C(11)	0.3472 (4)	0.0899 (5)	0.1358 (6)	0.053
C(12)	0.2928 (4)	0.1712 (5)	0.1223 (6)	0.050
C(13)	0.2665 (4)	0.1974 (5)	0.0258 (7)	0.056
C(14)	0.2938 (5)	0.1410 (5)	−0.0549 (7)	0.059
C(15)	0.3772 (7)	0.0612 (8)	0.2395 (7)	0.073
C(16)	0.2126 (6)	0.3005 (7)	0.1968 (9)	0.078

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

O(1)—N	1.213 (8)	O(2)—N	1.228 (7)
O(3)—C(12)	1.351 (7)	O(3)—C(16)	1.413 (9)
N—C(1)	1.471 (9)	C(1)—C(2)	1.376 (10)
C(1)—C(6)	1.365 (10)	C(2)—C(3)	1.365 (9)
C(3)—C(4)	1.389 (8)	C(4)—C(5)	1.420 (10)
C(4)—C(7)	1.463 (9)	C(5)—C(6)	1.357 (10)
C(7)—C(8)	1.315 (8)	C(8)—C(9)	1.481 (10)
C(9)—C(10)	1.378 (9)	C(9)—C(14)	1.367 (9)
C(10)—C(11)	1.370 (9)	C(11)—C(12)	1.402 (8)
C(11)—C(15)	1.513 (10)	C(12)—C(13)	1.399 (9)
C(13)—C(14)	1.387 (9)		
C(12)—O(3)—C(16)	118.0 (7)	O(1)—N—O(2)	124.3 (7)
O(1)—N—C(1)	119.2 (7)	O(2)—N—C(1)	116.5 (8)
N—C(1)—C(2)	120.2 (7)	N—C(1)—C(6)	118.4 (7)
C(2)—C(1)—C(6)	121.5 (7)	C(3)—C(2)—C(1)	119.6 (7)
C(2)—C(3)—C(4)	121.0 (7)	C(3)—C(4)—C(5)	117.5 (6)
C(3)—C(4)—C(7)	123.1 (6)	C(5)—C(4)—C(7)	119.4 (6)
C(4)—C(5)—C(6)	121.1 (7)	C(1)—C(6)—C(5)	119.3 (7)
C(4)—C(7)—C(8)	126.5 (7)	C(7)—C(8)—C(9)	127.2 (7)
C(8)—C(9)—C(10)	118.7 (6)	C(8)—C(9)—C(14)	123.6 (7)
C(10)—C(9)—C(14)	117.7 (8)	C(9)—C(10)—C(11)	123.5 (7)
C(10)—C(11)—C(12)	118.0 (6)	C(10)—C(11)—C(15)	121.4 (7)
C(12)—C(11)—C(15)	120.5 (7)	O(3)—C(12)—C(11)	116.0 (6)
O(3)—C(12)—C(13)	124.2 (6)	C(11)—C(12)—C(13)	119.8 (6)
C(12)—C(13)—C(14)	119.1 (7)	C(9)—C(14)—C(13)	121.9 (7)

O(3)—C(12)—C(11)—C(10)	178.1 (10)
O(3)—C(12)—C(13)—C(14)	−178.6 (11)
O(2)—N—C(1)—C(6)	170.4 (11)
O(1)—N—C(1)—C(6)	−7.8 (8)
N—C(1)—C(6)—C(5)	−179.4 (12)
C(3)—C(4)—C(7)—C(8)	1.5 (8)
C(5)—C(4)—C(7)—C(8)	−179.8 (13)
C(7)—C(8)—C(9)—C(10)	−175.9 (13)
C(8)—C(9)—C(10)—C(11)	178.2 (12)
C(13)—C(12)—C(11)—C(15)	179.2 (1)
C(16)—O(3)—C(12)—C(11)	−178.7 (9)
O(3)—C(12)—C(11)—C(15)	−1.0 (7)
O(2)—N—C(1)—C(2)	−10.5 (8)
O(1)—N—C(1)—C(2)	171.3 (12)
N—C(1)—C(2)—C(3)	179.8 (12)
C(2)—C(3)—C(4)—C(7)	−179.4 (11)
C(4)—C(7)—C(8)—C(9)	−179.1 (15)
C(6)—C(5)—C(4)—C(7)	179.7 (12)
C(7)—C(8)—C(9)—C(14)	4.8 (9)
C(8)—C(9)—C(14)—C(13)	−178.9 (12)
C(15)—C(11)—C(10)—C(9)	−178.5 (13)
C(16)—O(3)—C(12)—C(13)	1.0 (7)

