

Hydantoin and hydrogen-bonding
patterns in hydantoin derivativesFang-Lei Yu,^a Carl H. Schwalbe^{a*} and David J. Watkin^b^aAston Pharmacy School, Aston Triangle, Birmingham B4 7ET, England, and^bChemical Crystallography Laboratory, 9 Parks Road, Oxford OX1 3PD, England

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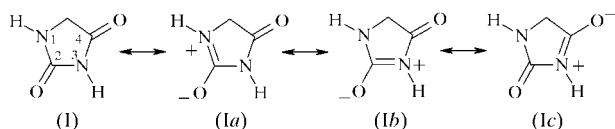
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The structure of hydantoin (imidazolidine-2,4-dione), C₃H₄N₂O₂, has been determined from a twinned crystal. The two carbonyl bond lengths are nearly equal, even though one of them adjoins electron-donating NH groups to either side while the other is adjacent to only one. *Ab initio* molecular-orbital calculations yield more negative Löwdin charge on the former than the latter. Hydantoin molecules form two chains linked by N—H···O hydrogen bonds, from which inversion centres create a ‘chain of rings’. Out of 50 hydantoin moieties in 49 independent molecules of hydantoin derivatives in the Cambridge Structural Database [Version 5.25; Allen (2002). *Acta Cryst.* B58, 380–388], five show this arrangement, six are a variant using the same O atom twice, five form a chain of edge-fused 12-membered hydrogen-bonded rings, and all but three of the remainder have one eight-membered ring and/or one chain connecting their hydantoin rings.

Comment

Hydantoin, (I), is of interest as the parent compound of the anti-epileptic drug diphenylhydantoin and as a supramolecular synthon in its own right. Possessing equal numbers of hydrogen-bond donor (two ring NH) groups and acceptor (two carbonyl O) atoms, it can form intricate networks, but with a different presentation of these groups compared with the six-membered rings so often studied. Probably due to the difficulties with twinning described below, no structure of hydantoin has appeared in the literature to date.



A view of (I) with the atom-numbering scheme is shown in Fig. 1. Electron donation from the ring N atoms to the carbonyl groups, as in resonance structures (Ia)–(Ic), would be expected to lengthen the C=O bonds and shorten the ring C–N bonds, C2=O2 being affected from both sides. The

experimental bond distances (Table 1) show no significant differences between the two carbonyl bond lengths or between N1–C2 and N3–C4, but C2–N3 is longer than their average by 0.024 Å (6σ). Thus, resonance structure (Ib) appears to be of limited importance. Both carbonyl groups are bent towards atom N3; the angle O2–C2–N1 exceeds O2–C2–N3 by 3.8 (3)° and the angle O4–C4–C5 exceeds O4–C4–N3 by 2.6 (3)°.

A search group was defined, consisting of a hydantoin ring with both NH groups unsubstituted and *sp*³ hybridization at C5. With disorder, errors or ions excluded and *R* < 0.1 required, a search (Bruno *et al.*, 2002) of the Cambridge Structural Database (CSD, Version 5.25; Allen, 2002) yielded 41 hits with 50 hydantoin rings in 49 independent molecules after removal of duplicate structure determinations. Mean values of the relevant bond distances, with standard error of the mean in parentheses, confirm the tendency in hydantoin: C2–O2 = 1.221 (1) Å, C4–O4 = 1.211 (1) Å, N1–C2 = 1.342 (2) Å, C2–N3 = 1.393 (2) Å and N3–C4 = 1.362 (1) Å. Bending of one carbonyl bond is common. The mean O2–C2–N1 angle is 127.9 (1)°, compared with 124.5 (1)° for O2–C2–N3, but the other C=O bond lies close to the exterior bisector, mean values being 126.8 (1)° for O4–C4–N3 and 126.3 (1)° for O4–C4–C5.

