the configuration at atoms C(2), C(3), C(6) and C(7) is *RSSS* (or *SRRR*). This gives rise to a *trans* arrangement in both compounds of the *tert*-butyl and hydroxyl substituents at chiral centres. In (2) pairs of molecules are linked through crystallographic symmetry centres by hydrogen bonds [O(9)···O(8)($1-x$, $1-y$, $1-z$) 2·809 (7) Å] to form dimers. Molecules of (5) are linked by hydrogen bonds [O(9)···O(8)(x, $-0·5+y$, $1·5-z$) 2·847 (7) Å] into infinite chains through crystallographic 2_1 screw axes. In (2) the hydroxyl group and the carbonyl group have a *cis* arrangement which facilitates dimer formation. Molecule (5) has an unfavourable arrangement for dimerization (the hydroxyl group and the carbonyl group are *trans* to each other) and hence chains are formed.

The geometry of the seven-membered ring in compound (2) is significantly different from that in compound (5) (see Table 3 for values of ring torsion angles). The seven-membered ring in compound (2) can best be described as a boat conformation with the atoms C(1), C(7), C(3) and C(4) defining the base plane [maximum deviation 0·042 (6) Å for C(3)], C(5) and C(6) defining the stern and C(2) the bow. Compound (5) has a sofa-type conformation with atoms C(2), C(3), C(4), C(5) and C(6) defining one plane [maximum deviation −0·060 (7) Å for C(3)] and C(6), C(7), C(1) and C(2) defining another [maximum deviation 0·126 (6) Å for C(1)], with interplanar angle 65 (2)°. In compound (2) all the interior angles of the ring [except for the angle at C(1)] are larger than would be expected on the basis of hybridization principles. Steric repulsions of substituent groups could only cause a reduction of the magnitude of the ring angles, therefore the angles in (2) probably indicate a highly strained system. The compound dimethyl 3-bromo-7-oxo-1-cyclohepten-1,2-dicarboxylate (Atwood, Williams, Garner & Cone, 1974) adopts a similar conformation to that of (2) and displays a similar pattern of interior angles. The argument for a strained system can also be presented in the case of (5) although the angle widening effect appears to be concentrated at the angles at C(4) and C(5). Steric repulsions (due to two extra methyl substituents) may also affect the interior bond angles in (5) and hence may account for the conformational differences from (2).

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Structure of 4-Deoxyphenobarbital

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Abstract. 5-Ethyl-5-phenyl-4,5-dihydro-2,6(1*H*,3*H*)-pyrimidinedione 1,4-dioxane solvate, $C_{12}H_{14}N_2O_2.0\cdot5C_4H_8O_2$, $M_r = 262\cdot29$, monoclinic, $P2_1/c$, $a = 6\cdot8256$ (5), $b = 27\cdot078$ (3), $c = 8\cdot5647$ (7) Å, $\beta = 116\cdot47$ (1)°, $V = 1417\cdot0$ Å^3, $Z = 4$, D_m (flotation in KI/H_2O) = 1·24 (1), $D_x = 1\cdot229$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0\cdot723$ mm^{-1}, $F(000) = 560$, $T = 282$ K, $R = 0\cdot046$ for 1720 observations, $wR = 0\cdot045$

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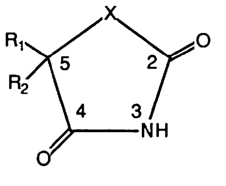
for all 2070 reflections. The 4-deoxyphenobarbital heterocyclic ring is twisted with C(5) displaced toward the phenyl group and C(4) displaced in the opposite direction from the least-squares plane defined by the other atoms in the ring. The N—C(carbonyl) bond distances are asymmetric with the N(3)—C(2) distance [1·314 (3) Å] significantly shorter than the N(1)—C(2) distance [1·378 (4) Å] indicating increased double-bond character in the former bond. Chains of intermolecular hydrogen bonds are formed between the acceptor O(2) and donors N(1) and N(3). The other acceptor atom, O(6), is not involved in any short intermolecular contacts. The 1,4-dioxane molecules, which sit on centers of symmetry, adopt the chair conformation and form normal van der Waals contacts with the deoxyphenobarbital molecules.

Introduction. Phenobarbital and diphenylhydantoin are two of the most widely used drugs for the control of epileptic seizures. These compounds possess two hydrophobic (phenyl, ethyl) groups attached to the tetrahedral carbon of a heterocyclic ring which contains two hydrogen-bond donor and at least two hydrogen-bond acceptor atoms. Although the mechanisms of action of these drugs are not known, relative placements of the ketonic O atoms and the hydrophobic groups in three-dimensional space are considered important stereochemical factors in determining anticonvulsant activity (Camerman & Camerman, 1974, 1977).



