Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Perhydrophthalimide

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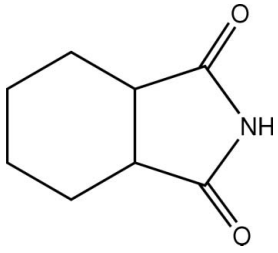
Received 23 August 2007; accepted 31 August 2007

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.090; data-to-parameter ratio = 17.7.

In the molecule of the title compound, $C_8H_{11}NO_2$, the cyclohexane ring adopts a chair conformation, while the pyrrole ring has an envelope conformation. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules.

Related literature

For related literature, see: Yamaguchi *et al.* (1998). For general background, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_{11}NO_2$
M_r = 153.18
Orthorhombic, $P2_12_12_1$
a = 6.7574 (12) Å
b = 7.8615 (14) Å
c = 14.371 (3) Å
V = 763.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.97, T_{max} = 0.98
4773 measured reflections
1843 independent reflections
1699 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.090
S = 1.07
1843 reflections
104 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.860 (17)	1.995 (17)	2.8537 (15)	175.8 (14)

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2317).

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  doi:10.1107/S1600536807042742

supplementary materials

Acta Cryst. (2007). E**63**, o3990 [**doi:10.1107/S1600536807042742**]

Perhydrophthalimide

D.-C. Wang, L. Jiang, W. Lin, Y. Pan and N.-N. Sun

Comment

Hexahydrophthalimide is a key intermediate for the synthesis of hypoglycemic drug mitiglinide (Yamaguchi *et al.*, 1998). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of the title compound, (I), (Fig. 1) the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987).

Ring A (C1—C6) is not planar, having total puckering amplitude, Q_T, of 0.519 (3) Å, and chair conformation [φ = −47.93 (3)° and θ = 161.98 (3)°] (Cremer & Pople, 1975). Ring B (C1/C6—C8/N1) has envelope conformation with atom C6 displaced by −0.394 (3) Å from the plane of the other four ring atoms.

In the crystal structure, the intermolecular N—H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they seem to be effective in the stabilization of the structure.

Experimental

Hexahydrophthalic anhydride (20 mmol) and urea (10 mmol) was added to a 25 ml flask. After the mixture was heated at reflux for 1 h, 5 ml of ice water was added, then the crystals of (I) were obtained by filtration. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

Refinement

H1A (for NH) was located in difference syntheses and only its position was refined [N—H = 0.860 (17) Å, U_{iso}(H) = 0.044 Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.98 and 0.97 Å for methine, and methylene H, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

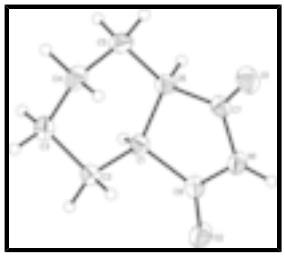

Fig. 1. A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

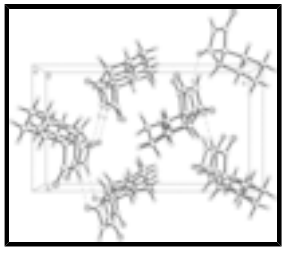

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

Perhydrophthalimide

Crystal data

$C_8H_{11}NO_2$	F_{000} = 328
M_r = 153.18	D_x = 1.333 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 6.7574 (12) Å	θ = 2.8–18.0º
b = 7.8615 (14) Å	μ = 0.10 mm^{-1}
c = 14.371 (3) Å	T = 273 (2) K
V = 763.4 (2) Å^3	Block, colorless
Z = 4	0.30 × 0.26 × 0.24 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.057
Radiation source: sealed tube	θ_{max} = 28.2º
Monochromator: graphite	θ_{min} = 2.8º
T = 273(2) K	h = −8→8
φ and ω scans	k = −10→6
Absorption correction: ψ scan (North *et al.*, 1968)	l = −19→14
T_{min} = 0.97, T_{max} = 0.98	3 standard reflections
4773 measured reflections	every 120 min
1843 independent reflections	intensity decay: 1%
1699 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.090	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.14 e Å^{-3}

