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}
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(1*H*)*-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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Acta Cryst. (1994). **C50**, 1772–1774

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

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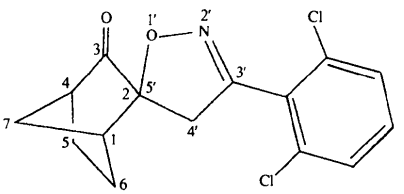
torsion angles O1′—C2—C3—O3 71.7 (4) and C4′—C2—C1—C6 −64.0 (4)°. The dihedral angle between the isoxazole and 2,6-dichlorophenyl rings is 73.3 (3)°.

Comment

The structure determination was performed to define the conformation of the title compound (1), the major of two isomeric products formed by a dipolar cycloaddition between 2,6-dichlorobenzonitrile oxide and 3-methylene-2-norbornanone, according to a previously reported method (Kelly-Basetti, Mackay, Pereira, Savage & Simpson, 1994). A perspective view of the 1*R*,2*S*,4*S* enantiomer is illustrated in Fig. 1.



(I)

The boat conformation of the cyclohexanone ring is fairly regular, with the asymmetry parameter (Duax & Norton, 1975) C_s^1 5.5°, while the pseudo-rotation parameters (Altona, Geise & Romers, 1968) are Δ = −34.2°, ψ = 57.6° for the cyclopentanone ring and Δ = 24.5°, ψ = 58.4° for the cyclopentane ring. The bond lengths and angles are given in Table 2. In the isoxazole ring, the O1′—N2′ bond length of 1.409 (4) Å, the N2′—C3′ bond length of 1.272 (4) Å and the N2—O1′—C2 and O1′—N2′—C3′ bond angles of 109.5 (2) and 109.2 (2)°, respectively, are typical for such systems. For example, the values for these bond lengths and angles in two other spiro Δ^2-isoxazolines were found to be 1.415 (4), 1.270 (5) Å and 109.2 (2), 109.9 (2)°, respectively (Pereira, Savage, Simpson, Greenwood & Mackay, 1993), and 1.429 (4), 1.272 (5) Å and 108.2 (2), 109.7 (2)°, respectively (Kelly-Basetti, Mackay, Pereira, Savage & Simpson, 1994). In the norbornanone moiety, the angle at C7 is contracted from the regular tetrahedral value to 95.3 (3)°, while the endocyclic angles of the five-membered rings at C1 and C4 also are significantly less than 109.5°, ranging in value from 100.9 (3) to 101.8 (3)°. Values very similar to these have been reported for two spiro-norbornane derivatives by Bordeaux, Gagnaire & Lajzerowicz (1983) [94.2 (3) and 94.8 (6)° for the angle at C7 and values in the ranges 101.2 (3)–102.3 (3) and 100.7 (6)–101.7 (6)° for the endocyclic angles at C1 and C4]. There being no hydrogen-bonding donors in the structure, the molecules are held together in the crystal by van der Waals interactions only. There is one close intermolecular approach in the structure. This is between the carbonyl O atoms [O3···O3($-x, -y, 2-z$) 3.095 (3) Å].

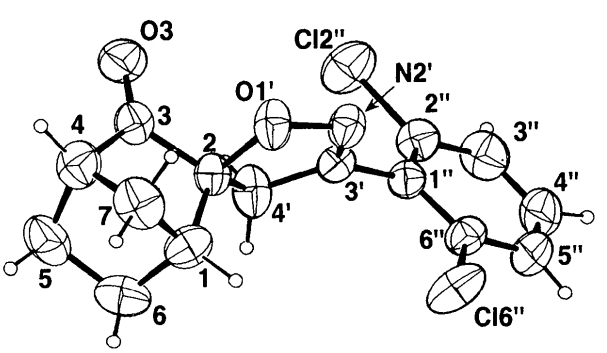


Fig. 1. The molecular structure of the (1*R*,2*S*,4*S*)-enantiomer with displacement ellipsoids scaled to 50% probability. The C symbols are omitted and the H atoms are denoted by spheres of arbitrary radii.

