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5'-Chlorospiro[1,3-dioxolane-2,3'-indolin]-2'-one: a potential anti-convulsant

Amitabha De

Department of Physics, Khalisani College, Chandannagar, Hooghly 712 138, India
Correspondence e-mail: amitde03@yahoo.com

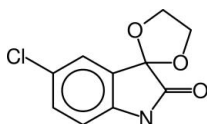
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
R factor = 0.050; wR factor = 0.190; data-to-parameter ratio = 14.4.

The title compound, $\text{C}_{10}\text{H}_8\text{ClNO}_3$, is a significant anti-convulsant agent. The indolinone system is essentially planar, the dihedral angle between the rings being 2.24 (8°). The dioxolane ring adopts an envelope conformation; the dihedral angle between the plane through its four coplanar atoms and the indolinone system is 89.8 (1°). The crystal structure is stabilized by a three-dimensional network of intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature, see: Coddington *et al.* (1984); De (1990, 1992); De & Kitagawa (1991a,b); De & Kusunoki (1991); Dickerson & Geis (1969); Itai *et al.* (1978); James & Williams (1972); Popp (1977, 1984); Rajopadhye & Popp (1988); Chakraborty & Talapatra (1985); Chakraborty *et al.* (1985).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{ClNO}_3$
 $M_r = 225.62$
Monoclinic, $I2/c$
 $a = 18.266$ (2) Å
 $b = 7.360$ (1) Å
 $c = 14.821$ (1) Å
 $\beta = 92.855$ (7°)

$V = 1990.0$ (4) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 298$ (2) K
 $0.50 \times 0.30 \times 0.30$ mm

Data collection

Rigaku AFC-4 diffractometer
Absorption correction: none

2479 measured reflections
2297 independent reflections

1591 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

3 standard reflections
every 100 reflections
intensity decay: 0.2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.189$
 $S = 0.83$
2297 reflections
160 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.52$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.82 (3)	2.11 (3)	2.885 (4)	157.4 (3)

Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z$.

Data collection: *AFC-4 Diffractometer Control Software* (Rigaku, 1997); cell refinement: *AFC-4 Diffractometer Control Software*; data reduction: *AFC-4 Diffractometer Control Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1983).

The author thanks Dr Frank D. Popp of the University of Missouri–Kansas City, for the gift of the sample used in this investigation, and the University Grants Commission, India, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2049).

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supplementary materials

Acta Cryst. (2008). E64, o562 [doi:10.1107/S160053680800336X]

5'-Chlorospiro[1,3-dioxolane-2,3'-indolin]-2'-one: a potential anticonvulsant

A. De

Comment

Anti-epileptic drugs have diverse chemical structures and complex physiological and pharmacological actions. The search for potential drugs and their mechanism of action has been difficult because of their complexity. A series of spiro(1,3-dioxolane-2,3'-indolin)-2'-one and structural analogues active against electrically and chemically induced seizures have been studied. These compounds contain both an oxoindole and a dioxolane moiety which have independently been seen in other anticonvulsants (Popp, 1977, 1984). The basic model compound, mentioned above, was used to study the effects of various electron-donating, electron withdrawing and hydrophobic groups on the activity of the molecule. A bulky hydrophobic substituent at the 1'-position (oxoindole) generally tends to decrease the activity. The present compound, a chloro analogue was found to be most potent in the MES test. Since no common target site has yet been established, X-ray analysis was undertaken to search structural information which may help in the understanding of the mechanism of action at the molecular level.