Ab initio molecular-orbital optimization of hydantoin with GAMESS (Schmidt *et al.*, 1990) in the 6-31G* basis set corroborates the near equality of C=O distances and yields N1–C2, C2–N3 and N3–C4 distances of 1.356, 1.391 and 1.367 Å, respectively. Atomic charges were calculated by the method of Löwdin (1950), chosen because it is based on orthogonalized orbitals and appears to be consistent with electronegativity. Values of –0.350 on atom N1, –0.382 on O2, –0.275 on N3 and –0.348 on O4 suggest that more negative charge is received by O2 than O4, and more given up by N3 than by N1.

As seen in Fig. 2, each molecule of (I) participates in N—H···O hydrogen bonds (Table 2), forming a chain of centrosymmetric rings with graph set C₂²(9) [R₂²(8)] [R₂²(8)] (Etter, 1990; Bernstein *et al.*, 1995). Of the 50 independent hydantoin rings found in the search of the CSD, this (pseudo-)centrosymmetric ‘chain of rings’ arrangement occurred in five (Table 3; the atom-numbering scheme has always been made to agree with the IUPAC system used in the present study),

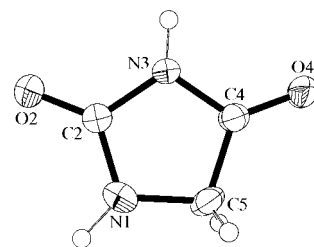


Figure 1

A view of the structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

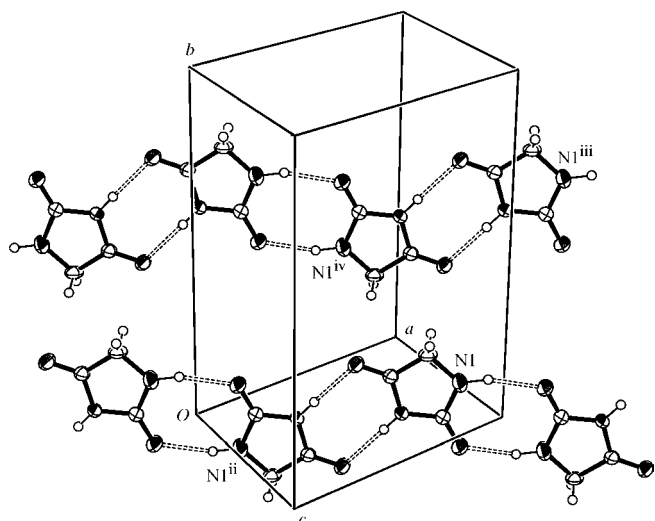
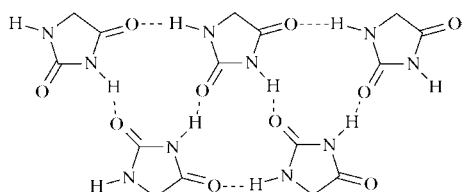


Figure 2

A view of two chains of (I) extending through the unit cell, with atom N1 of four representative molecules bearing a label. The symmetry codes are: (ii) $1-x, -y, 1-z$; (iii) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (iv) $\frac{3}{2}-x, \frac{1}{2}-y, 1-z$. Other molecules are generated from these by adding 1 to both x and z or subtracting 1 from both x and z . Hydrogen bonds are shown as dashed lines.

while another six exhibited a variant form in which the more electron-dense atom O2 simultaneously accepts N1—H1...O2 and N3—H3...O2 hydrogen bonds, while atom O4 accepts none (Table 4).

Chains are also possible. Hydantoin can create an infinite chain in graph set C(5) with atom N1 as donor and atom O4 as acceptor, simultaneously with another C(4) motif having atom N3 as donor and atom O2 as acceptor, thereby creating edge-fused $R_3^3(12)$ rings. This network had five occurrences, always in a non-centrosymmetric space group (Table 5).



