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8b,8c-Diphenyl-2,6-dioxa-3a,4a,7a,8a-tetraazaperhydrocyclopenta[*def*]-fluorene-4,8-dione

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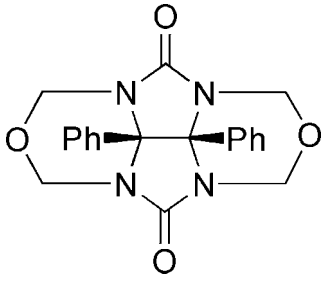
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.007 Å; R factor = 0.044; wR factor = 0.079; data-to-parameter ratio = 6.3.

The title compound, $C_{20}H_{18}N_4O_4$, is a glycoluril derivative. The oxadiazinane six-membered ring displays a normal chair conformation. Intermolecular C—H···O hydrogen bonds are present in the crystal structure.

Related literature

For related structures, see: Sijbesma *et al.* (1993). For related literature, see: Hof *et al.* (2002); van Nunen & Nolte (1997); Rowan *et al.* (1999); Wu *et al.* (2002).



Experimental

Crystal data

$C_{20}H_{18}N_4O_4$
M_r = 378.39
Monoclinic, *Cc*
a = 14.558 (3) Å
b = 14.870 (3) Å
c = 16.711 (3) Å
β = 91.695 (3)°
V = 3616.2 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 292 (2) K
0.34 × 0.18 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
13181 measured reflections
3192 independent reflections
1976 reflections with $I > 2\sigma(I)$
R_{int} = 0.103

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.079
S = 0.80
3192 reflections
505 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O8^{i}	0.97	2.40	3.188 (5)	138
C27—H27···O1^{ii}	0.93	2.30	3.208 (7)	166

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 2001); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek; 2003).

The authors thank Professor An-Xin Wu for technical assistance and Dr Meng Xiang-Gao for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2157).

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supplementary materials

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8b,8c-Diphenyl-2,6-dioxa-3a,4a,7a,8a-tetraazaperhydrocyclopenta[*def*]fluorene-4,8-dione

M. Gao and J.-J. Sun

Comment

The glycoluril skeleton has served as an important building block for the preparation of a wide variety of supramolecular assemblies (Wu *et al.*, 2002; Hof *et al.*, 2002). A molecular clip is a molecule with a rigid U-shaped cavity in which small aromatic guest molecules can be complexed by hydrogen bonding and aromatic stacking interactions (Rowan *et al.*, 1999). We report here the structure of the title glycoluril derivative (Fig. 1), which is an important intermediate for the preparation of molecular clips (Sijbesma *et al.*, 1993).

In the title glycoluril derivative, $C_{20}H_{18}N_4O_4$, the oxadiazinane six-membered ring displays a normal chair conformation. The crystal packing is stabilized by inversion-related intermolecular C—H···O hydrogen bonds, forming dimers (Table 1 and Fig. 2).

Experimental

The title compound was synthesized according to a reported procedure (Sijbesma *et al.*, 1993). Crystals appropriate for X-ray data collection were obtained by slow evaporation of a dichloromethane solution at 293 K.

Refinement

The value of R_{int} is rather high, due to the poor quality of the crystal. All H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Figures

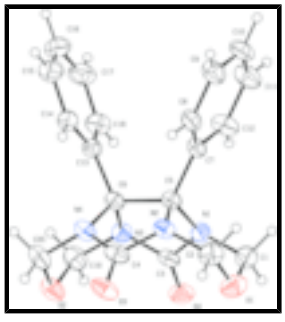

Fig. 1. The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

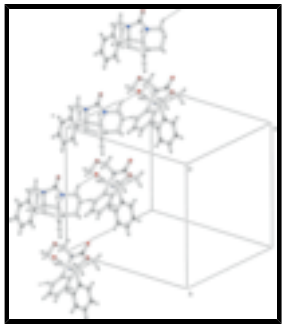

Fig. 2. A packing diagram for the title compound. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

8 b,8c-Diphenyl-2,6-dioxa-3a,4a,7a,8a-tetraazaperhydrocyclopenta[def]fluorene- 4,8-dione

Crystal data

$C_{20}H_{18}N_4O_4$	F_{000} = 1584
M_r = 378.39	D_x = 1.390 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 1636 reflections
a = 14.558 (3) Å	θ = 2.3–19.1º
b = 14.870 (3) Å	μ = 0.10 mm^{-1}
c = 16.711 (3) Å	*T* = 292 (2) K
β = 91.695 (3)º	Block, colorless
V = 3616.2 (11) Å^3	0.34 × 0.18 × 0.10 mm
Z = 8	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1976 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.103
Monochromator: graphite	θ_{max} = 25.0º
T = 292(2) K	θ_{min} = 2.0º
φ and ω scans	*h* = −17→17
Absorption correction: none	*k* = −17→17
13181 measured reflections	*l* = −19→19
3192 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.079	$(\Delta/\sigma)_{max}$ = 0.031
S = 0.80	$\Delta\rho_{max}$ = 0.21 e Å^{-3}