Space group and approximate cell dimensions of the crystal were determined using Weissenberg and precession photography (Suh, Suh, Ko, Aoki & Yamazaki, 1988). The systematic absences *hkl* for $k+l = 2n$, *0kl* for $k,l = 2n$, *h0l* for $h,l = 2n$ *hk0* for $k = 2n$, *h00* for $h = 2n$ and *0k0* for $k = 2n$ uniquely defined the non-centrosymmetric orthorhombic space group *Aba2*. Data were corrected for Lorentz and polarization effects. The structure was solved by the application of direct methods with *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and refined by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), with anisotropic displacement parameters for all the non-H atoms. All H atoms were located from difference Fourier maps. Geometric calculations were performed using *GEOM* (Shin, 1978). All computations were performed using the MicroVAX 3400 computer at Chungnam National University.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HL1045). 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-4(1*H*)-cinnolinone

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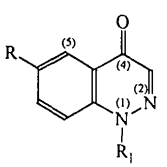
Abstract

The title compound, $C_8H_5ClN_2O$, crystallized with two molecules in the asymmetric unit. The geometries of the independent molecules are similar to within 2σ limits and closely resemble the geometries observed for unsubstituted and 1-alkyl-substituted

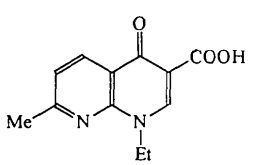
4(1*H*)-cinnolinones. Packing is determined by the planarity of the molecules and by two intermolecular hydrogen bonds, which join molecules into infinite chains running along [001].

Comment

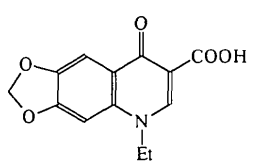
The structures of 4-cinnolinones, which are similar to those of 4-oxonaphthalene, naphthyridine, quinoline and cinnoline derivatives, are of interest because they form the cores of many pharmacologically active compounds. Many exibit considerable antibacterial activity, mainly against gram-negative bacteria [this is related to the 4-oxopyridine-3-carboxylic acid moiety (Timmers & Sternglanz, 1978)]. Small alkyl substituents at position 1 are of structural importance. Several 4(1*H*)-cinnolinones and their important analogues have been studied by X-ray diffraction methods: 4(1*H*)-cinnolinone, (2), and 1-methyl-4(1*H*)-cinnolinone, (3) (Palmer, Gould, Blake, Smith, Stephenson & Ames, 1987), cinoxacin (Rosales, Toscano, Barba-Behrens & García, 1985), nalidixic acid (Achari & Neidle, 1976; Huber, Godwa & Acharya, 1980), oxolinic acid (Cygler & Huber, 1985) and 5-aminooxolinic acid (Czugler, Argay, Frank, Mészáros, Kutschabsky & Reck, 1976). The present study was undertaken to broaden the structural data on 4-cinnolinones.



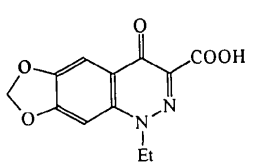
	R	R_1
(1)	Cl	H
(2)	H	H
(3)	H	CH_3
(4)	H	$CH_2CH_2C(O)NHNH_2$
(5)	H	$CH_2C(O)OCH_2CH_3$



Nalidixic acid



Oxolinic acid



Cinoxacin

Bond lengths and angles in the 4(1*H*)-cinnolinone frame are similar to those in all known structures (Table 2). The largest differences are found for the N(1)—N(2) distance (0.03 Å) and for the bond angle at N(1) (2.3°), and are obviously related to 1-substitution. Both symmetry-independent molecules of the title compound (1) have similar geometry within 2σ limits. The packing is determined by planarity of the molecules and by two intermolecular hydrogen bonds, which join molecules into infinite chains running along [001]. Hydrogen-bonding distances and angles are 2.769 (4) and 2.762 (4) Å (N···O), 1.810 (4) and 1.814 (4) Å (H···O), 176.1 (1) and 168.9 (1)° (N—H···O) for contacts between molecules *A* and *B*, and *A* and *B'* ($\frac{3}{2} - x$, $2 - y$, $z - \frac{1}{2}$), respectively.