1843 reflections

104 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.42 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.47695 (15)	0.91918 (12)	0.12610 (7)	0.0494 (3)
O2	−0.03151 (14)	0.63013 (12)	0.25855 (7)	0.0477 (3)
N1	0.20581 (16)	0.80816 (13)	0.20032 (7)	0.0368 (3)
H1A	0.148 (2)	0.903 (2)	0.2135 (11)	0.044*
C1	0.26087 (16)	0.51872 (14)	0.17742 (8)	0.0325 (3)
H1	0.2780	0.4225	0.2200	0.039*
C2	0.16634 (17)	0.46046 (16)	0.08509 (8)	0.0385 (3)
H2A	0.0623	0.3789	0.0982	0.046*
H2B	0.1066	0.5577	0.0545	0.046*
C3	0.3167 (2)	0.37982 (18)	0.02014 (9)	0.0454 (3)
H3A	0.3715	0.2787	0.0490	0.055*
H3B	0.2521	0.3461	−0.0372	0.055*
C4	0.48208 (18)	0.50434 (17)	−0.00141 (8)	0.0454 (3)
H4A	0.4270	0.6053	−0.0303	0.054*
H4B	0.5737	0.4528	−0.0451	0.054*
C5	0.59260 (17)	0.55445 (19)	0.08653 (10)	0.0455 (3)
H5A	0.6858	0.6443	0.0717	0.055*
H5B	0.6678	0.4574	0.1085	0.055*
C6	0.45644 (16)	0.61537 (15)	0.16426 (8)	0.0350 (3)
H6	0.5305	0.6043	0.2226	0.042*
C7	0.38956 (17)	0.79860 (15)	0.15769 (7)	0.0344 (3)
C8	0.12617 (18)	0.65170 (15)	0.21917 (8)	0.0340 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0569 (6)	0.0393 (5)	0.0521 (5)	−0.0139 (5)	0.0103 (4)	0.0003 (4)
O2	0.0505 (5)	0.0376 (5)	0.0551 (5)	−0.0033 (4)	0.0209 (4)	−0.0017 (4)
N1	0.0439 (5)	0.0273 (5)	0.0393 (5)	0.0005 (4)	0.0070 (4)	−0.0023 (4)
C1	0.0356 (5)	0.0284 (5)	0.0334 (5)	0.0012 (4)	0.0010 (4)	0.0028 (4)
C2	0.0345 (5)	0.0386 (6)	0.0424 (6)	−0.0026 (5)	−0.0014 (5)	−0.0081 (5)
C3	0.0486 (7)	0.0418 (7)	0.0459 (7)	0.0001 (6)	0.0026 (6)	−0.0123 (5)
C4	0.0481 (7)	0.0446 (7)	0.0435 (7)	0.0051 (6)	0.0121 (6)	−0.0024 (6)
C5	0.0320 (5)	0.0462 (7)	0.0584 (8)	0.0024 (5)	0.0070 (5)	−0.0020 (6)
C6	0.0318 (5)	0.0369 (6)	0.0363 (5)	0.0003 (5)	−0.0047 (4)	−0.0001 (5)
C7	0.0380 (6)	0.0349 (6)	0.0304 (5)	−0.0050 (5)	−0.0011 (4)	−0.0025 (4)
C8	0.0400 (6)	0.0316 (5)	0.0303 (5)	−0.0023 (5)	0.0027 (4)	0.0004 (4)

Geometric parameters (Å, °)

C1—C8	1.5104 (16)	C4—H4B	0.9700
C1—C6	1.5360 (15)	C5—C6	1.5243 (17)
C1—C2	1.5423 (16)	C5—H5A	0.9700
C1—H1	0.9800	C5—H5B	0.9700
C2—C3	1.5186 (17)	C6—C7	1.5127 (17)
C2—H2A	0.9700	C6—H6	0.9800
C2—H2B	0.9700	C7—O1	1.2055 (14)
C3—C4	1.5173 (18)	C7—N1	1.3867 (15)
C3—H3A	0.9700	C8—O2	1.2183 (15)
C3—H3B	0.9700	C8—N1	1.3696 (14)
C4—C5	1.5199 (19)	N1—H1A	0.860 (17)
C4—H4A	0.9700		
C8—C1—C6	103.01 (9)	H4A—C4—H4B	108.0
C8—C1—C2	107.32 (9)	C4—C5—C6	113.21 (10)
C6—C1—C2	113.41 (9)	C4—C5—H5A	108.9
C8—C1—H1	110.9	C6—C5—H5A	108.9
C6—C1—H1	110.9	C4—C5—H5B	108.9
C2—C1—H1	110.9	C6—C5—H5B	108.9
C3—C2—C1	112.06 (10)	H5A—C5—H5B	107.7
C3—C2—H2A	109.2	C7—C6—C5	115.70 (10)
C1—C2—H2A	109.2	C7—C6—C1	102.81 (9)
C3—C2—H2B	109.2	C5—C6—C1	117.01 (10)
C1—C2—H2B	109.2	C7—C6—H6	106.9
H2A—C2—H2B	107.9	C5—C6—H6	106.9
C4—C3—C2	110.42 (10)	C1—C6—H6	106.9
C4—C3—H3A	109.6	O1—C7—N1	124.22 (12)
C2—C3—H3A	109.6	O1—C7—C6	128.75 (11)
C4—C3—H3B	109.6	N1—C7—C6	106.95 (9)
C2—C3—H3B	109.6	O2—C8—N1	124.09 (11)
H3A—C3—H3B	108.1	O2—C8—C1	127.97 (11)