3192 reflections
505 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: none

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}
O2	0.2736 (2)	0.0000 (2)	1.07844 (17)	0.0625 (8)
N3	0.2004 (2)	−0.0824 (2)	0.9737 (2)	0.0427 (9)
O6	0.1405 (3)	0.2727 (2)	0.8421 (2)	0.0719 (11)
O4	0.32209 (19)	−0.1725 (2)	0.93918 (18)	0.0586 (8)
N7	0.1933 (2)	0.3802 (2)	0.6171 (2)	0.0495 (10)
C32	0.1428 (3)	0.5164 (3)	0.7808 (3)	0.0504 (12)
O3	0.3242 (2)	0.1647 (2)	0.92934 (18)	0.0636 (9)
O5	0.0984 (3)	0.2795 (2)	0.5416 (2)	0.0706 (11)
N2	0.2398 (2)	0.0739 (2)	0.8416 (2)	0.0441 (9)
O8	−0.0516 (2)	0.2753 (2)	0.7105 (2)	0.0715 (10)
N5	0.0513 (3)	0.3769 (2)	0.7679 (2)	0.0484 (10)
C4	0.2627 (3)	0.1102 (3)	0.9151 (3)	0.0487 (13)
O7	0.2886 (2)	0.2680 (2)	0.6714 (2)	0.0766 (11)
N1	0.2409 (2)	−0.0855 (2)	0.8472 (2)	0.0446 (9)
O1	0.3609 (2)	−0.0085 (2)	0.7869 (2)	0.0699 (9)
C33	0.1031 (3)	0.5211 (3)	0.6098 (3)	0.0490 (12)
N6	0.2125 (3)	0.3696 (2)	0.7499 (2)	0.0545 (11)
N8	0.0315 (3)	0.3751 (3)	0.6344 (2)	0.0524 (10)
C3	0.2633 (3)	−0.1176 (3)	0.9227 (3)	0.0418 (11)
C8	0.0670 (3)	0.0662 (3)	0.7534 (3)	0.0537 (12)
H8	0.0897	0.1222	0.7688	0.064*
C13	0.0529 (3)	−0.0009 (3)	0.9528 (2)	0.0430 (10)
C10	−0.0391 (3)	−0.0217 (3)	0.6751 (3)	0.0663 (14)
H10	−0.0875	−0.0252	0.6376	0.080*
N4	0.2018 (3)	0.0780 (2)	0.9695 (2)	0.0469 (9)
C19	0.2208 (3)	−0.0780 (3)	1.0588 (3)	0.0578 (12)
H19A	0.1638	−0.0767	1.0875	0.069*

H19B	0.2546	−0.1314	1.0755	0.069*
C26	0.1139 (3)	0.4291 (3)	0.6466 (3)	0.0466 (11)
C6	0.1544 (2)	−0.0026 (3)	0.9399 (2)	0.0396 (10)
C5	0.1822 (3)	−0.0061 (3)	0.8491 (2)	0.0412 (10)
C25	0.1305 (3)	0.4265 (3)	0.7406 (3)	0.0454 (11)
C24	0.0053 (3)	0.3347 (3)	0.7041 (3)	0.0553 (13)
C2	0.3063 (3)	0.0694 (3)	0.7790 (3)	0.0590 (13)
H2A	0.2743	0.0690	0.7272	0.071*
H2B	0.3453	0.1222	0.7816	0.071*
C20	0.2239 (3)	0.0786 (3)	1.0545 (3)	0.0604 (13)
H20A	0.2603	0.1315	1.0676	0.072*
H20B	0.1675	0.0820	1.0839	0.072*
C38	0.0201 (4)	0.3307 (4)	0.5589 (3)	0.0704 (16)
H38A	−0.0333	0.2917	0.5599	0.085*
H38B	0.0095	0.3751	0.5171	0.085*
C29	0.1627 (6)	0.6892 (4)	0.8406 (4)	0.091 (2)
H29	0.1692	0.7475	0.8601	0.110*
C37	0.1791 (4)	0.5719 (3)	0.5911 (3)	0.0608 (14)
H37	0.2373	0.5468	0.5974	0.073*
C18	0.0031 (4)	−0.0798 (4)	0.9634 (3)	0.0595 (14)
H18	0.0339	−0.1344	0.9682	0.071*
C40	0.0780 (5)	0.6480 (4)	0.8394 (3)	0.0802 (19)
H40	0.0272	0.6783	0.8584	0.096*
C23	0.2359 (3)	0.3305 (3)	0.6789 (3)	0.0552 (13)
C7	0.1050 (3)	−0.0101 (3)	0.7867 (2)	0.0416 (10)
C15	−0.0884 (4)	0.0822 (4)	0.9530 (3)	0.0724 (15)
H15	−0.1195	0.1368	0.9492	0.087*
C27	0.0685 (4)	0.5614 (3)	0.8097 (3)	0.0639 (14)
H27	0.0112	0.5336	0.8094	0.077*
C1	0.3063 (3)	−0.0867 (3)	0.7844 (3)	0.0591 (13)
H1A	0.3454	−0.1393	0.7904	0.071*
H1B	0.2740	−0.0907	0.7329	0.071*
C31	0.2287 (4)	0.5576 (4)	0.7824 (3)	0.0664 (15)
H31	0.2797	0.5279	0.7631	0.080*
C21	0.2212 (4)	0.3231 (3)	0.8259 (3)	0.0664 (16)
H21A	0.2736	0.2828	0.8251	0.080*
H21B	0.2322	0.3666	0.8684	0.080*
C12	0.0692 (3)	−0.0920 (3)	0.7631 (3)	0.0626 (13)
H12	0.0939	−0.1445	0.7851	0.075*
C30	0.2368 (5)	0.6445 (4)	0.8135 (4)	0.089 (2)
H30	0.2941	0.6722	0.8157	0.107*
C14	0.0050 (3)	0.0797 (4)	0.9487 (3)	0.0546 (13)
H14	0.0372	0.1332	0.9429	0.066*
C11	−0.0027 (3)	−0.0980 (3)	0.7074 (3)	0.0723 (15)
H11	−0.0261	−0.1538	0.6923	0.087*
C17	−0.0901 (4)	−0.0775 (4)	0.9669 (3)	0.0722 (15)
H17	−0.1229	−0.1307	0.9721	0.087*
C34	0.0177 (4)	0.5610 (4)	0.6019 (3)	0.0717 (16)
H34	−0.0347	0.5286	0.6143	0.086*

