$wR = 0,031$
$S = 0,21$
973 réflexions
146 paramètres
Toutes les paramètres des atomes d'hydrogène affinées
$\Delta\rho_{max} = 0,2$ e Å^{-3}
$\Delta\rho_{min} = -0,2$ e Å^{-3}
Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974, Tome IV, Tableau 2.2B)

Tableau 1. *Coordonnées atomiques et facteurs d'agitation thermique isotrope équivalents* (Å^2)

$B_{éq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{éq}$
O(1)	1,1236 (4)	0,0781 (2)	−0,3226 (3)	2,2 (1)
O(2)	0,6125 (4)	0,3994 (3)	−0,0096 (3)	2,9 (1)
O(3)	0,7794 (5)	0,2968 (3)	−0,3931 (3)	2,7 (1)
O(4)	1,1590	0,4624 (3)	0,0054	2,8 (1)
N	1,0376 (5)	0,1069 (3)	−0,1064 (3)	1,8 (1)
C(1)	1,4103 (7)	−0,2121 (4)	−0,2154 (4)	3,5 (2)
C(2)	1,2944 (5)	−0,1280 (3)	−0,1405 (3)	2,2 (2)
C(3)	1,1424 (5)	0,0301 (3)	−0,2009 (4)	2,1 (2)
C(4)	0,8873 (5)	0,2602 (3)	−0,1285 (3)	1,6 (1)
C(5)	1,0195 (5)	0,4322 (3)	−0,1319 (3)	2,2 (2)
C(6)	0,6997 (6)	0,2430 (4)	−0,2707 (4)	2,5 (2)
C(7)	0,7595 (6)	0,2514 (3)	−0,0028 (4)	1,9 (2)

Tableau 2. *Paramètres géométriques* (Å, °)

C(1)—C(2)	1,269 (4)	C(4)—C(6)	1,591 (4)
C(2)—C(3)	1,546 (4)	C(4)—C(7)	1,557 (4)
C(3)—O(1)	1,205 (3)	C(5)—O(4)	1,430 (3)
C(3)—N	1,337 (4)	C(6)—O(3)	1,415 (4)
C(4)—N	1,460 (4)	C(7)—O(2)	1,421 (4)
C(4)—C(5)	1,530 (4)		
C(1)—C(2)—C(3)	121,9 (3)	C(6)—C(4)—N	111,8 (2)
C(2)—C(3)—O(1)	121,9 (3)	C(6)—C(4)—C(7)	106,4 (2)
C(2)—C(3)—N	114,0 (2)	C(7)—C(4)—N	104,0 (2)
N—C(3)—O(1)	124,1 (3)	C(4)—C(5)—O(4)	109,2 (2)
C(5)—C(4)—N	111,6 (2)	C(4)—C(6)—O(3)	112,7 (2)
C(5)—C(4)—C(6)	109,5 (2)	C(4)—C(7)—O(2)	109,4 (2)
C(5)—C(4)—C(7)	113,2 (2)	C(3)—N—C(4)	127,6 (3)
C(1)—C(2)—C(3)—O(1)	1,6 (2)	C(5)—C(4)—C(7)—O(2)	56,4 (2)
C(1)—C(2)—C(3)—N	−178,0 (3)	C(6)—C(4)—C(5)—O(4)	169,8 (2)
C(2)—C(3)—N—C(4)	178,6 (1)	C(6)—C(4)—C(7)—O(2)	−64,0 (2)
C(3)—N—C(4)—C(5)	−75,1 (2)	C(7)—C(4)—C(5)—O(4)	51,2 (1)
C(3)—N—C(4)—C(6)	48,0 (2)	C(7)—C(4)—C(6)—O(3)	163,6 (3)
C(3)—N—C(4)—C(7)	162,5 (3)	N—C(4)—C(5)—O(4)	−65,8 (1)
C(4)—N—C(3)—O(1)	−1,0 (1)	N—C(4)—C(6)—O(3)	−83,4 (2)
C(5)—C(4)—C(6)—O(3)	40,9 (2)	N—C(4)—C(7)—O(2)	177,8 (3)

Pour definir l'origine dans le plan de glissement, l'atome O(4) est bloqué suivant x et z. Les facteurs d'agitation thermique des atomes d'hydrogène ont été fixés.

Collection des données: *CAD-4 Software* (Enraf–Nonius, 1977). Affinement des paramètres de la maille: *CAD-4 Software*. Réduction des données: programme élaboré au laboratoire du CRMC2. Programme(s) pour la solution de la structure: *MULTAN*80 (Main *et al.*, 1980). Programme(s) pour l'affinement de la structure: *SHELX*76 (Sheldrick, 1976). Les dessins ont été obtenus à l'aide de *ORTEP* (Johnson, 1965).

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Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances et angles des atomes d'hydrogène, ont été déposées au dépôt d'archives de l'UICr (Référence: DU1031). On peut en obtenir des copies en s'adressant à: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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N-Phenylsulfonyl-*N*-*o*-chlorophenylmethacrylamide, $C_{16}H_{14}ClNO_3S$

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Abstract

N-Phenylsulfonyl-*N*-*o*-chlorophenylmethacrylamide was obtained as a by-product in the synthesis of *N*-*o*-chlorophenylmethacrylamide. The geometry of the

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amide group substituted by the *N*-sulfonyl group and the conformation around the S^{IV}—N bond have been studied.

