fine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994), *SHELXTL-Plus* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1266). Services for accessing these data are described at the back of the journal.

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5-(4-Pyridyl)-5-phenylhydantoin†

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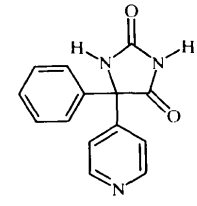
Abstract

The structure of the title compound, $C_{14}H_{11}N_3O_2$, is reported. The hydantoin ring is nearly planar. Bond distances and angles within the hydantoin ring are similar to those of the known 5,5-diphenylhydantoin. The angles between the hydantoin and the phenyl and pyridyl rings are very unequal, unlike those in 5,5-diphenylhydantoin. There is an intermolecular hydrogen-bonding network involving the amido H atoms as donors and the pyridyl-N atom and one carbonyl-O atom as acceptors; there is no intramolecular hydrogen bonding.

Comment

As part of an ongoing search for new stable *N*-halamine bactericides for treatment of aqueous solutions (Kohl *et al.*, 1980; Burkett *et al.*, 1981; Worley & Burkett, 1984; Barnela *et al.*, 1987; Worley *et al.*, 1987; Worley & Williams, 1988; Eknoian *et al.*, 1998), the title compound, (I), was prepared as a precursor. The structure was determined as part of the characterization process.



(I)

The compound crystallizes as a molecular lattice; the molecule is depicted in Fig. 1. Each molecule donates two intermolecular hydrogen bonds (H1 and H2) and accepts two (through N3 and O1); there is no intramolecular hydrogen bonding. The hydantoin ring is nearly planar; no atom deviates from the least-squares plane through the five atoms by more than 0.03 Å. If the ring is viewed as an envelope, with C3 out of the plane of the other atoms, the fold at the C2—N2 line is only 0.9 (2)°. This contrasts strongly with two oxazolidinone structures recently reported

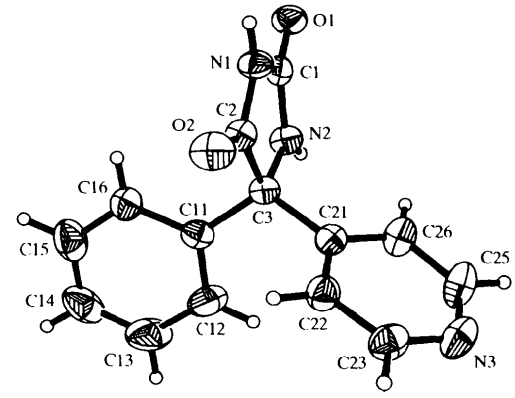


Fig. 1. View of 5-(4-pyridyl)-5-phenylhydantoin (50% probability ellipsoids).

† IUPAC name: 5-phenyl-5-(4-pyridyl)-2,4-imidazolidinedione.

from this laboratory (Eknoian *et al.*, 1998), in which the corresponding folds were 9.4 (2) and 19.5 (2)°. The phenyl and pyridyl rings are planar. Efforts to determine the correct enantiomorph (Flack, 1983) were inconclusive.

The geometry of the hydantoin ring is quite comparable to that reported for 5,5-diphenylhydantoin (Camerman & Camerman, 1971; Chattopadhyay *et al.*, 1993; Uno & Shimizu, 1980, report an adduct of diphenylhydantoin with a substituted pyrazolinone). In all of these structures, one C=O bond distance is *ca* 0.02 Å longer than the other. In the title structure, as in the adduct structure, the carbonyl bond (C1=O1) remote from the quaternary carbon (C3) is the longer. In diphenylhydantoin itself, the remote bond is shorter. We attribute this subtle difference to hydrogen bonding. In both the title compound and the adduct structure, the remote oxygen is a hydrogen-bond acceptor; the other oxygen does not accept any hydrogen bond. In diphenylhydantoin itself, both O atoms are hydrogen-bond acceptors. The remote oxygen forms the longer (and presumably weaker) H···O interaction, and therefore the remote carbonyl should have a slightly higher CO bond order and the shorter C=O bond. The angles between the hydantoin plane and the phenyl and pyridyl planes are quite different; the hydantoin–phenyl angle is 81.72 (7)°, whereas the hydantoin–pyridyl angle is 62.34 (7)°. In 5,5-diphenylhydantoin, the two hydantoin–phenyl angles are almost equal at approximately 114°. In the diphenylhydantoin–pyrazolinone adduct the angles are 55.2 and 73.7°. In the more distantly related structure of 5,5-diphenyl-1,3-oxazolidine-2,4-dione (Codding, 1984), the two oxazolidine–phenyl angles (reported as obtuse angles) are 115.2 (5) and 105.7 (5)°. Some of these differences may reflect an accommodation to the requirements of a hydrogen-bonding network. The phenyl–pyridyl interplanar angle, 75.23 (7)°, compares with phenyl–phenyl angles of *ca* 90° in 5,5-diphenylhydantoin, 96.2 in the adduct and 79.7 (5)° in the oxazolidinedione.