C39	0.1772 (4)	0.3359 (4)	0.5424 (3)	0.0643 (15)
H39A	0.1695	0.3808	0.5006	0.077*
H39B	0.2306	0.3000	0.5303	0.077*
C22	0.0637 (4)	0.3313 (4)	0.8440 (3)	0.0701 (16)
H22A	0.0732	0.3753	0.8863	0.084*
H22B	0.0089	0.2971	0.8554	0.084*
C35	0.0098 (5)	0.6486 (4)	0.5756 (4)	0.088 (2)
H35	−0.0477	0.6756	0.5704	0.106*
C9	−0.0044 (3)	0.0610 (3)	0.6977 (3)	0.0631 (13)
H9	−0.0292	0.1132	0.6753	0.076*
C16	−0.1361 (4)	0.0036 (5)	0.9628 (3)	0.0780 (17)
H16	−0.1996	0.0050	0.9668	0.094*
C41	0.0865 (6)	0.6952 (4)	0.5574 (4)	0.092 (2)
H41	0.0807	0.7545	0.5403	0.111*
C36	0.1708 (5)	0.6584 (4)	0.5633 (3)	0.0835 (19)
H36	0.2224	0.6909	0.5489	0.100*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.071 (2)	0.065 (2)	0.0495 (19)	0.0027 (19)	−0.0192 (15)	0.0024 (17)
N3	0.045 (2)	0.041 (2)	0.042 (2)	0.0059 (18)	−0.0019 (18)	0.0072 (17)
O6	0.082 (3)	0.052 (2)	0.081 (3)	0.000 (2)	−0.004 (2)	0.0136 (18)
O4	0.0484 (18)	0.0577 (19)	0.069 (2)	0.0152 (16)	−0.0124 (16)	0.0025 (16)
N7	0.046 (2)	0.045 (2)	0.058 (3)	0.001 (2)	0.002 (2)	−0.008 (2)
C32	0.063 (3)	0.037 (3)	0.051 (3)	−0.007 (3)	−0.013 (2)	0.001 (2)
O3	0.063 (2)	0.059 (2)	0.067 (2)	−0.0252 (18)	−0.0222 (17)	0.0044 (16)
O5	0.081 (3)	0.058 (2)	0.073 (3)	−0.011 (2)	0.003 (2)	−0.0200 (18)
N2	0.045 (2)	0.045 (2)	0.042 (2)	−0.0077 (18)	−0.0079 (18)	0.0072 (17)
O8	0.058 (2)	0.054 (2)	0.104 (3)	−0.016 (2)	0.0178 (19)	−0.021 (2)
N5	0.058 (3)	0.036 (2)	0.052 (2)	−0.008 (2)	0.002 (2)	−0.0065 (19)
C4	0.052 (3)	0.036 (3)	0.057 (3)	0.001 (2)	−0.020 (3)	0.009 (2)
O7	0.065 (2)	0.052 (2)	0.112 (3)	0.0186 (19)	−0.007 (2)	−0.010 (2)
N1	0.046 (2)	0.044 (2)	0.044 (2)	0.0087 (17)	−0.0023 (18)	−0.0031 (18)
O1	0.0510 (19)	0.081 (3)	0.078 (2)	−0.0008 (19)	0.0118 (16)	0.008 (2)
C33	0.059 (3)	0.042 (3)	0.045 (3)	0.003 (3)	−0.010 (2)	−0.001 (2)
N6	0.049 (2)	0.048 (2)	0.066 (3)	0.006 (2)	−0.014 (2)	0.001 (2)
N8	0.049 (2)	0.053 (2)	0.055 (3)	−0.014 (2)	−0.004 (2)	−0.010 (2)
C3	0.040 (3)	0.037 (3)	0.047 (3)	−0.001 (2)	−0.015 (2)	0.001 (2)
C8	0.059 (3)	0.056 (3)	0.045 (3)	0.009 (2)	−0.011 (2)	−0.008 (2)
C13	0.042 (2)	0.051 (3)	0.036 (2)	0.000 (3)	−0.0019 (18)	0.003 (2)
C10	0.061 (3)	0.080 (4)	0.056 (3)	0.006 (3)	−0.027 (3)	−0.007 (3)
N4	0.056 (2)	0.043 (2)	0.041 (2)	−0.0051 (19)	−0.0121 (19)	0.0018 (18)
C19	0.069 (3)	0.055 (3)	0.049 (3)	0.000 (3)	−0.007 (3)	0.009 (2)
C26	0.044 (3)	0.042 (3)	0.054 (3)	−0.001 (2)	−0.007 (2)	−0.003 (2)
C6	0.042 (2)	0.037 (2)	0.039 (2)	0.000 (3)	−0.007 (2)	0.002 (2)
C5	0.043 (2)	0.041 (2)	0.040 (2)	0.000 (2)	−0.0047 (19)	0.000 (2)
C25	0.046 (3)	0.038 (3)	0.052 (3)	−0.002 (2)	−0.005 (2)	−0.002 (2)