Many hydantoin derivatives carry polar substituents on the 5-position, which divert some or all of the hydrogen bonding away from the ring. Thus, 21 molecules participate in a single $R_2^2(8)$ motif, 11 involving atom N1 as donor and atom O2 as acceptor, seven with N3 and O2, two with N3 and O4, and one with heterogeneous involvement of N1 with O4 and N3 with O2 (Table 6). Single chains also occur frequently, two C(4) using atoms N1 and O2, five C(5) with N1 and O4, four C(4) with N3 and O2 but none with N3 and O4, and one with heterogeneous chains (Table 7). Finally, three rings only interact with side groups (Table 8). (The total exceeds 50 because two molecules forming one ring and one chain are double-counted.) Of the potential hydrogen-bond donors and acceptors, atom N1 is left unused only three times, O2 just once and N3 not at all, but the less highly charged and less

accessible atom O4 is unused 24 times. Thus, hydantoin is a versatile supramolecular synthon providing a challenge to attempts at *a priori* prediction.

Experimental

Crystals of (I), in habits ranging from stubby needles to tabular blocks, were grown by slowly cooling to room temperature a saturated aqueous solution prepared at 353 K. An attempt to obtain a more tractable crystal form by diffusing acetic acid vapour into a sample of hydantoin in 12.5% sodium hydroxide solution yielded a conglomerate of sticky needles showing the same unit cell, space group and propensity to form twins.

Crystal data

$C_3H_4N_2O_2$
 $M_r = 100.08$
 Monoclinic, $C2/c$
 $a = 9.3538$ (7) Å
 $b = 12.1757$ (11) Å
 $c = 7.2286$ (6) Å
 $\beta = 104.593$ (4)°
 $V = 796.70$ (11) Å³
 $Z = 8$
 $D_x = 1.669$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 730 reflections
 $\theta = 5-27^\circ$
 $\mu = 0.14$ mm⁻¹
 $T = 190$ K
 Plate, colourless
 $0.18 \times 0.10 \times 0.02$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (DENZO and SCALEPACK; Otwinowski & Minor, 1997)
 $T_{min} = 0.99$, $T_{max} = 1.00$
 1593 measured reflections

886 independent reflections
 886 reflections with $I > -10\sigma(I)$
 $R_{int} = 0.02$
 $\theta_{max} = 27.4^\circ$
 $h = -12 \rightarrow 12$
 $k = -14 \rightarrow 15$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R(F) = 0.061$
 $wR(F^2) = 0.131$
 $S = 1.13$
 886 reflections
 77 parameters
 Only coordinates of H atoms refined

$w = 1/[\sigma^2(F^*) + 1.89P]$
 where $P = \frac{1}{3}\max(F_o^2, 0) + \frac{2}{3}F_c^2$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.27$ e Å⁻³
 $\Delta\rho_{min} = -0.25$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C2—O2	1.222 (3)	C4—O4	1.225 (3)
C2—N1	1.371 (3)	C4—C5	1.460 (3)
C2—N3	1.393 (3)	C5—N1	1.457 (3)
N3—C4	1.367 (3)		
O2—C2—N1	128.2 (2)	O4—C4—N3	125.3 (2)
O2—C2—N3	124.4 (2)	C5—C4—N3	106.8 (2)
N3—C2—N1	107.4 (2)	N1—C5—C4	104.7 (2)
C4—N3—C2	111.67 (19)	C2—N1—C5	109.4 (2)
O4—C4—C5	127.9 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1...O2 ⁱ	0.95 (3)	2.01 (3)	2.913 (3)	158
N3—H3...O4 ⁱⁱ	0.87 (4)	1.98 (4)	2.852 (3)	176

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

Table 3

Hydantoin derivatives in the CSD forming the same $C_2^2(9)$ [$R_2^2(8)$] [$R_2^2(8)$] network as hydantoin or a comparable network with a pseudo-centre of inversion.

