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Diethyl 2,6-bis(3-methylphenyl)-4,8-dioxo-2,6,3a,4a,7a,8a-hexaazaperhydrocyclopenta[*def*]fluorene-8b,8c-dicarboxylate

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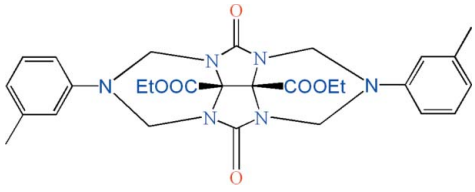
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.057; wR factor = 0.136; data-to-parameter ratio = 15.9.

In the title compound, $C_{28}H_{32}N_6O_6$, the dihedral angles between the two fused five-membered rings in the glycoluril unit and between the two terminal benzene rings are 71.78 (2) and 66.76 (2)°, respectively. The molecules are connected mainly by van der Waals forces.

Related literature

For the preparation of the title compound, see: Yin *et al.* (2006). For the structure of cucurbit[6]uril, see Freeman *et al.* (1981). For literature on clip molecules based on glycoluril, see Chen *et al.* (2007); Hu *et al.* (2007); Li *et al.* (2006); Rowan *et al.* (1999); She *et al.* (2007); Wang *et al.* (2006).



Experimental

Crystal data

$C_{28}H_{32}N_6O_6$
M_r = 548.60
Orthorhombic, *Pbca*
a = 17.8591 (13) Å
b = 15.4533 (11) Å
c = 19.5649 (14) Å
V = 5399.6 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
30333 measured reflections
5874 independent reflections
4406 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.136
S = 1.05
5874 reflections
370 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2358).

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supplementary materials

Acta Cryst. (2007). E**63**, o4577 [**doi:10.1107/S1600536807054803**]

Diethyl 2,6-bis(3-methylphenyl)-4,8-dioxo-2,6,3a,4a,7a,8a-hexaazaperhydrocyclopenta[*def*]fluorene-8b,8c-dicarboxylate

G.-D. Yin, Z.-H. Wang and J. Qin

Comment

Since Mock and co-workers first characterized the chemical nature and structure of cucurbit[6]uril (Freeman *et al.*, 1981), many clip molecules based on glycoluril as supramolecular receptor have been reported (Rowan *et al.*, 1999; Li *et al.*, 2006; Wang *et al.*, 2006; Chen *et al.*, 2007; Hu *et al.*, 2007; She *et al.*, 2007). As a continuation of our previous studies in this area, we herein report the crystal structure of the title compound, (I), a new type of receptor based on glycoluril (Fig. 1). Selected bond lengths and angles are listed in Table 1. Two six-membered rings (N1—C8—N2 –C12—N3—C9 and N4—C16—N5—C21—N6—C20) both adopt the chair conformation. The dihedral angle between the two fused five-membered rings in the glycoluril unit is 71.78 (2) °. The crystal packing is mainly governed by van der Waals forces.

Experimental

The title compound was synthesized according to the reported literature (Yin *et al.*, 2006). Crystals of (I) suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane-methanol (4:1) solution of the title compound under ambient conditions.

Refinement

One of the ethyl groups (C15) was found to be disordered over two orientations. The occupancies of the disordered positions C15/C15' refined to 0.64 (2):0.36 (2). All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, allowing for free rotation of the methyl groups. The constraint U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) (methyl C) was applied.

Figures

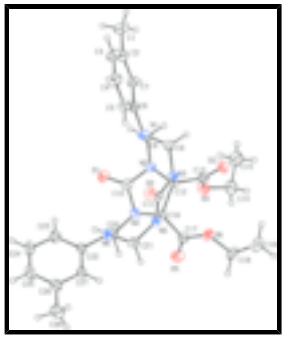


Fig. 1. A view of (I), showing the atom-labelling scheme, with displacement ellipsoids drawn at the 30% probability level.