C24	0.042 (3)	0.039 (3)	0.085 (4)	0.002 (3)	0.004 (3)	−0.017 (3)
C2	0.054 (3)	0.059 (3)	0.064 (3)	−0.005 (3)	0.003 (3)	0.012 (3)
C20	0.077 (3)	0.055 (3)	0.048 (3)	−0.004 (3)	−0.016 (3)	−0.009 (2)
C38	0.066 (4)	0.074 (4)	0.071 (4)	−0.012 (3)	−0.005 (3)	−0.028 (3)
C29	0.130 (6)	0.038 (3)	0.102 (5)	−0.006 (4)	−0.066 (5)	−0.003 (3)
C37	0.065 (4)	0.048 (3)	0.070 (4)	0.001 (3)	0.002 (3)	0.007 (3)
C18	0.053 (3)	0.065 (4)	0.060 (4)	−0.005 (3)	−0.005 (3)	0.008 (3)
C40	0.111 (5)	0.048 (3)	0.080 (4)	0.012 (3)	−0.026 (4)	−0.014 (3)
C23	0.047 (3)	0.048 (3)	0.070 (4)	−0.003 (3)	−0.005 (3)	−0.006 (3)
C7	0.046 (2)	0.046 (3)	0.033 (2)	0.000 (2)	−0.0050 (18)	0.000 (2)
C15	0.059 (4)	0.096 (5)	0.061 (3)	0.023 (3)	−0.009 (3)	0.004 (3)
C27	0.078 (4)	0.047 (3)	0.066 (3)	0.005 (3)	−0.014 (3)	−0.010 (3)
C1	0.058 (3)	0.067 (3)	0.052 (3)	0.009 (3)	−0.001 (3)	−0.002 (3)
C31	0.067 (4)	0.064 (3)	0.067 (4)	−0.015 (3)	−0.017 (3)	0.008 (3)
C21	0.072 (4)	0.053 (3)	0.072 (4)	0.012 (3)	−0.022 (3)	0.001 (3)
C12	0.061 (3)	0.053 (3)	0.072 (3)	−0.006 (2)	−0.024 (3)	0.007 (3)
C30	0.111 (6)	0.051 (4)	0.101 (5)	−0.030 (4)	−0.060 (5)	0.016 (3)
C14	0.049 (3)	0.067 (4)	0.047 (3)	0.012 (3)	−0.004 (2)	−0.005 (2)
C11	0.080 (4)	0.059 (3)	0.076 (4)	−0.008 (3)	−0.024 (3)	−0.009 (3)
C17	0.052 (3)	0.094 (4)	0.070 (4)	−0.017 (3)	−0.006 (3)	0.009 (3)
C34	0.069 (4)	0.060 (4)	0.085 (4)	0.005 (3)	−0.028 (3)	0.006 (3)
C39	0.065 (4)	0.059 (3)	0.069 (4)	0.000 (3)	0.009 (3)	−0.006 (3)
C22	0.091 (4)	0.059 (4)	0.061 (4)	−0.003 (3)	0.006 (3)	0.013 (3)
C35	0.087 (5)	0.070 (4)	0.105 (5)	0.026 (4)	−0.034 (4)	−0.004 (4)
C9	0.073 (3)	0.061 (3)	0.054 (3)	0.007 (3)	−0.017 (3)	−0.001 (3)
C16	0.044 (3)	0.124 (5)	0.065 (4)	0.013 (4)	−0.001 (2)	0.007 (4)
C41	0.133 (7)	0.056 (4)	0.086 (5)	0.018 (5)	−0.023 (5)	0.016 (3)
C36	0.103 (5)	0.061 (4)	0.088 (5)	−0.002 (4)	0.005 (4)	0.007 (3)

Geometric parameters (Å, °)

O2—C20	1.426 (5)	C19—H19A	0.9700
O2—C19	1.425 (5)	C19—H19B	0.9700
N3—C3	1.374 (5)	C26—C25	1.583 (5)
N3—C19	1.446 (5)	C6—C5	1.583 (5)
N3—C6	1.467 (5)	C5—C7	1.512 (5)
O6—C22	1.418 (6)	C2—H2A	0.9700
O6—C21	1.426 (6)	C2—H2B	0.9700
O4—C3	1.209 (4)	C20—H20A	0.9700
N7—C23	1.399 (6)	C20—H20B	0.9700
N7—C39	1.426 (6)	C38—H38A	0.9700
N7—C26	1.463 (6)	C38—H38B	0.9700
C32—C27	1.373 (7)	C29—C30	1.356 (8)
C32—C31	1.393 (7)	C29—C40	1.377 (9)
C32—C25	1.504 (6)	C29—H29	0.9300
O3—C4	1.226 (5)	C37—C36	1.371 (7)
O5—C38	1.409 (6)	C37—H37	0.9300
O5—C39	1.420 (6)	C18—C17	1.361 (7)
N2—C4	1.373 (5)	C18—H18	0.9300

