organic papers

Yi-Tao Li, Tao Long and An-Xin Wu*

Key Laboratory of Pesticide and Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China

Correspondence e-mail:
chwuax@mail.ccnu.edu.cn

Key indicators

Single-crystal X-ray study
$T = 292$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.063
wR factor = 0.151
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

2,6-Dibenzyl-8b,8c-diphenyl-2,3a,4a,6,7a,8a-hexaazaperhydrocyclopenta[def]fluorene-4,8-dione

The molecule of the title compound, $C_{34}H_{32}N_6O_2$, exhibits normal geometric parameters. The two terminal phenyl rings make a dihedral angle of 69.24 (2)° with each other. The crystal packing is stabilized mainly by van der Waals interactions.

Comment

Glycoluril derivatives have applications in many areas, such as explosives, slow-release fertilizers, crosslinkers, iodogens, stabilisers of organic compounds against photodegradation, and reagents in combinatorial chemistry (Wu *et al.*, 2002). In this paper, we present the structure of the title compound, (I) (Fig. 1), as a continuation of our previous studies in this area (Wei & Wu, 2005).


(I)
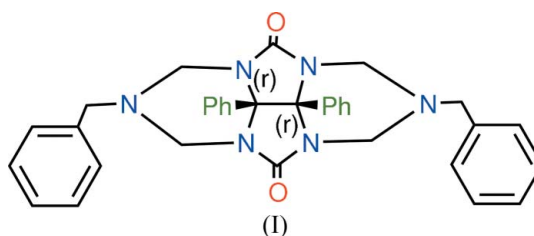
Experimental

Benzylamine (1.07 g, 10 mmol) and formaldehyde (2.4 g, 40 mmol) were added to a stirred solution of 3a,6a-diphenyl-tetrahydroimidazo[4,5-d]imidazole-2,5-dione (1.47 g, 5 mmol) in acetonitrile (50 ml) under a nitrogen atmosphere. The mixture was stirred overnight at room temperature. The solvent was evaporated to dryness and the compound was purified by column chromatography to yield (I) (2.50 g, 90%) as a colourless solid. Colourless block-like crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of an acetic acid–ethyl ester solution at 283 K.

Crystal data

$C_{34}H_{32}N_6O_2$
$M_r = 556.66$
Monoclinic, $P2_1/c$
$a = 11.7235$ (9) Å
$b = 18.3780$ (13) Å
$c = 14.1604$ (10) Å
$\beta = 109.903$ (1)°
$V = 2868.7$ (4) Å^3
$Z = 4$

$D_x = 1.289$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2525
 reflections
$\theta = 2.3–20.9°$
$\mu = 0.08$ mm^{-1}
$T = 292$ (2) K
Block, colourless
$0.20 \times 0.16 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector
 diffractometer
φ and ω scans
Absorption correction: multi-scan
 (*SADABS*; Bruker, 2000)
 $T_{min} = 0.984$, $T_{max} = 0.992$
16023 measured reflections

