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5,5'-Dichloro-3,3'-dinitro-2,2'-biphenyldiol*

BY D. G. HAY AND M. F. MACKAY

Department of Physical Chemistry, La Trobe University, Bundoora, Victoria 3083, Australia

Abstract. $C_{12}H_6Cl_2N_2O_6$, monoclinic, $C2/c$, $a = 17.114$ (2), $b = 7.343$ (1), $c = 11.676$ (1) Å, $\beta = 118.11$ (1)°, $U = 1294.2$ (5) Å^3, $D_m = 1.77$ (1), $D_c = 1.77$ Mg m^{-3}, $Z = 4$. $\mu(Cu\ K\alpha) = 4.67$ mm^{-1}; $R = 0.047$ for 969 observed reflections. The molecule has exact 2 symmetry and the dihedral angle between the perpendiculars to the phenyl rings is 43.6 (5)°.

Introduction. Niclofolan, the title compound, also known as Bilevon, Dertil, Distolan and Menichlopholan (Bard, 1972), is an anthelmintic (worm-killing) drug used against *Fasciola hepatica* (liver-fluke) infections in sheep and cattle (Australian Bureau of Animal Health, 1977). It is recognized that biphenol and bisphenol flukicides act by inhibiting ATP production in the flukes, and the active site is thought to be the succinate dehydrogenase complex (Metzger & Düwel, 1973). The molecular conformation of the biphenol reported here provides a comparison with those of several bisphenol flukicide structures reported earlier (Hay & Mackay, 1979, 1980; Hay, DeMunk & Mackay, 1980). The latter, which contain a bridging C atom, have the same ring substitutents as niclofolan in which the phenyl rings are directly linked.

Weissenberg photographs showed that the blade-like crystals grown from ethanol were monoclinic, and systematic absences indicated either the space group $C2$ or $C2/c$ (subsequently confirmed as $C2/c$). Intensities were measured with Cu $K\alpha$ radiation (graphite-crystal monochromator, $\lambda = 1.5418$ Å) from a crystal $0.40 \times 0.14 \times 0.04$ mm mounted on a Rigaku–AFC

four-circle diffractometer. Of the 1287 non-equivalent terms ($2\theta_{max} = 130°$) recorded by an ω–2θ scan of 2° min^{-1} and with 10s stationary background counts, the 969 for which $|F_o| > 2\sigma|F_o|$ were used for the structure refinement. The intensities were corrected for Lorentz and polarization effects and for absorption; no corrections for extinction were made. The scattering factors for Cl, O, N and C were from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were made for the non-hydrogen atoms with the values of Cromer & Liberman (1970).

The structure was solved by direct methods with *SHELX* 76 (Sheldrick, 1976). Full-matrix least-squares refinement with individual isotropic temperature factors for the non-hydrogen atoms yielded $R = 0.15$. After refinement of these atoms with anisotropic temperature factors, the H atoms were located on a difference map. Final refinement, in which the positional parameters and isotropic temperature factors of the H atoms were allowed to vary, converged at $R = 0.047$ and $R_w = (\sum w||F_o| - |F_c||^2/\sum w|F_o|^2)^{1/2} = 0.063$. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with the terms weighted according to $(\sigma^2|F_o| + 0.003|F_o|^2)^{-1}$. The final difference map contained no peaks >0.3 e Å^{-3}; the largest peaks were near the Cl atom. Final atomic coordinates are given in Table 1.† Figs. 1 and 2 have been prepared with *ORTEP* (Johnson, 1965).

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* Bisphenol Flukicides. III. Part I: Hay & Mackay (1979, 1980). Part II: Hay, DeMunk & Mackay (1980).

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35664 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic parameters* (×10⁴, H *atoms* ×10³), *with e.s.d.'s in parentheses, and equivalent isotropic temperature factors*

$$B_{eq} = 8\pi^2(U_1 U_2 U_3)^{1/3}.$$

	x	y	z	B_{eq} (Å²)
Cl	4199 (1)	3328 (1)	630 (1)	3·2
N	7197 (2)	892 (5)	1331 (3)	3·1
O(1)	7664 (2)	164 (5)	920 (3)	4·2
O(2)	7456 (2)	1173 (5)	2481 (3)	4·2
O(3)	6517 (2)	718 (4)	−1446 (2)	3·1
C(1)	5158 (2)	1998 (5)	−1791 (3)	2·4
C(2)	6021 (2)	1368 (4)	−929 (3)	2·4
C(3)	6302 (2)	1457 (5)	405 (3)	2·5
C(4)	5762 (2)	2076 (5)	915 (3)	2·6
C(5)	4918 (2)	2622 (5)	50 (3)	2·5
C(6)	4628 (2)	2608 (5)	−1275 (3)	2·4
H(4)	599 (3)	204 (5)	183 (4)	3·1
H(6)	406 (3)	314 (5)	−182 (4)	3·1
H(O3)	699 (4)	29 (8)	−80 (5)	7·1