The conformation of the title compound along with the atom-numbering scheme is shown in Fig 1. The carbonyl C atom to tetrahedral C atom distance is typical of a single bond. The C(2) - C(3) bond distance is slightly shorter but closely similar to the values found in other indoline nuclei (Itai *et al.*, 1978; Chakraborty & Talapatra, 1985; Chakraborty *et al.*, 1985; De & Kitagawa, 1991*a,b*; De, 1992). The lone pair of electrons on N(1) is involved in conjugation with the carbonyl group. This is also indicated by the slight lengthening of the C=O double bond [1.220 (4) Å] and the concomitant shortening of the two N - C(*sp*²) single bonds [1.341 (4) Å and 1.412 (4) Å] (Coddington *et al.*, 1984). The least-squares planes through the five- and six-membered rings are inclined to one another at 2.24 (8)° and each of them are almost planar. The plane containing the atoms C(2), O(2), O(3), C(9) and C(10) is inclined to the overall plane through the indolinone group by 89.8 (1)°. The C—NH—CO—C grouping resembles a *cis* peptide bond. Six atoms of this group [C(8), N(1), H1, C(1), O(1), C(2)] are almost planar. The OC—N bond distance [1.341 (4) Å] is not as short as the normal peptide bond (1.325 Å) (Dickerson & Geis, 1969). The packing of the molecule is shown in Fig. 2. The amide nitrogen, N(1), forms a hydrogen bond with the carbonyl oxygen O(1) [N(1) - H1] = 0.82 (3), N(1)⋯O(1) = 2.885 (4), H1⋯O(1) = 2.11 (3) Å, N(1) - H1⋯O(1) = 157.4 (3)°]. The molecules are thus held together by a three-dimensional network of hydrogen bonds.

Experimental

The synthesis of the compound has been described earlier (Rajopadhye & Popp, 1988). Diffraction quality crystals were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

All the hydrogen atoms in the structure were located in a difference map except the two H-atoms on C10. They were placed at geometrically idealized positions. Geometric calculations were performed using *SHELXL-97*, *PARST* (Nardelli, 1983) programs.

Figures

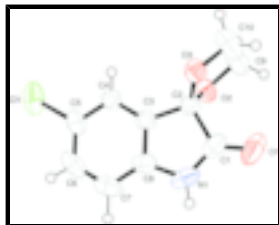


Fig. 1. ORTEP (Johnson, 1965) diagram of the Molecular structure with atom labels showing displacement ellipsoids at 50% probability.

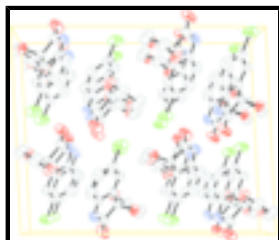


Fig. 2. Packing of the molecule viewed down the *b* axis.

5'-Chlorospiro[1,3-dioxolane-2,3'-indolin]-2'-one

Crystal data

$C_{10}H_8ClNO_3$

$M_r = 225.62$

Monoclinic, $I2/c$

Hall symbol: $-I\ 2yc$

$a = 18.266\ (2)\ \text{\AA}$

$b = 7.360\ (1)\ \text{\AA}$

$c = 14.821\ (1)\ \text{\AA}$

$\beta = 92.855\ (7)^\circ$

$V = 1990.0\ (4)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 928$

$D_x = 1.506\ \text{Mg m}^{-3}$

$D_m = 1.498\ \text{Mg m}^{-3}$

D_m measured by flotation

Melting point: 460 K

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 2.2\text{--}27.5^\circ$

$\mu = 0.37\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Needle, colourless

$0.50 \times 0.30 \times 0.30\ \text{mm}$

Data collection

Rigaku AFC-4 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ (2)\ \text{K}$