Refcode	Space group	$D-H \cdots A$	Reference
BCOCHY	$C2/m$	$N1-H \cdots O2$ $N3-H \cdots O4$	Smith-Verdier <i>et al.</i> (1979a)
GRNSHY, mol 1	$P\bar{1}$	$N1-H \cdots O2''$ $N3-H \cdots O4''$	Florencio <i>et al.</i> (1980)
GRNSHY, mol 2	$P\bar{1}$	$N1-H'' \cdots O2$ $N3-H'' \cdots O4$	
OCSHYD	$C2/c$	$N1-H \cdots O2$ $N3-H \cdots O4$	Miller & McPhail (1979)
VARBAR	$P2_1/c$	$N1-H \cdots O2$ $N3-H \cdots O4$	Rizzi <i>et al.</i> (1989)

Table 4

Hydantoin derivatives forming a $C_1^1(4)$ $C_1^1(4)$ [$R_2^2(8)$] network based on $N1-H \cdots O2$ and $N3-H \cdots O2$ hydrogen bonds with $O4$ not used.

Refcode	Space group	$D-H \cdots A$	Reference
ADUQOF	$P2_12_12_1$	$N1-H \cdots O2$ $N3-H \cdots O2$	Beilles <i>et al.</i> (2001)
BEPNIT	$P2_12_12_1$	$N1-H \cdots O2$ $N3-H \cdots O2$	Cassady & Hawkinson (1982)
HPHCMS	$P2_1$	$N1-H \cdots O2$ $N3-H \cdots O4$	Koch <i>et al.</i> (1975)
OGUVIV	$P2_1$	$N1-H \cdots O2$ $N3-H \cdots O4$	Stalker <i>et al.</i> (2002)
XERTUJ, mol 1	$P2_1$	$N1-H \cdots O2''$ $N3-H \cdots O2''$	Koos <i>et al.</i> (2000)
XERTUJ, mol 2	$P2_1$	$N1-H'' \cdots O2$ $N3-H'' \cdots O2$	

Table 5

Hydantoin derivatives forming a chain of edge-fused $R_3^3(12)$ rings.

Refcode	Space group	$D-H \cdots A$	Reference
DAFFIZ01	$P2_12_12_1$	$N1-H \cdots O4$ $N3-H \cdots O2$	Sarges <i>et al.</i> (1985)
LABTIR	$P2_12_12_1$	$N1-H \cdots O4$ $N3-H \cdots O2$	Coquerel <i>et al.</i> (1993)
PHYDAN	$Pn2_1a$	$N1-H \cdots O4$ $N3-H \cdots O2$	Camerman & Camerman (1971)
PIPVAL	$P2_1$	$N1-H \cdots O4$ $N3-H \cdots O2$	Modric <i>et al.</i> (1993)
YECDOZ	$P2_12_12_1$	$N1-H \cdots O4$ $N3-H \cdots O2$	Park <i>et al.</i> (1994)

H-atom positions were refined freely, and $U_{iso}(H)$ values were set initially to $1.2U_{eq}(C,N)$ and not refined further. The data were refined as a two-component twin, (1 0 0, 0 1 0, 0 0 1) and (1 0 0.652, 0 $\bar{1}$ 0, 0 0 $\bar{1}$), with twin element scale factors of 0.869 (6) and 0.131 (6).

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994) and SHELXL97 (Sheldrick, 1997); program(s) used to refine structure: CRYSTALS (Watkin *et al.*, 1999); molecular graphics: CAMERON (Watkin *et al.*, 1996) and ORTEPII (Johnson, 1976); software used to prepare material for publication: CRYSTALS.

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Table 6

Hydantoin derivatives forming a single $R_2^2(8)$ motif.