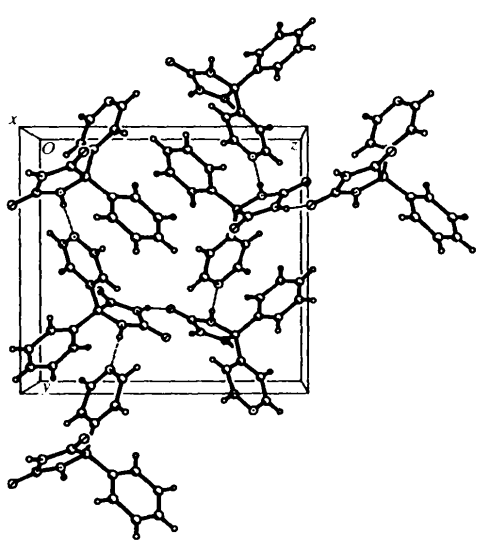


Fig. 2. Packing diagram for 5-(4-pyridyl)-5-phenylhydantoin.

Experimental

To a 250 ml round-bottom flask were added 4-benzoylpyridine (9.2 g, 0.05 mol), sodium cyanide (6.8 g, 0.13 mol), ammonium carbonate (28.8 g, 0.30 mol), ethanol (80 ml) and water (60 ml). The flask was sealed and the mixture was heated with stirring to 343 K for 24 h. The solution was cooled to room temperature and filtered. The solvent was removed from the filtrate under reduced pressure. The resulting solid was stirred with boiling water (150 ml) and filtered to isolate the product. Additional product precipitated when the filtrate cooled; this product was also recovered by filtration. The solid was dried in a vacuum oven, giving 5.9 g (47% yield) of a light-brown solid. The product was obtained as a racemate; no attempt was made to resolve it prior to growing the data crystal. The data crystal was grown from ethanol (m.p. 531–533 K); ^{1}H NMR (DMSO-d_6) δ: 7.36–7.41 (*m*, 8H), 8.60–8.63 (*m*, 2H), 9.48 (*s*, 1H) p.p.m. (TMS); ^{13}C NMR (DMSO-d_6) δ: 69.5, 121.5, 126.4, 128.4, 138.9, 148.1, 150.1, 156.1, 173.9 p.p.m. (TMS); IR (KBr pellet) 3036, 2807, 1775, 1725 cm^{-1}; MS (*m/z*) 253.

Crystal data

$C_{14}H_{11}N_3O_2$
M_r = 253.26
Orthorhombic
$P2_12_12_1$
a = 7.268 (2) Å
b = 12.747 (2) Å
c = 13.357 (4) Å
V = 1237.5 (5) Å^3
Z = 4
D_x = 1.357 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 7.54–14.31°
μ = 0.094 mm^{-1}
T = 294 (2) K
Thick plate
0.80 × 0.56 × 0.40 mm
Colorless