Phenobarbital $R1=C_6H_5$, $R2=CH_3CH_2$, $X = \overset{O}{\overset{\|}{C}}NH$
Diphenylhydantoin $R1=R2=C_6H_5$, $X=NH$

Chemically, the barbiturates differ from the hydantoins by the presence of a third carbonyl group adjacent to the tetrahedral carbon in their heterocyclic rings. Pharmacologically, they differ in that the barbiturates are potent sedatives in addition to their anticonvulsant properties while hydantoins are not. In light of the postulated stereochemical basis of anticonvulsant activity, phenobarbital's third carbonyl O atom may not be necessary for binding to the receptors that mediate anticonvulsant action but may be important for other pharmacological properties. Crystallographic studies on hydrogen-bonded complexes between barbiturates and adenine derivatives (Kim & Rich, 1968; Voet, 1972; Voet & Rich, 1972; Epstein, Zeiger, Crocker & Voet, 1976) have shown that the carbonyl groups adjacent to the tetrahedral carbon are poor hydrogen-bond acceptors. These results suggest that a derivative such as 4-deoxyphenobarbital, which has one of these carbonyl groups reduced to a methylene, would possess hydrogen-bonding properties and chemical features common to both phenobarbital and diphenylhydantoin and thus could be a potential new anticonvulsant.

The pharmacological profiles of 4- (or 6-) deoxybarbiturates are unknown at this time. However, derivatives such as 4-deoxyphenobarbital, as long as they fulfil the stereochemical criteria mentioned above, could provide a basis for the development of new anticonvulsant drugs. In view of this potential we synthesized 4-deoxyphenobarbital and determined its crystal and molecular structure in order to provide accurate stereochemical parameters for this class of barbiturate derivatives and to examine any other structural changes that may accompany the reduction of the carbonyl group to the methylene. The stereochemical parameters obtained will be useful in correlating structure with biological activity when pharmacological data become available.

Experimental. Compound prepared by the hydroboration method (Smissman, Matuszak & Corder, 1964). Needle-like crystals grown from methanol/dioxane mixture. Density measured by flotation in aqueous KI. Preliminary photographic data indicated a monoclinic system with systematic absences $0k0$, $k = 2n + 1$, $h0l$, $l = 2n + 1$ defining the space group as $P2_1/c$. Cell dimensions were refined by least squares using 18 reflections with $19 < 2\theta < 78°$ on a FACS-I Picker diffractometer. Intensity data from a crystal measuring $0·51 \times 0·13 \times 0·13$ mm were collected up to $2\theta = 120°$ ($0 \leq h \leq 7$, $0 \leq k \leq 30$, $-9 \leq l \leq 8$) with θ–2θ scans using Ni-filtered Cu $K\alpha$ radiation. Three standard reflections monitored at 200 reflection intervals showed no significant intensity variations. Total number of reflections measured, 2070; 1720 with $I > 2\sigma(I)$, where $\sigma(I)$ was obtained from counting statistics, were classified as observed. Lorentz and polarization factors were applied but no absorption corrections were made. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All H atoms except dioxane H atoms located from difference Fourier maps. Solvent H atoms were initially assigned idealized coordinates. Full-matrix least-squares refinement minimized $\sum w(|F_o| - k|F_c|)^2$, $w = 1/\sigma^2(F)$, k = scale factor. Non-H atoms refined with anisotropic temperature factors, H-atom thermal parameters were held fixed to the final isotropic values of their carrier atoms. Final $R = 0·046$ for the observed data and $wR = 0{\cdot}045$ for all data. $S = 3{\cdot}13$. Max. Δ/σ of 0·1 in last least-squares refinement cycle. Final difference Fourier map showed maximum and minimum peak heights of 0·16 and −0·17 e Å^{-3}. C, N, O scattering factors from Cromer & Mann (1968), H-atom scattering factors from Stewart, Davidson & Simpson (1965). All calculations were carried out at the University of Washington Computing Services on a Cyber 180-855 computer with the *XRAY*76 system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. The atomic parameters are given in Table 1* and a view of the 4-deoxyphenobarbital molecule is shown in Fig. 1. Bond lengths and angles are given in Table 2.

