C11, in which the hybridization state differs, exhibit an r.m.s. deviation of 0.005 Å, with the largest individual difference, 0.013 Å, for C8—C9. Both our compounds containing OH groups exhibit hydrogen bonding in the solid. The hydroxyl group of O1 of acetate (1) forms an intermolecular hydrogen bond with lactone carbonyl oxygen O3 at $x, y+1, z$ having O—O distance 2.746 (3) Å. The angle about the unrefined H-atom position is 173°. The hydroxyl compound (3) exhibits both intramolecular and intermolecular hydrogen bonding. The intramolecular bond involves O4 as donor and hydroxyl group O1 as acceptor, O—O distance 2.747 (2) Å, angle at H 143 (3)°. The intermolecular hydrogen bond involves O1 as donor and hydroxyl group O4 at $\frac{1}{2}+x, \frac{1}{2}-y, 2-z$, with O—O distance 2.841 (2) Å and angle at H 157 (3)°.

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Structure of a Potential Anticonvulsant: 5′-Bromospiro[1,3-dioxolane-2,3′-indolin]-2′-one

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Abstract. $C_{10}H_8BrNO_3$, $M_r = 270.08$, monoclinic, $C2/c$, $a = 22.924$ (1), $b = 7.5051$ (4), $c = 15.274$ (10) Å, $\beta = 129.071$ (3)°, $V = 2040.2$ (2) Å^3, $Z = 8$, $D_m = 1.760$ (by flotation), $D_x = 1.758$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 5.44$ mm^{-1}, $F(000) = 1072$, $T = 298$ K, $R = 0.071$ for 1424 reflections with $I > 2\sigma(I)$. The bond linking the C(=O) atom to the tetrahedral C atom is a single bond. The amide group exhibits delocalization of the π-electrons over the three atoms (N, C and O). The five- and six-membered rings are planar and inclined at a dihedral angle of 2.6 (5)°. The dioxolane ring is almost perpendicular [89.4 (7)°] to the indolinone group. The crystal structure is stabilized by a three-dimensional network of N(1)—H(N1)···O(1) hydrogen bonds.

Introduction. Anti-epileptic drugs have diverse chemical structures and complex physiological and pharmacological actions. The search for potential anti-epileptic drugs and their mechanism of action has been difficult because of their complexity. There is no single mechanism to explain the action of all anti-epileptic drugs. Some of these drugs have specific modes of action but others have not yet been linked with a specific binding site. A series of spiro[1,3-dioxane-2,3′-indolin]-2′-ones 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 effect of various electron-donating, electron-withdrawing and hydrophobic groups on the activity of the molecule. In general a bulky hydrophobic

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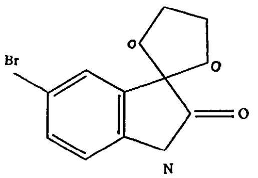
substituent at the 1′-position (oxoindole N) tends to decrease the activity. Introduction of a halogen in the oxindole portion of the model compound led to more active compounds. The position of the chlorine (4′, 5′ or 6′) does not seem to affect the activity of these compounds. The iodo derivatives are equally active in the MES test.



The present compound is a bromo analogue and it is seen to be less active. Since no common target site has yet been established, the properties of anticonvulsants must be sought from the structural information currently available and hence an X-ray analysis was undertaken as part of a project to search for the common conformational features which may help in the understanding of the mechanism of action of such compounds at the molecular level.

Experimental. The sample was a kind gift from Dr Frank Popp (Rajopadhye & Popp, 1988). Single crystals of the compound were prepared by slow evaporation of an ethanol solution at room temperature. Needle-shaped crystals appeared after one week. Space group and preliminary unit-cell dimensions determined from Weissenberg photographs. A crystal with dimensions 0.35 × 0.30 × 0.45 mm was used for the X-ray diffractometry. Rigaku AFC-5 diffractometer operating at 40 kV 200 mA, graphite-monochromatized Cu $K\alpha$ radiation. Accurate cell parameters and orientation angles determined by least-squares refinement of the θ values of 25 reflections with $18 < \theta < 32°$. Intensity data collected by $2\theta/\omega$ scan, scan rate $6° \text{ min}^{-1}$, scan range $(1.2 + 0.15\tan\theta)°$, $2\theta_{\max} = 125°$. 1772 reflections measured with $h = -26$ to 26, $k = 0$ to 8, $l = 0$ to 17. From the conditions limiting possible reflection, hkl: $h + k = 2n$, $h0l$: $l = 2n$, $0k0$: $k = 2n$ and the centric distribution of data, the space group $C2/c$ was assumed. Three reference reflections monitored after every 2 h of X-ray exposure time showed no crystal movement or decay. Intensities corrected for Lorentz and polarization effects, but no absorption correction was applied. Out of 1588 unique reflections 1424 reflections with $F_o > 4\sigma(F_o)$ were observed. $R_{\text{int}} = 0.020$.

Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refined on F by the full-matrix least-squares technique (*SHELX*76; Sheldrick, 1976). Function minimized: $\sum w(F_o - F_c)^2$ with $w = 1/\sigma^2(F_o)$. Non-H-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF synthesis and refined isotropically. Final $R = 0.071$, $wR = 0.071$ for 1424 observed reflections, 144 parameters refined. Max. Δ/σ in the final cycle of refinement is 0.02. Max. and min. peak heights in difference Fourier map were 0.63 and -1.43 e Å^{-3}. Atomic scattering factors were those incorporated in *SHELX*76. The geometrical parameters of the molecule were computed with the program *PLATON* (Spek, 1990).

Discussion. A perspective view of the molecule together with the atom-numbering scheme is shown in Fig. 1. The final atomic parameters are listed in Table 1,* while Table 2 lists the intramolecular bond distances and angles.

The carbonyl C atom to tetrahedral C atom distance, C(1)—C(2) = 1.539 (13) Å, is typical of a single bond. The C(2)—C(3) bond distance [1.509 (14) Å] is slightly shorter but closely similar to the values found in other indoline nuclei (Itai, Iitaka & Kubo, 1978; Chakraborty & Talapatra, 1985; De & Kitagawa, 1991). It is observed that the lone pair of electrons on N(1) is involved in conjugation with the carbonyl group. This is indicated by the slight lengthening of the C=O double bond [1.235 (18) Å] and the concomitant shortening of the two N—C(sp^2) single bonds [1.322 (16) and 1.391 (17) Å] (Codding, Lee & Richardson, 1984). The state of hybridization of the N atom is sp^2 as shown by the sum (360°) of the angles around it and from the perfect planarity of the plane C(1), O(1), N(1) and C(8). Similar geometries have also been observed in other structures (James & Williams, 1972; Codding, Lee & Richardson, 1984; De, 1990; De & Kusunoki,

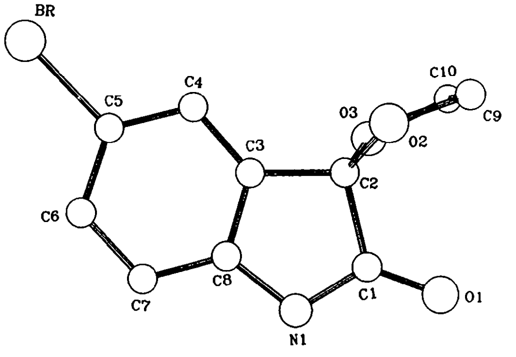


Fig. 1. Perspective view of the molecule (perpendicular to the plane of the amide group) showing the atomic labelling scheme.

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54651 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0485]

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for non*-H *atoms with their e.s.d.'s in parentheses*

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

	x	y	z	U_{eq} (Å^2)
Br	0.09282 (8)	−0.00706 (19)	0.02233 (11)	0.0457 (5)
O(1)	0.1815 (5)	0.5089 (14)	−0.3142 (8)	0.059 (3)
O(2)	0.1239 (4)	0.5911 (9)	−0.1890 (6)	0.030 (2)
O(3)	0.0421 (4)	0.4215 (9)	−0.3460 (5)	0.027 (2)
N(1)	0.2141 (5)	0.2563 (13)	−0.2065 (7)	0.030 (3)
C(1)	0.1753 (6)	0.4032 (17)	−0.2585 (9)	0.033 (4)
C(2)	0.1166 (5)	0.4249 (14)	−0.2407 (8)	0.023 (3)
C(3)	0.1329 (5)	0.2668 (13)	−0.1673 (8)	0.021 (3)
C(4)	0.1013 (5)	0.2178 (13)	−0.1183 (8)	0.020 (3)
C(5)	0.1316 (5)	0.0697 (14)	−0.0499 (8)	0.024 (3)
C(6)	0.1913 (6)	−0.0294 (15)	−0.0304 (9)	0.034 (4)
C(7)	0.2218 (6)	0.0271 (16)	−0.0828 (9)	0.034 (4)
C(8)	0.1918 (5)	0.1761 (13)	−0.1497 (8)	0.021 (3)
C(9)	0.0756 (8)	0.7171 (16)	−0.2804 (10)	0.046 (4)
C(10)	0.0161 (7)	0.6080 (17)	−0.3818 (10)	0.049 (4)