Data collection

Siemens *R3m* diffractometer
2θ–ω scans
Absorption correction:
ψ scan of five reflections (*XPREP* in *SHELXTL*; Siemens, 1994)
T_{min} = 0.940, T_{max} = 0.953
1285 measured reflections
1285 independent reflections
1199 reflections with $I > 2\sigma(I)$
θ_{max} = 25.05°
h = 0 → 8
k = 0 → 15
l = 0 → 15
2 standard reflections
every 98 reflections
intensity decay: <1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.078
S = 1.11
1285 reflections
180 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.1957P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Amide H atoms refined; others riding with fixed U_{iso}

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—O1	1.225 (2)	C12—C13	1.377 (3)
C1—N2	1.337 (3)	C13—C14	1.360 (4)
C1—N1	1.390 (3)	C14—C15	1.353 (4)
N1—C2	1.357 (3)	C15—C16	1.391 (3)
C2—O2	1.203 (2)	C21—C26	1.377 (3)
C2—C3	1.551 (3)	C21—C22	1.386 (3)
C3—N2	1.461 (3)	C22—C23	1.379 (3)
C3—C21	1.528 (3)	C23—N3	1.339 (3)
C3—C11	1.533 (3)	N3—C25	1.324 (3)
C11—C16	1.374 (3)	C25—C26	1.389 (3)
C11—C12	1.385 (3)		
O1—C1—N2	128.0 (2)	C16—C11—C3	120.9 (2)
O1—C1—N1	124.1 (2)	C12—C11—C3	120.4 (2)
N2—C1—N1	107.8 (2)	C13—C12—C11	120.5 (2)
C2—N1—C1	112.4 (2)	C14—C13—C12	120.6 (3)
O2—C2—N1	127.6 (2)	C15—C14—C13	119.5 (2)
O2—C2—C3	126.2 (2)	C14—C15—C16	121.1 (3)
N1—C2—C3	106.1 (2)	C11—C16—C15	119.7 (2)
N2—C3—C21	112.7 (2)	C26—C21—C22	117.8 (2)
N2—C3—C11	110.75 (15)	C26—C21—C3	121.7 (2)
C21—C3—C11	112.9 (2)	C22—C21—C3	120.5 (2)
N2—C3—C2	100.7 (2)	C23—C22—C21	119.2 (2)
C21—C3—C2	108.08 (15)	N3—C23—C22	123.8 (2)
C11—C3—C2	111.0 (2)	C25—N3—C23	115.9 (2)
C1—N2—C3	112.7 (2)	N3—C25—C26	124.8 (2)
C16—C11—C12	118.5 (2)	C21—C26—C25	118.4 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.82 (3)	2.02 (3)	2.832 (2)	169 (3)
N2—H2···N3^{ii}	0.88 (3)	2.08 (3)	2.950 (3)	173 (2)

Symmetry codes: (i) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$; (ii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$.

Data collection: *P3/PC* (Siemens, 1989). Cell refinement: *P3/PC*. Data reduction: *SHELXTL* (Siemens, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1423). Services for accessing these data are described at the back of the journal.

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6-[*N*-(2-Hydroxyphenyl)aminomethylene]-cyclohexa-2,4-dien-1-one

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Abstract

The structure determination of the title compound, $C_{13}H_{11}NO_2$, establishes the tautomeric keto form of the salicylaldimine. The asymmetric unit consists of two crystallographically independent molecules which are essentially planar and are approximately orthogonal to each other. Strong intramolecular N—H···O and intermolecular O—H···O interactions influence the conformation of the molecules and the crystal packing. Intermolecular hydrogen bonds link the molecules in infinite chains.

Comment

Salicylaldimines have been used extensively as ligands in coordination chemistry because of their diverse chelating ability (Long, 1995; Garnovski *et al.*, 1993). The intramolecular hydrogen bond between O and N atoms in these systems plays a vital role in the formation of Schiff base compounds in the solid state by proton transfer from the hydroxyl-O atom to the imine-N atom (Hadjoudis *et al.*, 1987; Elerman *et al.*, 1997). The charge transfer through overlapping intermolecular π-orbitals, with the associated change in the π-electron configuration in these compounds, also provides a basis for the development of molecular switches (Xu *et al.*, 1994). This X-ray crystallographic study was undertaken in order to establish the solid-state molecular structure of (I) and to build up a hierarchy for N-substituted salicylaldimines.