ω scans

Absorption correction: none

2479 measured reflections

2297 independent reflections

1591 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 2.2^\circ$

$h = -23 \rightarrow 23$

$k = 0 \rightarrow 9$

$l = 0 \rightarrow 19$

3 standard reflections

every 100 reflections

intensity decay: 0.2%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.189$	$w = 1/[\sigma^2(F_o^2) + (0.1365P)^2 + 2.6179P]$
$S = 0.83$	where $P = (F_o^2 + 2F_c^2)/3$
2297 reflections	$(\Delta/\sigma)_{\max} = 0.006$
160 parameters	$\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.52 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections except for 0 with very negative F^2 or flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > 2\sigma(F^2)$ is used only for calculating $_R_factor_obs$ etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{iso}^*/U_{eq}
Cl1	0.40898 (6)	-0.00775 (11)	0.43060 (5)	0.0825 (4)
O1	0.3184 (2)	0.5049 (4)	0.0001 (2)	0.0931 (9)
O2	0.37608 (10)	0.5934 (2)	0.18885 (12)	0.0534 (5)
O3	0.45532 (9)	0.4244 (2)	0.11210 (12)	0.0494 (4)
N1	0.28723 (12)	0.2542 (4)	0.0815 (2)	0.0612 (6)
C1	0.3263 (2)	0.4038 (4)	0.0651 (2)	0.0596 (7)
C2	0.38452 (12)	0.4261 (3)	0.1450 (2)	0.0442 (5)
C3	0.36853 (11)	0.2671 (3)	0.20354 (15)	0.0418 (5)
C4	0.40017 (12)	0.2147 (3)	0.2858 (2)	0.0446 (5)
C5	0.3712 (2)	0.0628 (3)	0.3264 (2)	0.0516 (6)
C6	0.3125 (2)	-0.0317 (4)	0.2873 (2)	0.0634 (8)
C7	0.2809 (2)	0.0217 (4)	0.2041 (2)	0.0606 (7)
C8	0.31030 (12)	0.1704 (3)	0.1639 (2)	0.0484 (5)
C9	0.4219 (2)	0.7216 (4)	0.1450 (2)	0.0678 (8)
C10	0.4787 (2)	0.6085 (4)	0.1035 (3)	0.0759 (9)
H4	0.4389 (16)	0.275 (4)	0.3142 (18)	0.051 (7)*

supplementary materials

H6	0.2955 (19)	-0.126 (5)	0.316 (2)	0.069 (9)*
H7	0.234 (2)	-0.037 (5)	0.181 (2)	0.077 (10)*
H1	0.2565 (18)	0.206 (5)	0.047 (2)	0.062 (9)*
H91	0.393 (2)	0.792 (6)	0.102 (3)	0.110 (14)*
H92	0.441 (2)	0.808 (7)	0.189 (3)	0.086 (11)*
H101	0.5260	0.6263	0.1351	0.08*
H102	0.4822	0.6401	0.0403	0.08*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.1293 (9)	0.0619 (5)	0.0567 (5)	0.0119 (4)	0.0092 (5)	0.0165 (3)
O1	0.099 (2)	0.100 (2)	0.077 (2)	0.0173 (14)	-0.0313 (13)	0.0273 (14)
O2	0.0595 (10)	0.0389 (9)	0.0630 (10)	0.0018 (7)	0.0167 (8)	-0.0016 (7)
O3	0.0459 (8)	0.0434 (9)	0.0594 (10)	0.0071 (7)	0.0089 (7)	0.0065 (7)
N1	0.0475 (11)	0.0723 (15)	0.0617 (13)	0.0053 (11)	-0.0181 (10)	-0.0182 (12)
C1	0.0552 (13)	0.065 (2)	0.0573 (14)	0.0149 (12)	-0.0140 (11)	0.0000 (12)
C2	0.0427 (10)	0.0426 (11)	0.0467 (11)	0.0043 (8)	-0.0024 (8)	0.0014 (9)
C3	0.0367 (9)	0.0384 (10)	0.0500 (11)	0.0013 (8)	-0.0004 (8)	-0.0026 (9)
C4	0.0426 (11)	0.0400 (11)	0.0509 (12)	-0.0019 (9)	-0.0016 (9)	-0.0015 (9)
C5	0.0677 (14)	0.0371 (11)	0.0514 (12)	0.0043 (10)	0.0157 (11)	0.0016 (9)
C6	0.077 (2)	0.0349 (11)	0.081 (2)	-0.0119 (12)	0.031 (2)	-0.0082 (12)
C7	0.0508 (13)	0.0505 (14)	0.081 (2)	-0.0119 (11)	0.0094 (12)	-0.0206 (13)
C8	0.0393 (10)	0.0447 (11)	0.0608 (13)	0.0020 (9)	0.0001 (9)	-0.0156 (10)
C9	0.083 (2)	0.0459 (14)	0.076 (2)	-0.0025 (13)	0.022 (2)	0.0066 (14)
C10	0.086 (2)	0.051 (2)	0.095 (2)	-0.0020 (15)	0.041 (2)	0.005 (2)