N2—C2	1.448 (5)	C40—C27	1.385 (7)
N2—C5	1.463 (5)	C40—H40	0.9300
O8—C24	1.217 (5)	C7—C12	1.379 (5)
N5—C24	1.391 (5)	C15—C14	1.364 (7)
N5—C22	1.447 (6)	C15—C16	1.372 (7)
N5—C25	1.454 (6)	C15—H15	0.9300
C4—N4	1.374 (6)	C27—H27	0.9300
O7—C23	1.214 (5)	C1—H1A	0.9700
N1—C3	1.378 (5)	C1—H1B	0.9700
N1—C1	1.439 (5)	C31—C30	1.396 (7)
N1—C5	1.458 (5)	C31—H31	0.9300
O1—C2	1.409 (5)	C21—H21A	0.9700
O1—C1	1.408 (5)	C21—H21B	0.9700
C33—C34	1.380 (6)	C12—C11	1.383 (5)
C33—C37	1.383 (6)	C12—H12	0.9300
C33—C26	1.507 (6)	C30—H30	0.9300
N6—C23	1.373 (6)	C14—H14	0.9300
N6—C21	1.449 (6)	C11—H11	0.9300
N6—C25	1.467 (5)	C17—C16	1.381 (7)
N8—C24	1.375 (6)	C17—H17	0.9300
N8—C38	1.430 (6)	C34—C35	1.379 (8)
N8—C26	1.452 (5)	C34—H34	0.9300
C8—C9	1.377 (5)	C39—H39A	0.9700
C8—C7	1.372 (5)	C39—H39B	0.9700
C8—H8	0.9300	C22—H22A	0.9700
C13—C14	1.387 (6)	C22—H22B	0.9700
C13—C18	1.393 (6)	C35—C41	1.357 (8)
C13—C6	1.500 (6)	C35—H35	0.9300
C10—C11	1.357 (6)	C9—H9	0.9300
C10—C9	1.377 (6)	C16—H16	0.9300
C10—H10	0.9300	C41—C36	1.345 (8)
N4—C20	1.447 (5)	C41—H41	0.9300
N4—C6	1.462 (5)	C36—H36	0.9300
C20—O2—C19	109.7 (3)	N4—C20—H20B	109.3
C3—N3—C19	120.5 (3)	H20A—C20—H20B	108.0
C3—N3—C6	112.0 (3)	O5—C38—N8	110.9 (4)
C19—N3—C6	114.9 (3)	O5—C38—H38A	109.5
C22—O6—C21	109.6 (4)	N8—C38—H38A	109.5
C23—N7—C39	117.4 (4)	O5—C38—H38B	109.5
C23—N7—C26	110.6 (4)	N8—C38—H38B	109.5
C39—N7—C26	114.4 (4)	H38A—C38—H38B	108.0
C27—C32—C31	119.7 (5)	C30—C29—C40	119.9 (6)
C27—C32—C25	120.4 (4)	C30—C29—H29	120.1
C31—C32—C25	119.6 (5)	C40—C29—H29	120.1
C38—O5—C39	109.7 (4)	C36—C37—C33	121.7 (5)
C4—N2—C2	121.0 (4)	C36—C37—H37	119.1
C4—N2—C5	111.6 (3)	C33—C37—H37	119.1
C2—N2—C5	114.9 (3)	C17—C18—C13	120.5 (5)
C24—N5—C22	120.5 (4)	C17—C18—H18	119.7

C24—N5—C25	110.9 (4)	C13—C18—H18	119.7
C22—N5—C25	115.6 (4)	C29—C40—C27	119.9 (6)
O3—C4—N2	126.1 (5)	C29—C40—H40	120.0
O3—C4—N4	125.7 (4)	C27—C40—H40	120.0
N2—C4—N4	108.1 (4)	O7—C23—N6	126.0 (5)
C3—N1—C1	121.3 (3)	O7—C23—N7	126.5 (5)
C3—N1—C5	112.6 (3)	N6—C23—N7	107.3 (4)
C1—N1—C5	115.3 (3)	C8—C7—C12	118.0 (4)
C2—O1—C1	111.1 (3)	C8—C7—C5	122.0 (4)
C34—C33—C37	117.9 (4)	C12—C7—C5	120.0 (4)
C34—C33—C26	120.8 (5)	C14—C15—C16	119.4 (5)
C37—C33—C26	120.9 (4)	C14—C15—H15	120.3
C23—N6—C21	122.6 (4)	C16—C15—H15	120.3
C23—N6—C25	112.0 (4)	C32—C27—C40	120.5 (6)
C21—N6—C25	114.7 (4)	C32—C27—H27	119.7
C24—N8—C38	121.2 (4)	C40—C27—H27	119.7
C24—N8—C26	111.8 (4)	O1—C1—N1	110.6 (4)
C38—N8—C26	116.9 (4)	O1—C1—H1A	109.5
O4—C3—N3	126.6 (4)	N1—C1—H1A	109.5
O4—C3—N1	126.2 (4)	O1—C1—H1B	109.5
N3—C3—N1	107.0 (3)	N1—C1—H1B	109.5
C9—C8—C7	121.0 (4)	H1A—C1—H1B	108.1
C9—C8—H8	119.5	C32—C31—C30	118.7 (6)
C7—C8—H8	119.5	C32—C31—H31	120.7
C14—C13—C18	118.1 (4)	C30—C31—H31	120.6
C14—C13—C6	120.3 (4)	O6—C21—N6	111.4 (4)
C18—C13—C6	121.5 (4)	O6—C21—H21A	109.4
C11—C10—C9	120.1 (4)	N6—C21—H21A	109.3
C11—C10—H10	119.9	O6—C21—H21B	109.4
C9—C10—H10	119.9	N6—C21—H21B	109.4
C4—N4—C20	121.2 (4)	H21A—C21—H21B	108.0
C4—N4—C6	111.6 (3)	C11—C12—C7	121.4 (4)
C20—N4—C6	115.2 (3)	C11—C12—H12	119.3
O2—C19—N3	110.9 (3)	C7—C12—H12	119.3
O2—C19—H19A	109.5	C29—C30—C31	121.3 (6)
N3—C19—H19A	109.5	C29—C30—H30	119.4
O2—C19—H19B	109.5	C31—C30—H30	119.4
N3—C19—H19B	109.5	C15—C14—C13	121.5 (5)
H19A—C19—H19B	108.0	C15—C14—H14	119.3
N8—C26—N7	109.6 (3)	C13—C14—H14	119.3
N8—C26—C33	111.6 (4)	C10—C11—C12	119.6 (4)
N7—C26—C33	112.8 (4)	C10—C11—H11	120.2
N8—C26—C25	103.1 (4)	C12—C11—H11	120.2
N7—C26—C25	102.9 (3)	C18—C17—C16	120.2 (5)
C33—C26—C25	116.0 (4)	C18—C17—H17	119.9
N4—C6—N3	109.1 (3)	C16—C17—H17	119.9
N4—C6—C13	113.2 (3)	C35—C34—C33	120.2 (6)
N3—C6—C13	113.5 (3)	C35—C34—H34	119.9
N4—C6—C5	102.7 (3)	C33—C34—H34	119.9