Fig. 1. Perspective view of the molecule with thermal ellipsoids scaled to 50% probability, showing bond distances (Å) and angles (°). E.s.d.'s for the bond lengths are given in parentheses; for the angles involving the H atoms they are 3° and for all other angles they range from 0·3 to 0·4°. (H atoms are numbered according to the atoms to which they are bonded.)

Fig. 2. Crystal packing.

Discussion. The niclofolan molecule (Fig. 1) has exact 2 (C_2) symmetry, the angle between the perpendiculars to the phenyl-ring planes being 43·6 (5)°. Although this value is similar to those observed for the inter-ring angles (36–42°) in a number of simple *meta* and *para* biphenyls (Chawdhury, Hargreaves & Sullivan, 1968; Lin & Williams, 1975; Brock, Kuo & Levy, 1978), it is considerably smaller than the values observed in a series of *ortho*-substituted biphenyls. These compounds, 2,2'-difluoro-, 2,2'-dichloro-, 2,2'-dibromo- and 2,2'-diiodobiphenyl, have been studied by electron diffraction, and values for the inter-ring angles were found to range from 60 to 79° (Bastiansen, 1950; Bastiansen & Smedvik, 1954). A recent X-ray analysis of 2,2'-dichlorobiphenyl (Rømming, Seip & Oymo, 1974) gave 66·8° for the inter-ring angle and 69·2° for the torsion angle, φ, C(2)–C(1)–C(1')–C(2'). Thus, the conformation of the latter (and the other biphenyls in the series) is closer to the *syn* form ($\varphi = 0°$) than to the *anti* form ($\varphi = 180°$). This contrasts with the conformation of niclofolan for which $\varphi = 135·6$ (5)° and so is closer to the *anti* form.

The bond lengths and angles (uncorrected for thermal effects) do not deviate significantly from their expected values, Fig. 1. The mean deviation of the C atoms from the phenyl-ring least-squares plane defined by the equation $-0·3689X - 0·9286Y - 0·0407Z + 4·8992 = 0$* is 0·010 Å; the maximum deviation is 0·015 Å. The nitro group is rotated through 8·3 (5)° from coplanarity with the ring. Although the C(3)–N and C(2)–O(3) bonds are bent from the ring plane by 1·5 (10) and 2·6 (7)° respectively, so that N and O(3) lie on opposite sides of the plane, the rotation of the nitro group brings O(1) closer to O(3) and H(O3); the latter lies 0·16 (6) Å from the phenyl-ring plane. The geometry of the six-membered ring containing the intramolecular hydrogen bond agrees well with those reported for other *ortho* nitrophenols including 2,2'-(2,2,2-trichloroethylidene)bis(4-chloro-6-nitrophenol) (Hay, DeMunk & Mackay, 1980), Fig. 1. The O(1)···O(3) and O(1)···H(O3) distances are 2·557 (4) and 1·79 (6) Å, respectively, and the O(3)–H(O3)···O(1) angle is 148 (6)°. The ring atoms, coplanar within ±0·04 Å, make an angle of 4 (2)° with the phenyl-ring plane.

The crystal packing is illustrated in Fig. 2. The molecules are oriented in the crystals with their long molecular axes approximately parallel to [501] and form layers parallel to the (100) planes. Each layer contains both mirror-image conformers. A short intermolecular contact of 2·824 (5) Å between O(3) (the hydroxyl O atom) and O(2) of the nitro group of a molecule related by the *c* glide (and a unit translation in *c*) is indicative of intermolecular hydrogen bonding. However, as the
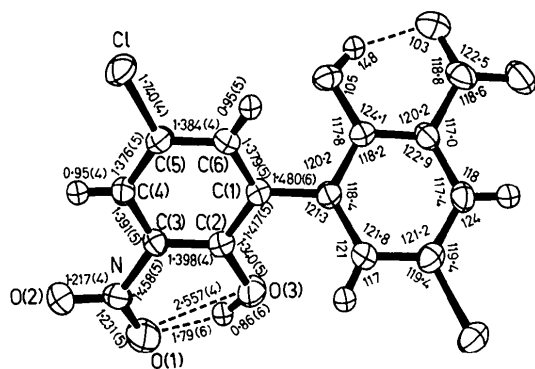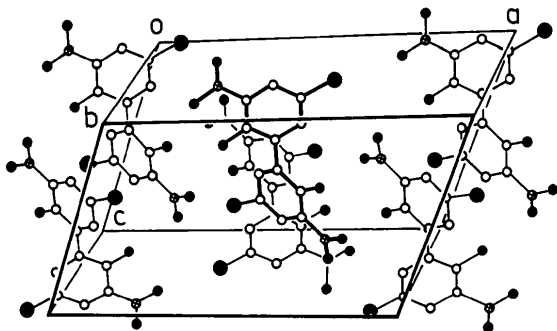
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* X, Y, Z are expressed in Å and referred to orthogonal axes parallel to a, b and c^*.