Experimental

Crystal data

$C_{15}H_{13}Cl_2NO_2$
M_r = 310.2
Monoclinic
$P2_1/c$
a = 7.261 (1) Å
b = 16.913 (2) Å
c = 11.574 (1) Å
β = 94.85 (1)°
V = 1416.3 (5) Å^3
Z = 4
D_x = 1.454 Mg m^{-3}
D_m = 1.450 (5) Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 18–33°
μ = 4.02 mm^{-1}
T = 291 (1) K
Prismatic
0.26 × 0.38 × 0.38 mm
Colourless
Crystal source: ethanol solution

Data collection

Rigaku AFC diffractometer
$\omega/2\theta$ scans
Absorption correction: *SHELX*76 (Sheldrick, 1976)
T_{min} = 0.230, T_{max} = 0.421
2346 measured reflections
2218 independent reflections
1734 observed reflections [$F > 4.0\sigma(F)$]

R_{int} = 0.035
θ_{max} = 65.0°
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 13$
3 standard reflections monitored every 50 reflections
intensity variation: <3%

Refinement

Refinement on F
R = 0.046
wR = 0.066
S = 1.55
1734 reflections
234 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + 0.00118F^2]$
$(\Delta/\sigma)_{max}$ = 0.004

$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELX*76 (Sheldrick, 1976)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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

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

	x	y	z	B_{eq}
C1	0.2177 (6)	0.1990 (2)	0.9675 (4)	4.5 (1)
C2	0.1581 (5)	0.1276 (2)	0.8919 (3)	3.3 (1)
C3	−0.0271 (5)	0.1068 (2)	0.9404 (3)	3.5 (1)
O3	−0.1219 (4)	0.0500 (1)	0.9115 (2)	4.8 (1)
C4	−0.0628 (6)	0.1701 (2)	1.0257 (4)	4.8 (1)
C5	0.0699 (6)	0.1509 (3)	1.1339 (4)	5.4 (1)
C6	0.2610 (6)	0.1701 (3)	1.0925 (4)	5.6 (1)
C7	0.0330 (6)	0.2407 (2)	0.9750 (5)	5.2 (1)
O1′	0.1074 (3)	0.1548 (1)	0.7725 (2)	4.1 (1)
N2′	0.2302 (4)	0.1220 (2)	0.6976 (2)	3.6 (1)
C3′	0.3350 (4)	0.0724 (2)	0.7535 (3)	3.0 (1)
C4′	0.2960 (6)	0.0618 (2)	0.8772 (3)	4.3 (1)
C1″	0.4862 (4)	0.0354 (2)	0.6960 (3)	3.2 (1)
C2″	0.4858 (5)	−0.0446 (2)	0.6664 (3)	3.9 (1)
C3″	0.6290 (6)	−0.0791 (2)	0.6126 (4)	5.0 (1)
C4″	0.7786 (6)	−0.0334 (3)	0.5892 (4)	5.2 (1)
C5″	0.7853 (5)	0.0456 (3)	0.6174 (4)	4.8 (1)
C6″	0.6406 (4)	0.0788 (2)	0.6691 (3)	3.6 (1)
Cl2″	0.2979 (1)	−0.1021 (1)	0.6950 (1)	5.90 (3)
Cl6″	0.6527 (1)	0.1790 (1)	0.7034 (1)	5.84 (3)