N3—C6—C5	102.5 (3)	O5—C39—N7	113.1 (4)
C13—C6—C5	114.8 (3)	O5—C39—H39A	108.9
N1—C5—N2	108.6 (3)	N7—C39—H39A	108.9
N1—C5—C7	112.2 (3)	O5—C39—H39B	109.0
N2—C5—C7	112.9 (3)	N7—C39—H39B	109.0
N1—C5—C6	102.3 (3)	H39A—C39—H39B	107.8
N2—C5—C6	102.6 (3)	O6—C22—N5	110.3 (4)
C7—C5—C6	117.2 (3)	O6—C22—H22A	109.6
N5—C25—N6	109.0 (3)	N5—C22—H22A	109.6
N5—C25—C32	113.3 (4)	O6—C22—H22B	109.6
N6—C25—C32	112.3 (4)	N5—C22—H22B	109.6
N5—C25—C26	103.0 (3)	H22A—C22—H22B	108.1
N6—C25—C26	102.6 (3)	C41—C35—C34	119.5 (6)
C32—C25—C26	115.7 (4)	C41—C35—H35	120.2
O8—C24—N8	126.7 (5)	C34—C35—H35	120.2
O8—C24—N5	124.9 (5)	C8—C9—C10	119.9 (4)
N8—C24—N5	108.2 (4)	C8—C9—H9	120.1
O1—C2—N2	110.9 (4)	C10—C9—H9	120.1
O1—C2—H2A	109.5	C15—C16—C17	120.3 (5)
N2—C2—H2A	109.5	C15—C16—H16	119.9
O1—C2—H2B	109.5	C17—C16—H16	119.9
N2—C2—H2B	109.5	C36—C41—C35	122.1 (6)
H2A—C2—H2B	108.0	C36—C41—H41	119.0
O2—C20—N4	111.5 (3)	C35—C41—H41	119.0
O2—C20—H20A	109.3	C41—C36—C37	118.5 (6)
N4—C20—H20A	109.3	C41—C36—H36	120.7
O2—C20—H20B	109.3	C37—C36—H36	120.7
C2—N2—C4—O3	24.0 (6)	C31—C32—C25—N5	158.4 (4)
C5—N2—C4—O3	164.0 (4)	C27—C32—C25—N6	−151.2 (4)
C2—N2—C4—N4	−158.9 (4)	C31—C32—C25—N6	34.4 (6)
C5—N2—C4—N4	−18.9 (4)	C27—C32—C25—C26	91.4 (5)
C19—N3—C3—O4	26.4 (6)	C31—C32—C25—C26	−83.0 (5)
C6—N3—C3—O4	166.1 (4)	N8—C26—C25—N5	−4.0 (4)
C19—N3—C3—N1	−159.5 (3)	N7—C26—C25—N5	−118.0 (3)
C6—N3—C3—N1	−19.7 (4)	C33—C26—C25—N5	118.3 (4)
C1—N1—C3—O4	−24.4 (6)	N8—C26—C25—N6	109.2 (3)
C5—N1—C3—O4	−167.0 (4)	N7—C26—C25—N6	−4.8 (4)
C1—N1—C3—N3	161.4 (4)	C33—C26—C25—N6	−128.5 (4)
C5—N1—C3—N3	18.8 (4)	N8—C26—C25—C32	−128.1 (4)
O3—C4—N4—C20	−23.6 (7)	N7—C26—C25—C32	117.9 (4)
N2—C4—N4—C20	159.2 (3)	C33—C26—C25—C32	−5.8 (5)
O3—C4—N4—C6	−164.5 (4)	C38—N8—C24—O8	−23.3 (7)
N2—C4—N4—C6	18.3 (4)	C26—N8—C24—O8	−167.7 (4)
C20—O2—C19—N3	59.5 (5)	C38—N8—C24—N5	160.0 (4)
C3—N3—C19—O2	83.4 (5)	C26—N8—C24—N5	15.6 (5)
C6—N3—C19—O2	−55.3 (5)	C22—N5—C24—O8	25.2 (7)
C24—N8—C26—N7	102.2 (4)	C25—N5—C24—O8	164.8 (4)
C38—N8—C26—N7	−43.8 (5)	C22—N5—C24—N8	−158.1 (4)
C24—N8—C26—C33	−132.0 (4)	C25—N5—C24—N8	−18.5 (5)