Comment

The N(1)—C(1) bond length in the title compound, (I), is significantly longer than the 1.32–1.35 Å usually found in amides (Kashino, Iwamoto, Yamamoto & Shiraga, 1994). The C(5)—N(1)—C(1)—C(2) torsion angle is −32.9 (4)°, and S(1)—N(1)—C(1)—O(1) is −13.4 (4)°, both deviating significantly from the value of 0° for the ideal *cis* conformation. The N(1)—S(1) bond length 1.703 (2) Å is close to that found in *N*-substituted arylsulfonamides (Kálmán, Czugler & Argay, 1981). The C(11)—S(1)—N(1)—C(5) torsion angle is −91.4 (2)°, while C(11)—S(1)—N(1)—C(1) is 72.0 (2)°. The significant deviation of the latter from 90° is compatible with the intramolecular repulsion between O(1) and O(3) [O(1)···O(3) 2.807 (3) Å].

(I)

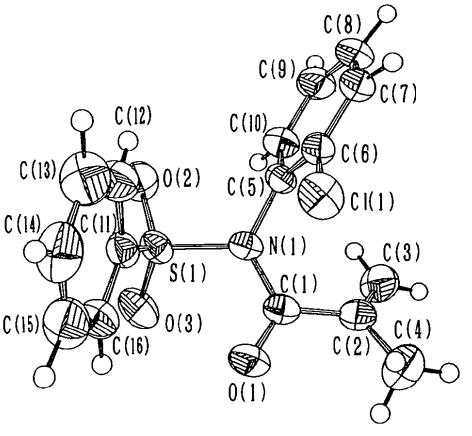

Fig. 1. The displacement ellipsoids with atomic numbering. Ellipsoids of 50% probability are drawn for the non-H atoms; the H atoms are represented as spheres equivalent to B = 1.0 Å^2.

Experimental

The compound was obtained as a by-product in the preparation of *N*-*o*-chlorophenylmethacrylamide (Kashino *et al.*, 1994) by the method described by James & Ciotti (1955). The crystals were grown from a benzene solution of the reaction mixture by slow evaporation. The density D_m was measured by flotation in aqueous KI solution.

Crystal data

$C_{16}H_{14}ClNO_3S$
M_r = 335.81
Monoclinic
$P2_1/n$
a = 14.369 (1) Å
b = 13.5367 (7) Å
c = 8.2496 (7) Å
β = 102.330 (9)°
V = 1567.6 (2) Å^3
Z = 4
D_x = 1.422 Mg m^{-3}
D_m = 1.42 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 20 reflections
θ = 17.5–20.5°
μ = 3.36 mm^{-1}
T = 298 K
Prismatic along c
0.25 × 0.20 × 0.18 mm
Colorless

Data collection

Rigaku AFC-5 diffractometer
ω–2θ scans
Absorption correction: none
2831 measured reflections
2509 independent reflections
2340 observed reflections [$F > \sigma(F)$]
R_{int} = 0.016
θ_{max} = 62.5°
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 97 reflections
intensity decay: 1%

Refinement

Refinement on F
R = 0.047
wR = 0.060
S = 1.412
2340 reflections
256 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.0003|F_o| + 0.0009|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.59
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: secondary extinction
Extinction coefficient: 3.325 × 10^{-6}
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)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Cl(1)	0.15282 (5)	0.10133 (5)	0.30558 (9)	4.54 (3)
S(1)	0.24132 (4)	0.30172 (4)	0.00470 (7)	3.36 (3)
O(1)	0.3739 (1)	0.1606 (1)	0.1832 (2)	4.57 (9)
O(2)	0.1701 (1)	0.3768 (1)	−0.0173 (2)	4.60 (9)
O(3)	0.3310 (1)	0.3216 (1)	−0.0343 (2)	5.11 (9)
N(1)	0.2626 (1)	0.2755 (1)	0.2112 (2)	3.12 (8)
C(1)	0.3380 (1)	0.2099 (2)	0.2757 (3)	3.3 (1)
C(2)	0.3742 (1)	0.2062 (2)	0.4598 (3)	3.6 (1)
C(3)	0.3756 (2)	0.2865 (2)	0.5539 (3)	4.6 (1)
C(4)	0.4179 (2)	0.1109 (2)	0.5233 (4)	5.2 (1)
C(5)	0.1900 (1)	0.2980 (2)	0.3005 (3)	2.95 (9)
C(6)	0.1362 (1)	0.2239 (2)	0.3536 (3)	3.2 (1)
C(7)	0.0709 (2)	0.2458 (2)	0.4490 (3)	4.3 (1)
C(8)	0.0567 (2)	0.3435 (2)	0.4895 (3)	5.0 (1)
C(9)	0.1074 (2)	0.4176 (2)	0.4333 (3)	4.5 (1)
C(10)	0.1736 (2)	0.3954 (2)	0.3401 (3)	3.7 (1)
C(11)	0.1908 (2)	0.1948 (2)	−0.0983 (3)	3.3 (1)
C(12)	0.2465 (2)	0.1297 (2)	−0.1651 (3)	4.4 (1)
C(13)	0.2042 (2)	0.0463 (2)	−0.2456 (4)	5.4 (1)
C(14)	0.1088 (2)	0.0290 (2)	−0.2589 (4)	5.4 (1)
C(15)	0.0535 (2)	0.0946 (2)	−0.1945 (4)	5.7 (2)
C(16)	0.0946 (2)	0.1788 (2)	−0.1129 (4)	4.7 (1)

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.

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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.

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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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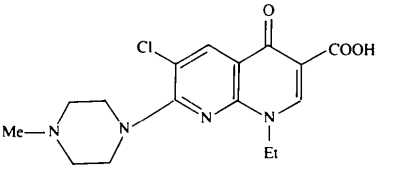
Abstract

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).