Table 2. *Intermolecular approach distances* < 3.6 Å *with e.s.d.'s in parentheses*

$O(1)\cdots Cl^{I}$	3·110 (4)	$O(1)\cdots O(3)^{V}$	3·267 (5)
$O(2)\cdots Cl^{II}$	3·496 (4)	$O(2)\cdots O(3)^{VIII}$	2·824 (5)
$O(3)\cdots Cl^{III}$	3·512 (3)	$O(2)\cdots O(3)^{V}$	3·430 (5)
$C(2)\cdots Cl^{IV}$	3·504 (3)	$N\cdots O(3)^{VIII}$	3·512 (5)
$C(5)\cdots Cl^{IV}$	3·590 (4)	$N\cdots O(3)^{V}$	3·283 (5)
$C(6)\cdots Cl^{IV}$	3·475 (4)	$C(4)\cdots O(3)^{VIII}$	3·409 (4)
$C(2)\cdots O(1)^{V}$	3·395 (5)	$C(3)\cdots C(5)^{III}$	3·545 (5)
$O(1)\cdots O(2)^{VI}$	3·532 (5)	$C(5)\cdots C(5)^{IV}$	3·510 (5)
$C(6)\cdots O(2)^{VII}$	3·412 (5)	$C(4)\cdots C(6)^{III}$	3·566 (5)

Symmetry code

(I)	$\frac{1}{2}+x, -\frac{1}{2}+y, z$	(V)	$1\frac{1}{2}-x, \frac{1}{2}-y, -z$
(II)	$\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$	(VI)	$1\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$
(III)	$1-x, -y, -z$	(VII)	$-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$
(IV)	$1-x, 1-y, -z$	(VIII)	$x, -y, \frac{1}{2}+z$

site of H(O3), clearly defined in the difference map and subsequently refined, is 2·71 Å from O(2), the hydroxyl group is involved only in the intramolecular hydrogen bond. There is one intermolecular approach less than the van der Waals contact distance, Table 2. The nitro group O(1) is 3·110 (4) Å from the Cl of a molecule related by the *C* centring (and a unit translation in *b*). This distance is 0·09 Å less than the sum of the van der Waals radii (Pauling, 1960).

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Structure of (±)-6,*t*-8b,9,10,11,12,13,*t*-13a-Octahydro-5*H*-7-thia-12a-azabenzo[*f*]naphth[1,2,3-*cd*]azulene*

BY A. MONGE AND E. GUTIÉRREZ-PUEBLA

Instituto de Química Inorgánica Elhuyar, CSIC Serrano 113, Madrid-6, Spain and Departamento de Química Inorgánica, Facultad de Químicas, Universidad Complutense, Madrid-3, Spain

Abstract. $C_{19}H_{21}NS$, $M_r = 295·4$, monoclinic, $P2_1/c$, $a = 8·2807$ (8), $b = 21·491$ (1), $c = 8·6243$ (8) Å, $\beta = 97·57$ (4)°, $U = 1521·4$ (2) Å^3, $Z = 4$, $D_c = 1·29$ Mg m^{-3}, $\mu = 0·196$ mm^{-1}. Final $R = 0·036$ for 2013 observed reflexions. The H atoms bonded to the two asymmetric C atoms are *trans* with respect to the corresponding ring.

Introduction. The title compound was synthetized by Arribas & Vega (1980). A pale-yellow prismatic crystal was used to collect the data. 3357 reflexions were measured in the $\omega/2\theta$ scan mode on a Nonius CAD-4

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* This numbering differs from the crystallographic numbering used throughout the paper.