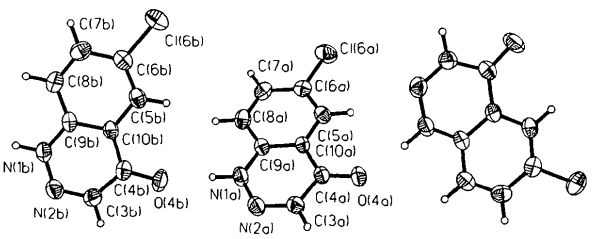


Fig. 1. Atom-numbering scheme, general view of molecules *A* and *B* and hydrogen bonding. [Unlabelled molecule is *B'* at ($\frac{3}{2} - x$, $2 - y$, $z - \frac{1}{2}$).]

Experimental

Crystal data

$C_8H_5ClN_2O$
$M_r = 180.59$
Orthorhombic
$P2_12_12_1$
$a = 3.7930$ (6) Å
$b = 15.704$ (2) Å
$c = 25.035$ (2) Å
$V = 1491.2$ Å^3
$Z = 8$
$D_x = 1.608$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 18$–$26°$
$\mu = 4.15$ mm^{-1}
$T = 293$ K
Prisms
0.22 × 0.14 × 0.11 mm
Yellow
Crystal source: recrystallized from methanol

Data collection

Kuma KM-4 diffractometer
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.574$, $T_{max} = 0.754$
2905 measured reflections
2339 independent reflections
2165 observed reflections [$F_o > 4\sigma(F_o)$]
$R_{int} = 0.0305$
$\theta_{max} = 75°$
$h = 0 \rightarrow 4$
$k = -19 \rightarrow 19$
$l = 0 \rightarrow 31$
2 standard reflections monitored every 100 reflections
intensity variation: < 3%

Refinement

$R = 0.043$
$wR = 0.058$
$S = 0.77$
2165 reflections
228 parameters
$w = 1/[\sigma^2(F_o) + 0.00186F_o^2]$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1990)
Extinction coefficient: 0.0032 (6)
Atomic scattering factors from *SHELXTL* (Sheldrick, 1990)

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

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

	x	y	z	U_{eq}
N(1*A*)	0.5607 (10)	1.0724 (2)	0.8880 (1)	0.0385 (10)
N(2*A*)	0.6969 (11)	1.1484 (2)	0.8978 (1)	0.0439 (11)
C(3*A*)	0.7488 (12)	1.1699 (2)	0.9475 (1)	0.0402 (13)
C(4*A*)	0.6622 (12)	1.1177 (2)	0.9932 (1)	0.0333 (11)
O(4*A*)	0.7224 (9)	1.1436 (2)	1.0392 (1)	0.0475 (10)
C(5*A*)	0.4060 (11)	0.9776 (2)	1.0199 (1)	0.0332 (11)
C(6*A*)	0.2611 (11)	0.9020 (2)	1.0047 (1)	0.0353 (12)
Cl(6*A*)	0.1328 (4)	0.82896 (7)	1.05250 (4)	0.0514 (4)
C(7*A*)	0.2120 (11)	0.8812 (3)	0.9506 (2)	0.0384 (12)
C(8*A*)	0.3108 (11)	0.9377 (3)	0.9119 (1)	0.0372 (12)
C(9*A*)	0.4575 (10)	1.0156 (2)	0.9265 (1)	0.0301 (10)
C(10*A*)	0.5077 (10)	1.0368 (2)	0.9803 (1)	0.0295 (11)
N(1*B*)	0.6367 (11)	0.9169 (2)	0.6406 (1)	0.0406 (10)
N(2*B*)	0.4892 (10)	0.9930 (2)	0.6413 (1)	0.0443 (11)
C(3*B*)	0.4377 (13)	1.0292 (3)	0.6880 (2)	0.0421 (13)
C(4*B*)	0.5405 (11)	0.9935 (3)	0.7384 (1)	0.0365 (12)
O(4*B*)	0.4825 (10)	1.0321 (2)	0.7810 (1)	0.0568 (12)
C(5*B*)	0.8195 (11)	0.8661 (3)	0.7805 (1)	0.0354 (11)
C(6*B*)	0.9667 (11)	0.7886 (3)	0.7746 (2)	0.0368 (12)
Cl(6*B*)	1.1145 (3)	0.73367 (7)	0.83065 (4)	0.0532 (4)
C(7*B*)	1.0094 (11)	0.7507 (3)	0.7240 (2)	0.0423 (13)
C(8*B*)	0.8979 (13)	0.7931 (3)	0.6795 (1)	0.0416 (13)
C(9*B*)	0.7492 (10)	0.8740 (3)	0.6848 (1)	0.0338 (11)
C(10*B*)	0.7012 (10)	0.9112 (2)	0.7352 (1)	0.0322 (11)