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (× 10^3) *for non-*H *atoms with 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)
N(1)	5892 (3)	4328 (1)	150 (3)	49 (1)
C(2)	7566 (4)	4592 (1)	62 (3)	47 (1)
O(2)	7323 (3)	5044 (1)	−260 (3)	62 (1)
N(3)	9324 (3)	4342 (1)	293 (3)	48 (1)
C(4)	9651 (4)	3833 (1)	890 (3)	44 (1)
C(5)	7539 (4)	3533 (1)	16 (3)	38 (1)
C(6)	5752 (4)	3826 (1)	226 (3)	43 (2)
O(6)	4216 (3)	3635 (1)	361 (2)	59 (1)
C(7)	6718 (4)	3462 (1)	−1958 (3)	43 (1)
C(8)	7893 (5)	3610 (1)	−2836 (4)	57 (2)
C(9)	7059 (6)	3531 (1)	−4628 (4)	73 (2)
C(10)	5114 (6)	3299 (1)	−5537 (4)	77 (2)
C(11)	3945 (5)	3148 (1)	−4681 (4)	77 (2)
C(12)	4730 (4)	3229 (1)	−2916 (3)	60 (2)
C(13)	7872 (4)	3034 (1)	950 (3)	47 (2)
C(14)	9517 (5)	2702 (1)	725 (4)	60 (2)
O(1*D*)	632 (6)	9533 (1)	757 (4)	117 (2)
C(2*D*)	2046 (8)	9835 (2)	362 (8)	144 (5)
C(3*D*)	1552 (9)	10356 (2)	392 (7)	139 (4)

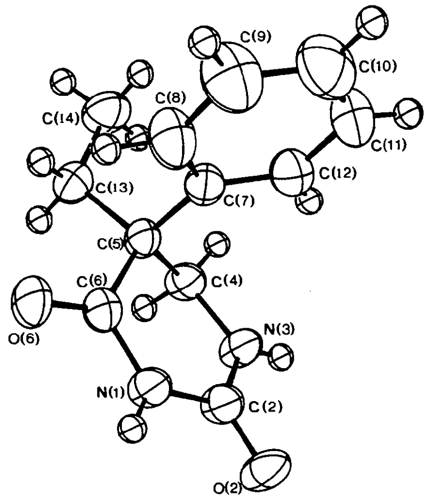


Fig. 1. *ORTEP*II drawing (Johnson, 1976) of 4-deoxyphenobarbital with 50% probability thermal ellipsoids for non-H atoms; H-atom spheres have been set to an arbitrary size.

The asymmetric unit contains one molecule of 4-deoxyphenobarbital and a half molecule of dioxane. As expected the reduction of the C(4) carbonyl group to methylene has destroyed the molecular symmetry about the the median plane through C(5), C(2) and O(2). Of particular interest is the shorter C(2)—N(3) distance [1·314 (3) Å] compared to the C(2)—N(1) distance [1·378 (4) Å]. The C(2)—N(3) and C(2)—N(1) bond distances found in phenobarbital and other barbiturates fall in the range between 1·36 and 1·37 Å (Singh, 1965; Bolton, 1963; Craven, Vizzini & Rodrigues, 1969; Craven & Vizzini, 1969; Williams, 1973; Voet, 1972; Voet & Rich, 1972; Kim & Rich, 1968; Epstein, Zeiger, Crocker & Voet, 1976). Crystal structure results on another clinically useful anticonvulsant, primidone (Yeates & Palmer, 1975), which has C(2)═O(2) reduced to CH_2 rather than C(4)═O(4), determined the carbonyl C—N distances to be 1·329 (6) Å which again are significantly shorter than those found in the normal barbiturate rings. These findings are consistent with increased double-bond character in these bonds since N atoms adjacent to the methylene group in deoxybarbiturates participate in electron delocalization with only one carbonyl group rather than two.

The 4-deoxy heterocyclic ring might best be described as twisted with C(5) displaced 0·444 (49) Å toward the phenyl group from the least-squares plane defined by N(1), C(2), N(3), O(2), C(6) and O(6), and C(4) displaced 0·266 (49) Å from this plane in the opposite direction. The C(5) displacement is equivalent to what is found in phenobarbital (Kim & Rich, 1968; Williams, 1973) and the overall geometry of the 4-deoxy ring is more similar to phenobarbital than to the boat-shaped ring found in primidone (Yeates & Palmer, 1975). The phenyl ring is planar to within 0·004 Å. The equations for the least-squares planes and deviations of the atoms from the planes are given in Table 3. The dihedral angle between the phenyl plane and barbiturate plane defined above is similar [88·9 (4)°] to the equivalent angles found in phenobarbital (87°) and primidone (86°).