C3—C4—C5	111.06 (10)	N1—C8—C1	107.83 (10)
C3—C4—H4A	109.4	C8—N1—C7	112.99 (10)
C5—C4—H4A	109.4	C8—N1—H1A	123.5 (10)
C3—C4—H4B	109.4	C7—N1—H1A	123.5 (10)
C5—C4—H4B	109.4		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O2^{i}	0.860 (17)	1.995 (17)	2.8537 (15)	175.8 (14)

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

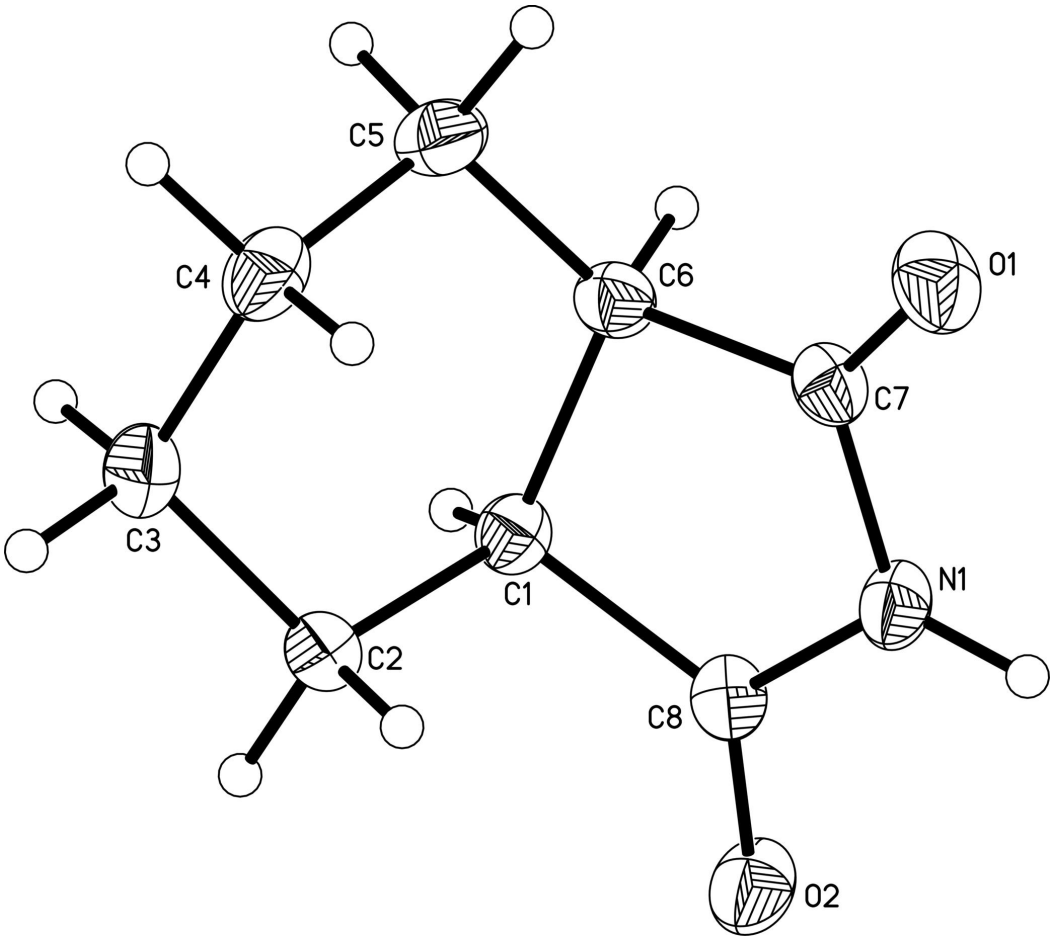

Fig. 2