C38—N8—C26—C33	82.0 (5)	C1—O1—C2—N2	58.4 (5)
C24—N8—C26—C25	−6.8 (5)	C4—N2—C2—O1	85.2 (5)
C38—N8—C26—C25	−152.8 (4)	C5—N2—C2—O1	−53.6 (4)
C23—N7—C26—N8	−92.6 (4)	C19—O2—C20—N4	−58.6 (5)
C39—N7—C26—N8	42.7 (5)	C4—N4—C20—O2	−86.3 (5)
C23—N7—C26—C33	142.3 (4)	C6—N4—C20—O2	53.3 (5)
C39—N7—C26—C33	−82.5 (5)	C39—O5—C38—N8	−57.4 (5)
C23—N7—C26—C25	16.6 (4)	C24—N8—C38—O5	−90.0 (6)
C39—N7—C26—C25	151.8 (4)	C26—N8—C38—O5	52.7 (6)
C34—C33—C26—N8	28.2 (6)	C34—C33—C37—C36	−1.7 (7)
C37—C33—C26—N8	−158.7 (4)	C26—C33—C37—C36	−174.9 (5)
C34—C33—C26—N7	152.3 (4)	C14—C13—C18—C17	2.1 (6)
C37—C33—C26—N7	−34.7 (6)	C6—C13—C18—C17	−173.3 (4)
C34—C33—C26—C25	−89.4 (5)	C30—C29—C40—C27	−0.6 (9)
C37—C33—C26—C25	83.6 (5)	C21—N6—C23—O7	−22.5 (7)
C4—N4—C6—N3	97.8 (4)	C25—N6—C23—O7	−165.0 (5)
C20—N4—C6—N3	−45.6 (4)	C21—N6—C23—N7	161.8 (4)
C4—N4—C6—C13	−134.8 (4)	C25—N6—C23—N7	19.2 (5)
C20—N4—C6—C13	81.8 (4)	C39—N7—C23—O7	27.9 (7)
C4—N4—C6—C5	−10.4 (4)	C26—N7—C23—O7	161.6 (5)
C20—N4—C6—C5	−153.8 (3)	C39—N7—C23—N6	−156.4 (4)
C3—N3—C6—N4	−95.5 (4)	C26—N7—C23—N6	−22.6 (5)
C19—N3—C6—N4	46.6 (4)	C9—C8—C7—C12	−0.6 (6)
C3—N3—C6—C13	137.2 (3)	C9—C8—C7—C5	−178.8 (4)
C19—N3—C6—C13	−80.6 (4)	N1—C5—C7—C8	−151.5 (4)
C3—N3—C6—C5	12.8 (4)	N2—C5—C7—C8	−28.4 (5)
C19—N3—C6—C5	155.0 (3)	C6—C5—C7—C8	90.6 (5)
C14—C13—C6—N4	34.0 (5)	N1—C5—C7—C12	30.2 (5)
C18—C13—C6—N4	−150.7 (4)	N2—C5—C7—C12	153.4 (4)
C14—C13—C6—N3	159.1 (4)	C6—C5—C7—C12	−87.7 (5)
C18—C13—C6—N3	−25.6 (5)	C31—C32—C27—C40	1.0 (7)
C14—C13—C6—C5	−83.5 (5)	C25—C32—C27—C40	−173.5 (5)
C18—C13—C6—C5	91.8 (5)	C29—C40—C27—C32	−0.6 (8)
C3—N1—C5—N2	97.7 (4)	C2—O1—C1—N1	−58.8 (5)
C1—N1—C5—N2	−47.3 (4)	C3—N1—C1—O1	−87.0 (5)
C3—N1—C5—C7	−136.8 (3)	C5—N1—C1—O1	54.6 (4)
C1—N1—C5—C7	78.3 (4)	C27—C32—C31—C30	−0.1 (7)
C3—N1—C5—C6	−10.4 (4)	C25—C32—C31—C30	174.3 (4)
C1—N1—C5—C6	−155.3 (3)	C22—O6—C21—N6	−59.5 (5)
C4—N2—C5—N1	−96.0 (4)	C23—N6—C21—O6	−88.0 (5)
C2—N2—C5—N1	46.6 (4)	C25—N6—C21—O6	53.6 (5)
C4—N2—C5—C7	138.9 (3)	C8—C7—C12—C11	0.3 (7)
C2—N2—C5—C7	−78.5 (4)	C5—C7—C12—C11	178.6 (4)
C4—N2—C5—C6	11.8 (4)	C40—C29—C30—C31	1.4 (9)
C2—N2—C5—C6	154.4 (3)	C32—C31—C30—C29	−1.1 (8)
N4—C6—C5—N1	111.8 (3)	C16—C15—C14—C13	1.0 (7)
N3—C6—C5—N1	−1.4 (3)	C18—C13—C14—C15	−1.5 (6)
C13—C6—C5—N1	−124.9 (3)	C6—C13—C14—C15	174.0 (4)
N4—C6—C5—N2	−0.8 (3)	C9—C10—C11—C12	0.0 (8)