The 4-deoxy derivatives are linked together in a chain of hydrogen bonds involving the O(2), N(1) and N(3) atoms. Fig. 2 shows a view of the unit-cell contents indicating the hydrogen-bonding scheme. The hydrogen bonds [N(1)···O(2), 2·811 (3),

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* Lists of observed and calculated structure factors, H-atom parameters, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53615 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bonding parameters* (Å, °)

N(1)—C(2)	1·378 (4)	C(7)—C(12)	1·386 (3)
N(1)—C(6)	1·366 (3)	C(8)—C(9)	1·395 (4)
C(2)—N(3)	1·314 (3)	C(9)—C(10)	1·357 (5)
C(2)—O(2)	1·248 (3)	C(10)—C(11)	1·365 (6)
N(3)—C(4)	1·453 (3)	C(11)—C(12)	1·377 (4)
C(4)—C(5)	1·529 (3)	C(13)—C(14)	1·516 (5)
C(5)—C(6)	1·532 (4)		
C(5)—C(7)	1·537 (3)	1,4-Dioxane	
C(5)—C(13)	1·535 (3)	O(1*D*)—C(2*D*)	1·416 (8)
C(6)—O(6)	1·220 (4)	O(1*D*)—C(3*D*)′	1·405 (6)
C(7)—C(8)	1·381 (5)	C(2*D*)—C(3*D*)	1·454 (8)

C(2)—N(1)—C(6)	126·6 (2)	N(1)—C(6)—O(6)	120·5 (2)
N(1)—C(2)—N(3)	116·7 (2)	C(5)—C(7)—C(8)	123·2 (2)
N(1)—C(2)—O(2)	119·2 (2)	C(5)—C(7)—C(12)	119·3 (3)
O(2)—C(2)—N(3)	124·1 (3)	C(8)—C(7)—C(12)	117·6 (2)
C(2)—N(3)—C(4)	121·9 (2)	C(7)—C(8)—C(9)	120·2 (3)
N(3)—C(4)—C(5)	111·7 (2)	C(8)—C(9)—C(10)	121·2 (4)
C(4)—C(5)—C(6)	107·0 (2)	C(9)—C(10)—C(11)	119·1 (3)
C(4)—C(5)—C(7)	114·0 (2)	C(10)—C(11)—C(12)	120·5 (3)
C(4)—C(5)—C(13)	109·0 (2)	C(11)—C(12)—C(7)	121·4 (3)
C(6)—C(5)—C(7)	105·9 (2)	C(5)—C(13)—C(14)	113·7 (3)
C(6)—C(5)—C(13)	109·7 (2)		
C(7)—C(5)—C(13)	111·1 (2)	1,4-Dioxane	
C(5)—C(6)—N(1)	115·8 (2)	C(2*D*)—O(1*D*)—C(3*D*)′	109·4 (4)
C(5)—C(6)—O(6)	123·7 (2)	O(1*D*)—C(2*D*)—C(3*D*)	111·6 (6)
		C(2*D*)—C(3*D*)—O(1*D*)′	112·2 (4)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	∠*D*—H···*A*
N(1)—H···O(2^{i})	0·84 (3)	1·98 (3)	2·811 (3)	171 (2)
N(3)—H···O(2^{ii})	0·83 (3)	2·01 (2)	2·839 (3)	171 (2)

Symmetry code: (i) $1-x$, $1-y$, $-z$; (ii) $2-x$, $1-y$, $-z$.

Table 3. *Equations of least-squares planes and atomic distances* (Å) *from planes*

Plane *A*: N(1), C(2), O(2), N(3), C(6), O(6)

$$-0·46512x + 4·03749y + 7·82336z - 1·53157 = 0,\ \sigma = 0·049\ \text{Å}$$

Plane *B*: C(7), C(8), C(9), C(10), C(11), C(12)

$$-2·92052x + 23·96583y + 0·22841z - 6·28782 = 0,\ \sigma = 0·004\ \text{Å}$$

Distances from *A*		Distances from *B*	
N(1)	0·059	C(7)	0·002
C(2)	0·019	C(8)	−0·006
O(2)	−0·039	C(9)	0·006
N(3)	0·017	C(10)	−0·002
C(6)	−0·078	C(11)	−0·002
O(6)	0·022	C(12)	0·002
C(4)	0·263	C(5)	−0·023
C(5)	−0·444		

Angle between plane *A* and plane *B* = 88·9 (4)°.

N(3)···O(2), 2·839 (3) Å] are formed between molecules related by a center of symmetry and unit-cell translations along **a**. Table 2 gives the hydrogen-bonding parameters. There are no short intermolecular contacts involving the O(6) atom nor are there any short contacts between the solvent dioxane molecules and 4-deoxyphenobarbital. It appears that the dioxane merely fills the channels between the chains of hydrogen-bonded deoxyphenobarbital molecules. By sitting on a crystallographic center of symmetry, the six-membered dioxane ring is restricted to the chair conformation.