Refcode	Space group	$D-H \cdots A$	Reference
BICSIP	$P2_1/n$	$N1-H \cdots O2$	Florencio <i>et al.</i> (1982)
CECKUQ	$P\bar{1}$	$N1-H \cdots O2$	Galvez <i>et al.</i> (1983)
DPHEAD20, mol 1	$P\bar{1}$	$N1-H \cdots O2$	Mastropaolo <i>et al.</i> (1983)
EZOSHY	$P2_1/c$	$N1-H \cdots O2$	Smith-Verdier <i>et al.</i> (1979b)
MANHDT10	$P2_1/n$	$N1-H \cdots O2$	Vilches <i>et al.</i> (1981)
MESYIS, mol 1	$P1$	$N1-H \cdots O2''$	Koos <i>et al.</i> (2001)
MESYIS, mol 2	$P1$	$N1-H'' \cdots O2$	
MGSHYD10, mol 1	$P2_1$	$N1-H \cdots O2''$	Florencio <i>et al.</i> (1978a)
MGSHYD10, mol 2	$P2_1$	$N1-H'' \cdots O2$	
MZBSHY	$P2_1/n$	$N1-H \cdots O2$	Florencio <i>et al.</i> (1979)
TRSHYD10	$P2_1/c$	$N1-H \cdots O2$	Smith-Verdier <i>et al.</i> (1977)
ALATIN01	$P2_1/c$	$N3-H \cdots O2$	Zhang <i>et al.</i> (1992)
DPHEAD20, mol 2	$P\bar{1}$	$N3-H \cdots O2$	Mastropaolo <i>et al.</i> (1983)
DPHPZL	$P\bar{1}$	$N3-H \cdots O2$	Uno & Shimizu (1980)
HEGRAH	$P2_1/c$	$N3-H \cdots O2$	Florencio <i>et al.</i> (1978b)
VAPZUH	$P2_1/c$	$N3-H \cdots O2$	Rizzi <i>et al.</i> (1989)
XERTOD, mol 1	$P4_3$	$N3-H \cdots O2''$	Koos <i>et al.</i> (2000)
XERTOD, mol 2	$P4_3$	$N3-H'' \cdots O2$	
AHINEK	$P2_1/c$	$N3-H \cdots O4$	SethuSankar <i>et al.</i> (2002)
NIVZOH	$P2_1/c$	$N3-H \cdots O4$	Benedetti <i>et al.</i> (1997)
COQEE†	$P2_1$	$N1-H \cdots O4''$ $N3-H'' \cdots O2$	Mullica <i>et al.</i> (1998)

† Although $Z' = 1$, the molecule has an independent hydantoin ring at each end.

Table 7

Hydantoin derivatives forming a single chain motif.

Refcode	Space group	$D-H \cdots A$	Reference
NIVZOH	$P2_1/c$	$N1-H \cdots O2$	Benedetti <i>et al.</i> (1997)
TOTPIB	$P2_1$	$N1-H \cdots O2$	Bravo <i>et al.</i> (1996)
BAGXOW	$P2_1/c$	$N1-H \cdots O4$	Terzis <i>et al.</i> (1981)
HOIMCU, mol 1	$P2_1/n$	$N1-H \cdots O4$	Poje <i>et al.</i> (1980)
ROKSOZ	$Pna2_1$	$N1-H \cdots O4$	Gauthier <i>et al.</i> (1997)
SINZEU	$P2_12_12_1$	$N1-H \cdots O4$	Galdecki & Karolak-Wojciechowska (1986)
VAPZUH	$P2_1/c$	$N1-H \cdots O4$	Rizzi <i>et al.</i> (1989)
GODRAS	$P2_12_12_1$	$N3-H \cdots O2$	Eknoian <i>et al.</i> (1999)
GOPZIU, mol 1	$P2_1$	$N3-H \cdots O2''$	Agasimundin <i>et al.</i> (1998)
GOPZIU, mol 2	$P2_1$	$N3-H'' \cdots O2$	
ROKSUF	$P2_1/c$	$N3-H \cdots O2$	Gauthier <i>et al.</i> (1997)
COQEE†	$P2_1$	$N3-H \cdots O2''$ $N1-H'' \cdots O4$	Mullica <i>et al.</i> (1998)

† Although $Z' = 1$, the molecule has an independent hydantoin ring at each end.

Table 8

Hydantoin derivatives without ring-to-ring hydrogen bonding.

Refcode	Space group	$D-H \cdots A$	Reference
HOIMCU, mol 2	$P2_1/n$	None	Poje <i>et al.</i> (1980)
JOPPAF	$P2_1$	None	Yamagishi <i>et al.</i> (1992)
QIBNIY	$P2_1/c$	None	SethuSankar <i>et al.</i> (2001)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1332). Services for accessing these data are described at the back of the journal.

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