N3—C6—C5—N2	−114.0 (3)	C7—C12—C11—C10	0.0 (8)
C13—C6—C5—N2	122.5 (3)	C13—C18—C17—C16	−2.3 (7)
N4—C6—C5—C7	−125.1 (3)	C37—C33—C34—C35	0.3 (7)
N3—C6—C5—C7	121.7 (3)	C26—C33—C34—C35	173.5 (5)
C13—C6—C5—C7	−1.8 (5)	C38—O5—C39—N7	58.8 (5)
C24—N5—C25—N6	−94.9 (4)	C23—N7—C39—O5	79.7 (5)
C22—N5—C25—N6	46.8 (5)	C26—N7—C39—O5	−52.4 (5)
C24—N5—C25—C32	139.2 (4)	C21—O6—C22—N5	59.7 (5)
C22—N5—C25—C32	−79.1 (5)	C24—N5—C22—O6	82.3 (5)
C24—N5—C25—C26	13.5 (4)	C25—N5—C22—O6	−55.5 (5)
C22—N5—C25—C26	155.2 (4)	C33—C34—C35—C41	0.2 (9)
C23—N6—C25—N5	100.2 (4)	C7—C8—C9—C10	0.5 (7)
C21—N6—C25—N5	−45.5 (5)	C11—C10—C9—C8	−0.3 (8)
C23—N6—C25—C32	−133.4 (4)	C14—C15—C16—C17	−1.2 (7)
C21—N6—C25—C32	81.0 (5)	C18—C17—C16—C15	1.8 (7)
C23—N6—C25—C26	−8.5 (5)	C34—C35—C41—C36	0.7 (10)
C21—N6—C25—C26	−154.1 (4)	C35—C41—C36—C37	−2.1 (10)
C27—C32—C25—N5	−27.1 (6)	C33—C37—C36—C41	2.6 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O8^{i}	0.97	2.40	3.188 (5)	138
C27—H27···O1^{ii}	0.93	2.30	3.208 (7)	166

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

Fig. 1

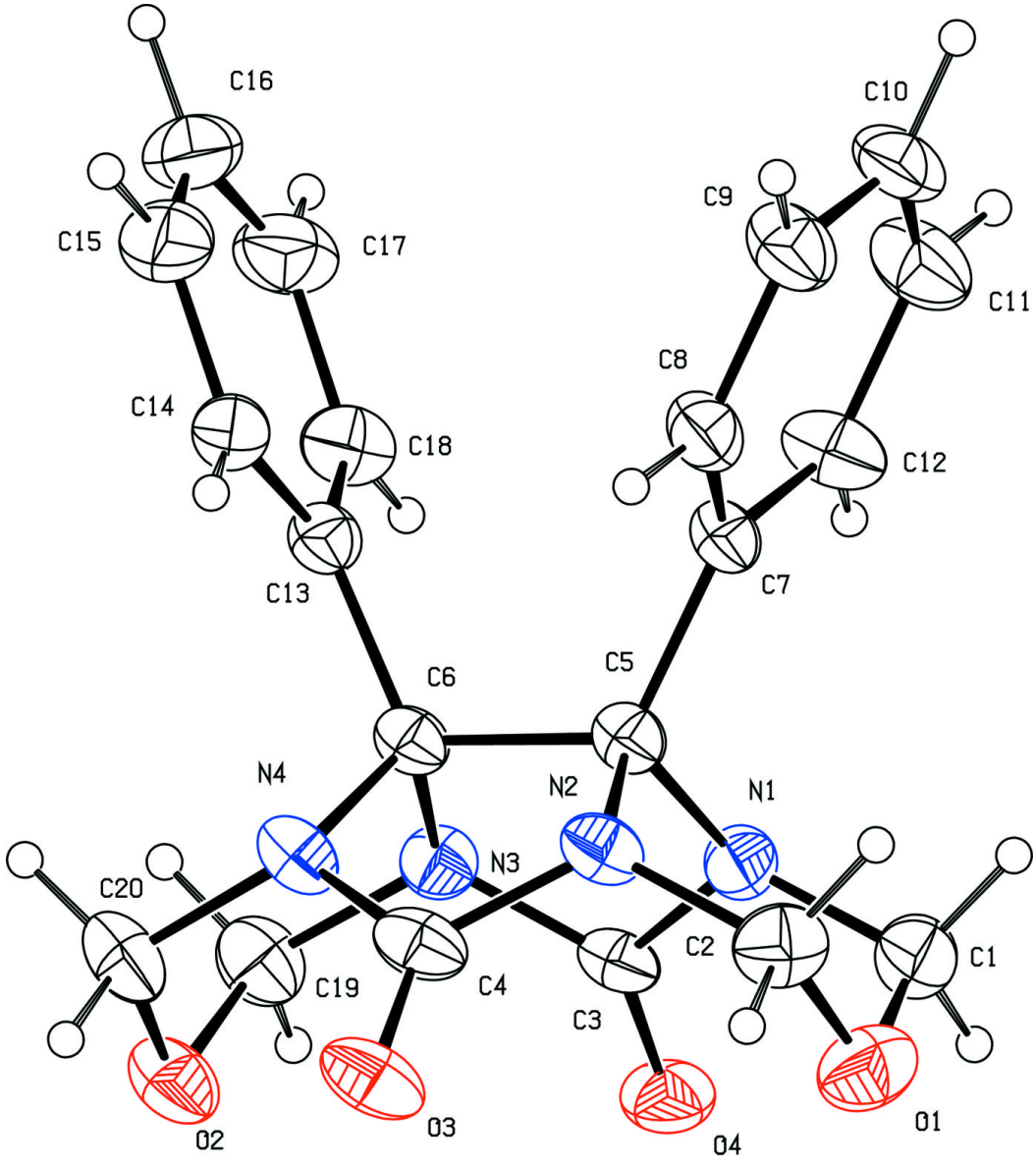
C16
C10
C9
C15
C17
C11
C8
C14
C18
C12
C13
C7
C6
C5
N4
N2
N1
N3
C20
C2
C1
C19
C4
C3
O2
O3
O4
O1

Fig. 2