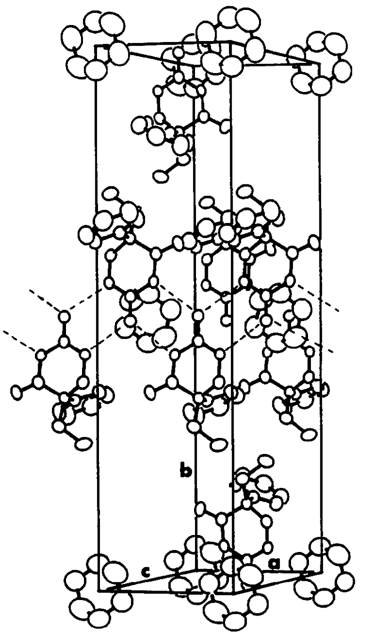

Fig. 2. View of the unit-cell packing. Dioxane molecules are located on centers of symmetry at 0,0,0 and $0,\frac{1}{2},\frac{1}{2}$. H atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds. Axial directions are as labeled.

Characterization of the chemical and pharmacological properties of 4-deoxyphenobarbital will be undertaken in collaboration with appropriate laboratories.

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Structure of 11,12-Bis(diazo)-11,12-dihydroindeno[2,1-*a*]fluorene

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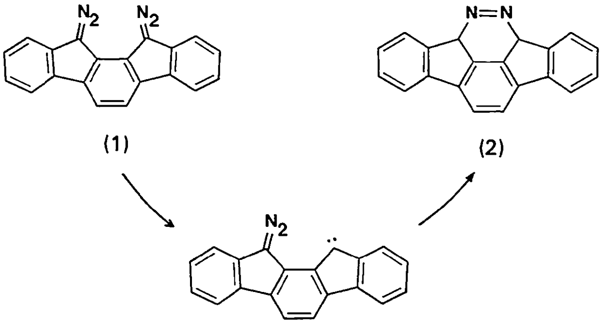
Abstract. $C_{20}H_{10}N_4$, $M_r = 306·32$, orthorhombic, *Pbcn*, $a = 14·53$ (2), $b = 10·007$ (9), $c = 20·033$ (4) Å, $V = 2913$ (4) Å^3, $Z = 8$, $D_x = 1·397$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·805$ cm^{-1}, $F(000) = 1264$, $T = 290$ K, $R = 0·059$ for 1259 observed independent reflections. The indenofluorene ring is planar and considerable distortion of bond angles and bond lengths is observed in the two closely located diazo groups. In the crystal structure, pairs of molecules with antiparallel orientations form a herringbone packing pattern, commonly observed in polycondensed aromatic hydrocarbons.

Introduction. Photochemical or thermal decomposition of 11,12-bis(diazo)-11,12-dihydroindeno[2,1-*a*]fluorene (1) is expected to give two carbene centers which are capable of conjugation. It is interesting to compare the spin multiplicity and electronic structure of the dicarbene to those of *m*-phenylenebis(phenylmethylene) which has quintet spin multiplicity. The diazo compound (1) was obtained as green needles with the characteristic double absorption maxima at 490 ($\varepsilon = 36$) and 568 nm ($\varepsilon = 16$), suggesting strong through-space interaction between the two diazo chromophores (Sugawara, Bethell & Iwamura, 1984). Ready formation of an aromatic cyclic azine (2) is further evidence for the close proximity of the two diazo groups in (1).

The present paper reports the X-ray crystal structure of the bis(diazo) compound (1), focusing attention on the local environment of the two closely located diazo groups.



Experimental. The title compound (1) was prepared and crystallized by the method reported previously (Bethell, Gallagher & Bott, 1989); dark-green crystal with dimensions 0·70 × 0·20 × 0·20 mm. Data collection: Rigaku AFC-5 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation; cell parameters refined with 25 strong reflections ($20 < 2\theta < 25°$), ω–2θ scan, scan speed 4° min^{-1}. Three standard reflections (301, $\bar{1}0\bar{5}$ and $19\bar{4}$) showed no significant variations; 3791 reflections ($2 < 2\theta < 45°$; $0 \leq h \leq 15$, $0 \leq k \leq 10$, $0 \leq l \leq 21$) collected, 1259 [$|F_o| > 3\sigma(F_o)$] were used for analysis. Data corrected for Lorentz and polarization effects, no absorption correction applied ($\mu r < 1$). Structure solved by direct methods using *SAPI85* (Fan, 1986), block-diagonal least-squares refinement on *F*, scattering factors from *International Tables for X-ray Crystal-*

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