5026 independent reflections
3155 reflections with $I > 2\sigma(I)$
$R_{int} = 0.081$
$\theta_{max} = 25.0°$
$h = -10 \rightarrow 13$
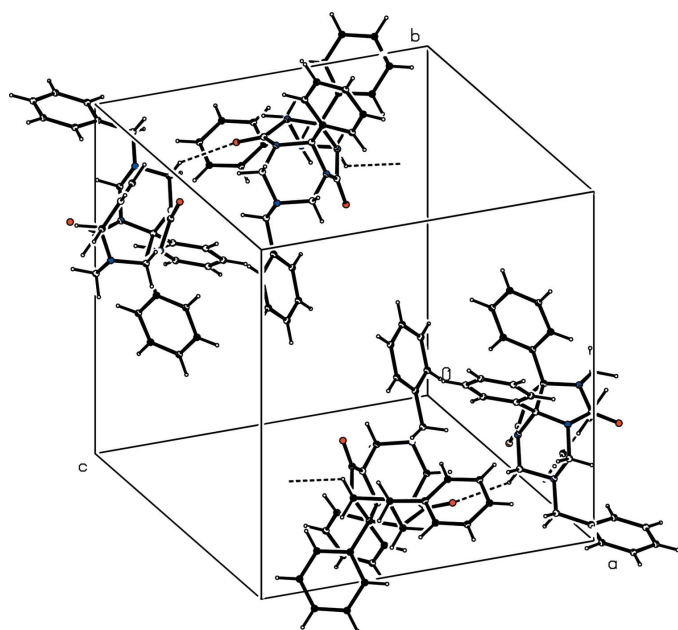$k = -19 \rightarrow 21$
$l = -16 \rightarrow 16$
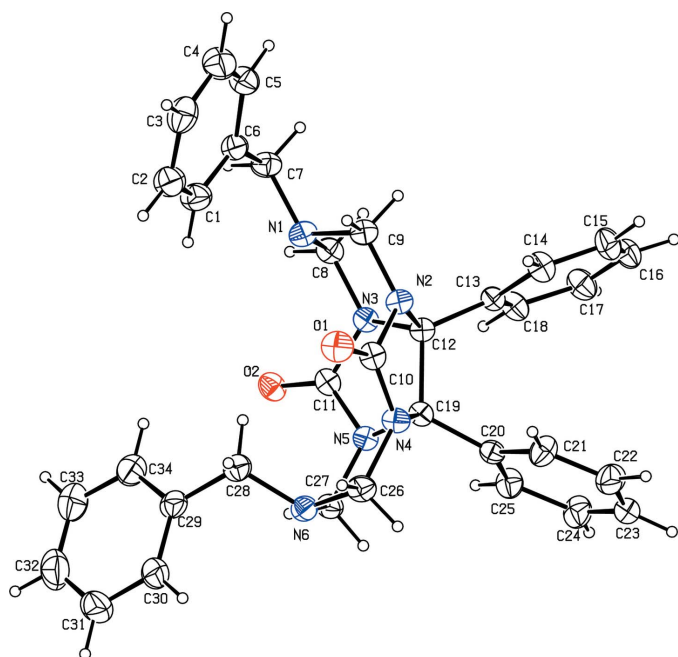
Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.063$	$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2]$
$wR(F^2) = 0.151$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
5026 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
379 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Figure 2
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

C6—C7	1.521 (4)	C12—N2	1.460 (3)
C7—N1	1.457 (3)	C12—C13	1.508 (3)
C9—N2	1.452 (3)	C12—C19	1.583 (3)
C9—N1	1.461 (3)	C13—C18	1.381 (3)
C10—O1	1.217 (3)	C13—C14	1.385 (3)
C10—N2	1.385 (3)	C14—C15	1.382 (4)
C5—C6—C7	121.1 (3)	C18—C13—C14	117.1 (2)
N1—C7—C6	112.1 (2)	C18—C13—C12	120.3 (2)
N2—C9—N1	110.2 (2)	C14—C13—C12	122.4 (2)
O1—C10—N2	125.1 (3)	C15—C14—C13	121.4 (3)
N4—C10—N2	108.3 (2)	C7—N1—C9	110.0 (2)
N3—C12—N2	110.54 (19)	C9—N1—C8	108.8 (2)
N2—C12—C13	112.6 (2)	C10—N2—C9	119.5 (2)
N2—C12—C19	102.54 (17)	C10—N2—C12	111.3 (2)
C13—C12—C19	115.82 (19)	C9—N2—C12	115.90 (18)
C5—C6—C7—N1	133.2 (3)	N2—C9—N1—C7	178.4 (2)
N3—C12—C13—C18	−40.0 (3)	N2—C9—N1—C8	−60.3 (2)
C19—C12—C13—C18	77.2 (3)	N3—C8—N1—C9	59.5 (2)
N3—C12—C13—C14	145.1 (2)	O1—C10—N2—C9	−25.5 (4)
C19—C12—C13—C14	−97.8 (3)	O1—C10—N2—C12	−164.9 (2)
C18—C13—C14—C15	−0.1 (4)	N1—C9—N2—C10	−83.4 (3)
C12—C13—C14—C15	175.0 (3)	C13—C12—N2—C10	−137.1 (2)
C13—C14—C15—C16	−1.6 (4)	O2—C11—N3—C8	26.8 (4)
C6—C7—N1—C9	−62.5 (3)	C13—C12—N3—C8	−82.8 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C8—H8A⋯O1^i	0.97	2.49	3.107 (3)	122

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

Methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.98 Å and U_{iso}(H) = $1.5U_{eq}$(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and with U_{iso}(H) = $1.2U_{eq}$(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

References

Bruker (2000). *SMART* (Version 5.618), *SAINT* (Version 6.02), *SADABS* (Version 2.03) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wei, F. & Wu, A. (2005). *Acta Cryst.* E**61**, o1453–o1455.
Wu, A., Fettinger, J. C. & Isaacs, L. (2002). *Tetrahedron*, **58**, 9769–9777.