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

C1—C2	1.532 (5)	N2′—C3′	1.272 (4)
C1—C6	1.534 (6)	C3′—C4′	1.494 (5)
C1—C7	1.524 (6)	C3′—C1″	1.471 (5)
C2—C4′	1.516 (5)	C1″—C2″	1.396 (5)
C2—C3	1.541 (5)	C1″—C6″	1.397 (4)
C3—C4	1.494 (5)	C2″—C3″	1.385 (6)
C3—O3	1.212 (4)	C2″—Cl2″	1.730 (4)
C4—C5	1.549 (6)	C3″—C4″	1.379 (6)
C4—C7	1.524 (6)	C4″—C5″	1.375 (7)
C5—C6	1.540 (7)	C5″—C6″	1.373 (5)
O1′—N2′	1.409 (4)	C6″—Cl6″	1.741 (4)
O1′—C2	1.473 (4)		
C6—C1—C7	101.8 (3)	C2—O1′—N2′	109.5 (2)
C2—C1—C6	108.2 (3)	O1′—N2′—C3′	109.2 (2)
C2—C1—C7	100.9 (3)	N2′—C3′—C4′	114.4 (2)
C1—C2—C3	100.7 (3)	N2′—C3′—C1″	119.2 (2)
C1—C2—O1′	109.0 (2)	C4′—C3′—C1″	126.3 (3)
C1—C2—C4′	119.0 (3)	C2—C4′—C3′	101.5 (3)
C3—C2—O1′	104.9 (2)	C3′—C1″—C2″	122.5 (2)
C3—C2—C4′	118.6 (3)	C3′—C1″—C6″	121.4 (2)
O1′—C2—C4′	103.8 (2)	C2″—C1″—C6″	116.1 (2)
O3—C3—C4	128.6 (3)	C1″—C2″—C3″	122.2 (3)
C2—C3—O3	125.0 (3)	C1″—C2″—Cl2″	118.9 (2)
C2—C3—C4	106.4 (3)	C3″—C2″—Cl2″	118.9 (3)
C3—C4—C5	104.7 (3)	C2″—C3″—C4″	119.1 (3)
C3—C4—C7	101.3 (3)	C3″—C4″—C5″	120.7 (3)
C5—C4—C7	101.7 (3)	C4″—C5″—C6″	119.2 (3)
C4—C5—C6	102.7 (3)	C1″—C6″—C5″	122.7 (3)
C1—C6—C5	104.0 (3)	C1″—C6″—Cl6″	119.1 (2)
C1—C7—C4	95.3 (3)	C5″—C6″—Cl6″	118.1 (3)
C1—C2—C3—C4	5.5 (4)	C7—C1—C2—C3	−39.1 (4)
C2—C3—C4—C5	−75.5 (4)	C1—C2—C3—O3	−175.2 (3)
C3—C4—C5—C6	70.1 (4)	O1′—C2—C3—O3	71.7 (4)
C4—C5—C6—C1	0.8 (4)	C4′—C2—C1—C6	−64.0 (4)
C5—C6—C1—C2	−72.1 (4)	C1—C7—C4—C5	55.0 (4)
C6—C1—C2—C3	67.4 (4)	C7—C4—C5—C6	−35.1 (4)
C2—C3—C4—C7	29.9 (4)	C5—C6—C1—C7	33.7 (4)
C3—C4—C7—C1	−52.8 (4)	C6—C1—C7—C4	−54.4 (4)
C4—C7—C1—C2	57.1 (4)	C4′—C3′—C1″—C2″	73.3 (5)

Data collection: *AFC Software* (Rigaku Corporation, 1974). Cell refinement: *AFC Software*. Data reduction: *AFC Software*. Program(s) used to solve structure: *SHELX*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *Xtal*3.0 (Hall & Stewart, 1990). Calculations were performed on a VAX 8800 computer.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: C940462–HR1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Molecular Structure of Caroxazone, a Reversible Monoamine Oxidase Inhibitor

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Abstract

Caroxazone, 2-oxo-2*H*-1,3-benzoxazine-3(4*H*)-acetamide (I), $C_{10}H_{10}N_2O_3$, is a potent monoamine oxidase (MAO) inhibitor. The two molecules in the asymmetric unit have different conformations of the acetamide chain. The oxazine ring is not very distorted and the distances between the different atoms of the ring indicate electronic delocalization within this moiety. Crystal cohesion is assumed to be mainly due to intermolecular hydrogen bonding between the amidic N and carbonyl O atoms.

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