Geometric parameters (\AA , $^\circ$)

C11—C5	1.740 (3)	C4—C5	1.386 (3)
O1—C1	1.220 (4)	C4—H4	0.92 (3)
O2—C2	1.404 (3)	C5—C6	1.381 (4)
O2—C9	1.438 (3)	C6—C7	1.392 (5)
O3—C2	1.405 (3)	C6—H6	0.88 (4)
O3—C10	1.429 (4)	C7—C8	1.369 (4)
N1—C1	1.341 (4)	C7—H7	1.01 (4)
N1—C8	1.412 (4)	C9—C10	1.487 (4)
N1—H1	0.82 (3)	C9—H91	0.96 (4)
C1—C2	1.560 (3)	C9—H92	0.96 (4)
C2—C3	1.494 (3)	C10—H101	0.97
C3—C4	1.378 (3)	C10—H102	0.97
C3—C8	1.386 (3)		
C2—O2—C9	106.6 (2)	C4—C5—C11	118.8 (2)
C2—O3—C10	107.9 (2)	C5—C6—C7	120.6 (2)
C1—N1—C8	112.1 (2)	C5—C6—H6	118.9 (23)
C1—N1—H1	126.8 (23)	C7—C6—H6	120.5 (23)
C8—N1—H1	120.7 (23)	C8—C7—C6	117.2 (2)
O1—C1—N1	127.0 (3)	C8—C7—H7	123.0 (21)

O1—C1—C2	125.8 (3)	C6—C7—H7	119.1 (21)
N1—C1—C2	107.3 (2)	C7—C8—C3	122.4 (2)
O3—C2—O2	107.0 (2)	C7—C8—N1	128.2 (2)
O3—C2—C3	113.9 (2)	C3—C8—N1	109.4 (2)
O2—C2—C3	112.9 (2)	O2—C9—C10	104.7 (2)
O3—C2—C1	109.8 (2)	O2—C9—H91	109.6 (26)
O2—C2—C1	110.9 (2)	C10—C9—H91	113.8 (26)
C3—C2—C1	102.3 (2)	O2—C9—H92	109.0 (23)
C4—C3—C8	120.7 (2)	C10—C9—H92	114.7 (24)
C4—C3—C2	130.3 (2)	H91—C9—H92	105.0 (36)
C8—C3—C2	108.9 (2)	O3—C10—C9	106.0 (2)
C3—C4—C5	117.2 (2)	O3—C10—H101	110.3
C3—C4—H4	123.2 (17)	C9—C10—H101	111.0
C5—C4—H4	119.6 (17)	O3—C10—H102	110.3
C6—C5—C4	122.0 (3)	C9—C10—H102	110.3
C6—C5—C11	119.2 (2)	H101—C10—H102	110.0
C9—O2—C2—O3	28.9 (2)	O2—C2—C3—C8	118.8 (2)
C9—O2—C2—C1	-90.9 (2)	O3—C2—C3—C4	63.6 (3)
C9—O2—C2—C3	155.0 (2)	O3—C2—C3—C8	-118.9 (2)
C2—O2—C9—C10	-23.5 (3)	C1—C2—C3—C4	-177.9 (2)
C10—O3—C2—O2	-22.6 (3)	C1—C2—C3—C8	-0.4 (2)
C10—O3—C2—C1	97.9 (2)	C2—C3—C4—C5	177.2 (2)
C10—O3—C2—C3	-148.1 (2)	C2—C3—C8—N1	0.9 (3)
C2—O3—C10—C9	7.5 (3)	C2—C3—C8—C7	-176.9 (2)
C8—N1—C1—O1	-179.1 (3)	C4—C3—C8—N1	178.7 (2)
C8—N1—C1—C2	0.8 (3)	C4—C3—C8—C7	0.9 (4)
C1—N1—C8—C3	-1.1 (3)	C3—C4—C5—C11	-179.6 (2)
C1—N1—C8—C7	176.6 (3)	C3—C4—C5—C6	-0.9 (4)
O1—C1—C2—O2	59.0 (4)	C11—C5—C6—C7	179.8 (2)
O1—C1—C2—O3	-59.1 (4)	C4—C5—C6—C7	1.0 (4)
O1—C1—C2—C3	179.7 (3)	C5—C6—C7—C8	-0.2 (4)
N1—C1—C2—O2	-120.9 (2)	C6—C7—C8—N1	-178.1 (3)
N1—C1—C2—O3	121.0 (2)	C6—C7—C8—C3	-0.7 (4)
N1—C1—C2—C3	-0.2 (3)	O2—C9—C10—O3	9.7 (3)
O2—C2—C3—C4	-58.7 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O1^i$	0.82 (3)	2.11 (3)	2.885 (4)	157.4 (3)

