = 16.6°$ (Cremer & Pople, 1975). The four *endo*-C—N distances in this fragment are consistent with the observed mean [1.493 (3) Å] for *endo*-C—N bonds in the structure of 1-benzhydryl-4-(2-benzoylethyl)piperazinium tetrachlorocuprate(II) hydrate (Macíček, Tcholakova & Parvanova, 1993). The piperazine fragment is in an extended conformation with respect to the naphthyridine ring plane, the dihedral angle between the two planes being 27.9 (3)°.

The three-dimensional crystal structure is stabilized by non-bonded interactions.

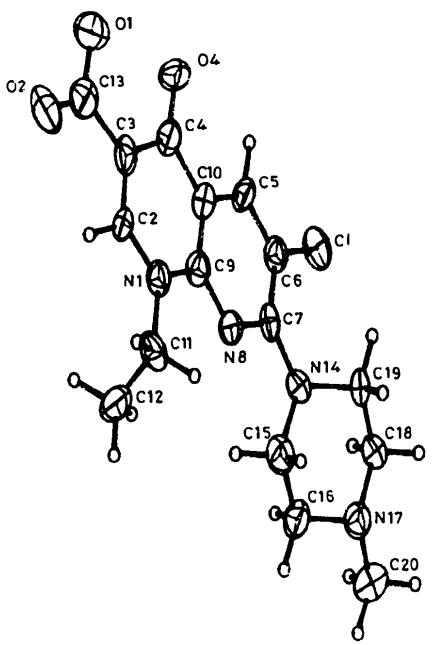


Fig. 1. A view of the molecule with 50% probability anisotropic displacement ellipsoids for the non-H atoms and atomic numbering scheme.

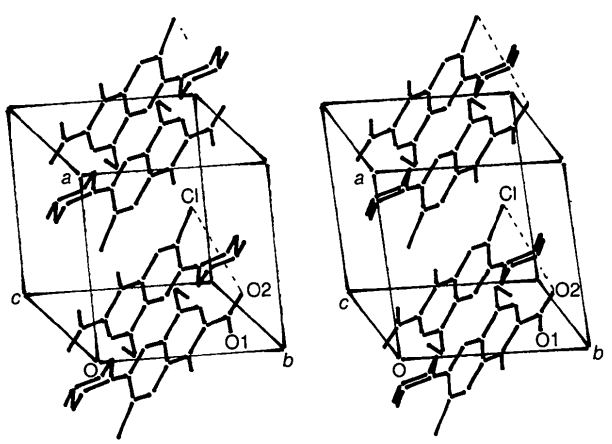


Fig. 2. Stereoview of the crystal structure.

Experimental

The compound was synthesized and supplied by Jun-ichi Matsumoto of Research Laboratories, Dainippon Pharmaceutical Co. Ltd, Japan. Transparent colourless plate-like crystals were obtained by slow evaporation from dimethylformamide solution.

Crystal data

$C_{16}H_{19}ClN_4O_3$
$M_r = 350.8$
Triclinic
$P\bar{1}$
$a = 8.876$ (3) Å
$b = 9.550$ (1) Å
$c = 10.465$ (3) Å
$\alpha = 97.25$ (2)°
$\beta = 107.37$ (3)°
$\gamma = 100.24$ (2)°
$V = 817.8$ (4) Å^3
$Z = 2$
$D_x = 1.425$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 14$–$46°$
$\mu = 2.28$ mm^{-1}
$T = 293$ K
Platelet
0.50 × 0.30 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2036 measured reflections
2036 independent reflections
1405 observed reflections [$I > 2\sigma(I)$]
$\theta_{max} = 55°$
$h = -9 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.083$
$wR = 0.089$
$S = 0.293$
1405 reflections
217 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.192046F^2]$
$(\Delta/\sigma)_{max} = 0.013$
$\Delta\rho_{max} = 0.388$ e Å^{-3}
$\Delta\rho_{min} = -0.437$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
Cl	0.4200 (2)	0.2116 (2)	0.4148 (2)	0.050 (1)
N1	0.9847 (6)	0.6930 (5)	0.6623 (5)	0.038 (2)
N8	0.8026 (6)	0.4979 (5)	0.6849 (5)	0.036 (2)

N14	0.6258 (6)	0.3170 (5)	0.7260 (5)	0.043 (2)
N17	0.5202 (6)	0.1872 (6)	0.9257 (5)	0.044 (2)
O1	1.0030 (6)	0.8048 (6)	0.2355 (5)	0.065 (2)
O2	1.1582 (7)	0.9571 (6)	0.4269 (6)	0.073 (3)
O4	0.8089 (5)	0.5812 (5)	0.2452 (4)	0.051 (2)
C2	1.0377 (8)	0.7759 (6)	0.5856 (6)	0.039 (3)
C3	0.9835 (8)	0.7460 (7)	0.4455 (7)	0.042 (3)
C4	0.8605 (7)	0.6169 (7)	0.3722 (6)	0.040 (3)
C5	0.6600 (7)	0.4150 (6)	0.4026 (6)	0.037 (3)
C6	0.6010 (7)	0.3413 (6)	0.4883 (6)	0.037 (2)
C7	0.6777 (7)	0.3818 (6)	0.6327 (6)	0.036 (3)
C9	0.8572 (7)	0.5744 (6)	0.6016 (6)	0.035 (3)
C10	0.7935 (7)	0.5352 (6)	0.4598 (6)	0.036 (2)
C11	1.0541 (8)	0.7313 (7)	0.8130 (6)	0.047 (3)
C12	0.9700 (10)	0.8257 (8)	0.8752 (7)	0.070 (4)
C13	1.0571 (9)	0.8469 (8)	0.3716 (8)	0.052 (3)
C15	0.6902 (9)	0.3871 (8)	0.8681 (7)	0.054 (3)
C16	0.5637 (9)	0.3447 (7)	0.9383 (7)	0.052 (3)
C18	0.4538 (8)	0.1196 (7)	0.7814 (6)	0.045 (3)
C19	0.5779 (8)	0.1571 (6)	0.7116 (7)	0.046 (3)
C20	0.3996 (9)	0.1520 (9)	0.9936 (8)	0.064 (3)