Table 2. *Bond distances* (Å) *and bond angles* (°) *of non*-H *atoms with their e.s.d.'s in parentheses*

Br—C(5)	1.891 (13)	O(1)—C(1)	1.235 (18)
O(2)—C(2)	1.429 (13)	O(2)—C(9)	1.456 (14)
O(3)—C(2)	1.429 (13)	O(3)—C(10)	1.484 (15)
N(1)—C(1)	1.322 (16)	N(1)—C(8)	1.391 (17)
C(1)—C(2)	1.539 (13)	C(2)—C(3)	1.509 (14)
C(3)—C(4)	1.380 (18)	C(3)—C(8)	1.379 (18)
C(4)—C(5)	1.376 (14)	C(5)—C(6)	1.415 (20)
C(6)—C(7)	1.418 (15)	C(7)—C(8)	1.372 (15)
C(9)—C(10)	1.506 (18)		

C(2)—O(2)—C(9)	106.5 (8)	C(2)—O(3)—C(10)	108.4 (8)
C(1)—N(1)—C(8)	111.3 (12)	O(1)—C(1)—N(1)	128.6 (15)
O(1)—C(1)—C(2)	123.1 (12)	N(1)—C(1)—C(2)	108.3 (11)
O(2)—C(2)—O(3)	106.2 (9)	O(2)—C(2)—C(1)	112.3 (10)
O(2)—C(2)—C(3)	112.8 (8)	O(3)—C(2)—C(1)	110.9 (9)
O(3)—C(2)—C(3)	112.5 (9)	C(1)—C(2)—C(3)	102.3 (10)
C(2)—C(3)—C(4)	130.1 (11)	C(2)—C(3)—C(8)	106.7 (11)
C(4)—C(3)—C(8)	123.2 (9)	C(3)—C(4)—C(5)	116.1 (12)
Br—C(5)—C(4)	119.4 (10)	Br—C(5)—C(6)	117.7 (8)
C(4)—C(5)—C(6)	122.9 (12)	C(5)—C(6)—C(7)	118.6 (10)
C(6)—C(7)—C(8)	118.0 (13)	N(1)—C(8)—C(3)	111.4 (9)
N(1)—C(8)—C(7)	127.5 (13)	C(3)—C(8)—C(7)	121.1 (12)
O(2)—C(9)—C(10)	106.3 (9)	O(3)—C(10)—C(9)	103.9 (10)

1991; De & Kitagawa, 1991). In the indolinone group the six-membered and five-membered rings are planar within experimental error. The least-squares planes through the five- and six-membered rings are inclined to one another at 2.6 (5)°. 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 89.4 (7)°. The C—NH—CO—C grouping resembles a *cis* peptide bond. Six atoms of this group [C(8), N(1), H(N1), C(1), O(1) and C(2)] are almost planar. The OC—N bond distance [1.322 (16) Å] is short as a normal peptide bond [1.325 Å] (Dickerson & Geis, 1969) but there are some distortions in the bond angles. This may be due to the substituents at C(2) and the cyclic nature of the system which hinders the favourable overlapping of atomic orbitals.

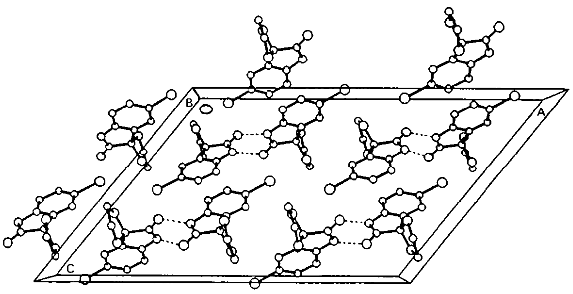

Fig. 2. Packing of the molecule (*PLUTO*; Motherwell & Clegg, 1978).

The packing of the molecule and the hydrogen-bonding scheme is shown in Fig. 2. The amide nitrogen, N(1), forms a hydrogen bond with the carbonyl oxygen O(1), [N(1)—H(N1) = 0.880 (12), N(1)···O(1)(0.5 − x, 0.5 + y, 0.5 − z) = 2.885 (17), H(N1)···O(1) = 2.111 (12) Å, N(1)—H(N1)···O(1) = 146.3 (7)°]. The molecules are thus held together by a three-dimensional network of hydrogen bonds.

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