Table 2. *Selected geometric parameters* (Å, °) *for 4(1H)-cinnolinones and their 1-alkyl derivatives*

	(1)[a]	(2)[b]	(3)[c]	(4)[d]	(5)[e]
N(1)—N(2)	1.323 (5)	1.329 (3)	1.340 (3)	1.350 (4)	1.342 (3)
	1.319 (5)		1.332 (3)		
N(2)—C(3)	1.305 (5)	1.303 (4)	1.300 (4)	1.292 (4)	1.296 (4)
	1.313 (5)		1.293 (3)		
C(3)—C(4)	1.445 (5)	1.434 (4)	1.425 (4)	1.441 (5)	1.442 (5)
	1.435 (5)		1.426 (4)		
N(1)—C(9)	1.370 (5)	1.364 (3)	1.368 (3)	1.375 (4)	1.376 (3)
	1.364 (5)		1.368 (3)		
C(4)—C(10)	1.435 (5)	1.457 (4)	1.448 (4)	1.454 (5)	1.442 (4)
	1.433 (6)		1.443 (3)		
C(9)—N(1)—N(2)	124.6 (3)	124.2 (2)	122.8 (2)	122.4 (2)	123.3 (2)
	124.7 (3)		123.0 (2)		
N(1)—N(2)—C(3)	117.9 (3)	117.8 (2)	119.2 (2)	119.3 (3)	118.2 (3)
	117.9 (3)		119.1 (2)		
N(2)—C(3)—C(4)	125.1 (4)	125.9 (3)	125.2 (3)	125.5 (3)	126.1 (3)
	124.9 (4)		125.3 (2)		
C(3)—C(4)—C(10)	114.7 (3)	114.2 (2)	114.2 (2)	113.9 (3)	114.1 (2)
	114.7 (3)		114.3 (2)		

Notes: (*a*) this study; (*b*) 4(1*H*)-cinnolinone (Palmer *et al.*, 1987); (*c*) 1-methyl-4(1*H*)-cinnolinone (Palmer *et al.*, 1987); (*d*) 3-(4-cinnolinon-1-yl)propanoic acid hydrazide (Główka, Iwanicka & Stańczak, 1994*b*); (*e*) 1-(ethoxycarbonylmethyl)-4(1*H*)-cinnolinone (Główka, Iwanicka & Stańczak, 1994*a*).

H atoms were treated as rigid aromatic C—H groups with C—H distances constrained at calculated values 0.96 Å. *SHELXTL* (Sheldrick, 1990) was used throughout the structure determination.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: KA1066). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3′-(2,6-Dichlorophenyl)bicyclo[2.2.1]-heptane-2-spiro-5′(4′*H*)-isoxazole-3-one

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Abstract

The title compound, $C_{15}H_{13}Cl_2NO_2$, resulted from a 1,3-dipolar cycloaddition (a class of reactions of significant importance in heterocyclic chemistry). The isoxazole ring atoms are coplanar to within 0.05 (1) Å and the cyclohexanone ring of the norbornanone moiety adopts the expected boat form with the two five-membered rings in envelope conformations. The orientation of the isoxaxole ring to the cyclohexanone ring is given by the