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

Cl—C6	1.738 (5)	C13—O1	1.341 (9)
N1—C2	1.321 (9)	C13—O2	1.203 (8)
N1—C9	1.379 (7)	N14—C15	1.446 (8)
N1—C11	1.481 (8)	N14—C19	1.486 (7)
C4—O4	1.250 (7)	C16—N17	1.464 (9)
C7—N8	1.345 (7)	N17—C18	1.460 (7)
C7—N14	1.368 (9)	N17—C20	1.466 (11)
N8—C9	1.349 (9)		
C9—N1—C11	119.7 (5)	N1—C11—C12	113.4 (6)
C2—N1—C11	120.7 (5)	C3—C13—O2	124.0 (7)
C2—N1—C9	119.6 (5)	C3—C13—O1	114.6 (7)
N1—C2—C3	124.4 (6)	O1—C13—O2	121.4 (7)
C3—C4—O4	123.5 (6)	C7—N14—C19	122.8 (5)
C10—C4—O4	122.5 (6)	C7—N14—C15	119.2 (5)
Cl—C6—C5	117.2 (5)	C15—N14—C19	111.0 (5)
Cl—C6—C7	122.1 (5)	N14—C15—C16	109.5 (6)
C6—C7—N14	124.6 (5)	C15—C16—N17	110.5 (6)
C6—C7—N8	119.6 (5)	C16—N17—C20	108.6 (6)
N8—C7—N14	115.6 (5)	C16—N17—C18	109.2 (5)
C7—N8—C9	120.3 (5)	C18—N17—C20	110.4 (6)
N1—C9—N8	117.1 (5)	N17—C18—C19	110.3 (5)
N8—C9—C10	122.7 (6)	N14—C19—C18	109.7 (5)
N1—C9—C10	120.2 (6)		
C9—N1—C11—C12	−87.0 (7)	C6—C7—N14—C19	45.7 (9)
C2—N1—C11—C12	89.2 (8)	N8—C7—N14—C15	8.7 (8)
C6—C7—N14—C15	−166.3 (6)	N8—C7—N14—C19	−139.4 (6)

Refinement was by full-matrix least squares methods. Of the 19 H atoms, 18 were calculated and not refined.

Programs used to solve structure: *MULTAN*78 (Main *et al.*, 1978). Programs used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used for geometrical calculations and to prepare material for publication: *PARST* (Nardelli, 1983). All calculations were performed on a Super 32 computer (VECC, Calcutta).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1004). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Harvey, S. C. (1975). *The Pharmacological Basis of Therapeutics*, 5th ed., edited by L. S. Goodman & A. Gilman, pp. 987–1017. New York: Macmillan.

Huber, C. P., Sake Gowda, D. S. & Acharya, K. R. (1980). *Acta Cryst.* B**36**, 497–499.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Macíček, J., Tcholakova, J. & Parvanova, M. (1993). *Acta Cryst.* C**49**, 788–790.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Matsumoto, J., Miyamoto, T., Minamida, A., Nishimura, Y., Egawa, H. & Nishimura, H. (1984). *J. Med. Chem.* **27**, 292–301.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination.* Univ. of Cambridge, England.

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Methyl (2*S*,6*S*:2*R*,6*R*)-6-(2-Cyanoethyl)-4,6-dimethyl-2-morpholineacetate

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Abstract

The morpholine ring of the title compound, $C_{12}H_{20}N_2O_3$, adopts a chair conformation with an equatorial (methoxycarbonyl)methyl group. The cyanoethyl and (methoxycarbonyl)methyl groups are *trans* with respect to each other. The global minimum conformation, as computed by *PCMODEL* [Gajewski & Gilbert (1992). *Molecular Modeling Package*. Version 4.0], of the title compound agrees with that observed in the crystal. In the crystal, the torsion angles (N≡)C—CH_2—CH_2—C(O), (N≡C)CH_2—CH_2—C—O, O—CH—CH_2—C(OCH_3) and (O)CH—CH_2—C(O)—O(CH_3) have values −170.0 (1), −45.9 (2), −71.6 (2) and 142.8 (1)°, respectively.

Comment

As a part of an effort to synthesize the four possible stereoisomers of our reaction-intermediate analogues for carnitine acyltransferases (Gandour, Blackwell, Colucci, Chung, Bieber, Ramsay, Brass & Fronczek, 1992), we prepared the title compound, (I). We undertook the structure determination to assign the relative stereochemistry of this racemate and, by inference, that of the diastereomer. This isomer has a