Symmetry codes: (i) $-x+1/2, y-1/2, -z$.

Fig. 1

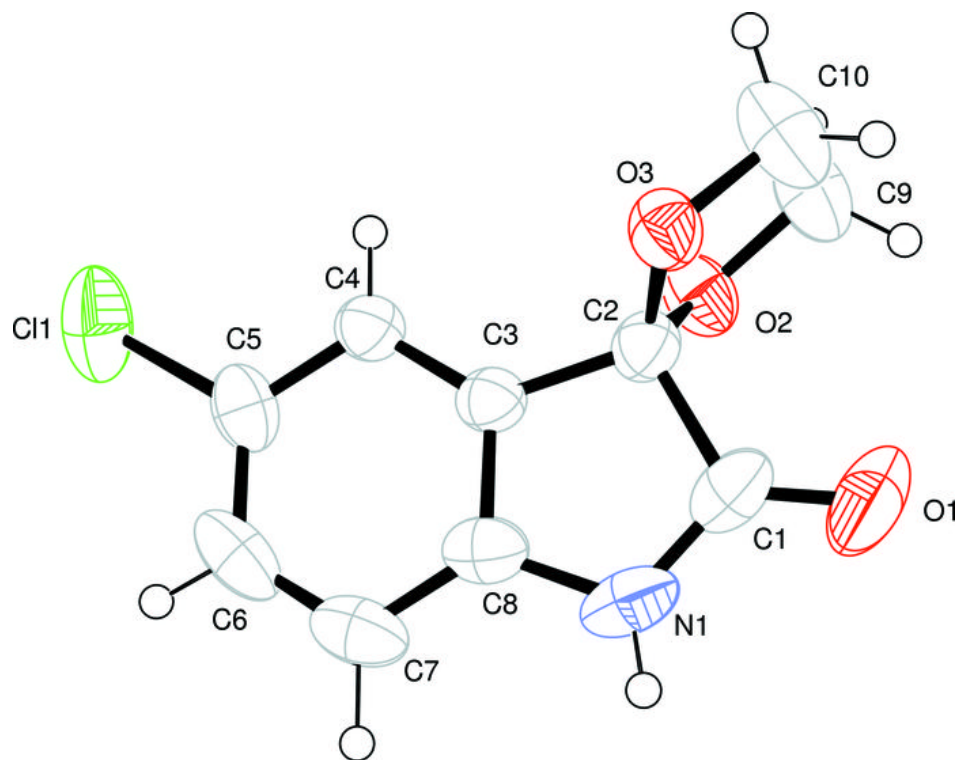


Fig. 2