Diethyl 2,6-bis(3-methylphenyl)-4,8-dioxo-2,6,3a,4a,7a,8a- hexaazaperhydrocyclopenta[*def*]fluorene-8 b,8c-dicarboxylate

Crystal data

$C_{28}H_{32}N_6O_6$	F_{000} = 2320
M_r = 548.60	D_x = 1.350 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P2ac2ab	Cell parameters from 4426 reflections
a = 17.8591 (13) Å	θ = 2.6–21.8º
b = 15.4533 (11) Å	μ = 0.10 mm^{-1}
c = 19.5649 (14) Å	*T* = 292 (2) K
V = 5399.6 (7) Å^3	Block, colorless
Z = 8	0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer	4406 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.050
Monochromator: graphite	θ_{max} = 27.0º
T = 292(2) K	θ_{min} = 2.0º
φ and ω scans	*h* = −22→22
Absorption correction: None	*k* = −19→19
30333 measured reflections	*l* = −16→24
5874 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.136	$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 2.134P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
5874 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
370 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
C1	0.13577 (17)	0.19168 (16)	0.46245 (14)	0.0655 (8)	
H1A	0.1142	0.1880	0.5073	0.098*	
H1B	0.1873	0.1741	0.4643	0.098*	
H1C	0.1089	0.1544	0.4318	0.098*	
C2	0.13103 (13)	0.28371 (14)	0.43719 (11)	0.0462 (6)	
C3	0.07028 (14)	0.33474 (17)	0.45309 (14)	0.0568 (7)	
H3	0.0317	0.3121	0.4796	0.068*	
C4	0.06592 (14)	0.41882 (17)	0.43031 (14)	0.0579 (7)	
H4	0.0245	0.4524	0.4415	0.069*	
C5	0.12232 (12)	0.45357 (15)	0.39119 (12)	0.0465 (6)	
H5	0.1188	0.5104	0.3758	0.056*	
C6	0.18480 (11)	0.40391 (13)	0.37455 (10)	0.0360 (5)	
C7	0.18868 (12)	0.31908 (14)	0.39802 (11)	0.0406 (5)	
H7	0.2303	0.2855	0.3874	0.049*	
C8	0.29814 (12)	0.38598 (13)	0.30343 (11)	0.0378 (5)	
H8A	0.3318	0.3658	0.3389	0.045*	
H8B	0.2745	0.3360	0.2826	0.045*	
C9	0.27758 (12)	0.51778 (13)	0.36519 (10)	0.0372 (5)	
H9A	0.2400	0.5548	0.3857	0.045*	
H9B	0.3110	0.4982	0.4010	0.045*	
C10	0.31559 (11)	0.43743 (13)	0.18532 (11)	0.0349 (5)	
C11	0.28227 (11)	0.62743 (12)	0.27600 (10)	0.0314 (4)	
C12	0.37140 (11)	0.51700 (12)	0.27303 (10)	0.0322 (4)	
C13	0.44683 (11)	0.50352 (14)	0.30994 (11)	0.0386 (5)	
C14	0.57290 (13)	0.45747 (18)	0.29232 (14)	0.0607 (7)	
H14A	0.5853	0.5060	0.3217	0.073*	0.64 (2)
H14B	0.6069	0.4583	0.2537	0.073*	0.64 (2)
H14C	0.6071	0.4865	0.2614	0.073*	0.36 (2)
H14D	0.5781	0.4834	0.3372	0.073*	0.36 (2)
C15	0.5831 (5)	0.3757 (6)	0.3308 (8)	0.081 (3)	0.64 (2)
H15A	0.5519	0.3763	0.3707	0.122*	0.64 (2)
H15B	0.6346	0.3702	0.3443	0.122*	0.64 (2)
H15C	0.5695	0.3277	0.3023	0.122*	0.64 (2)

C15'	0.5909 (11)	0.3650 (8)	0.2959 (12)	0.081 (3)	0.36 (2)
H15D	0.5555	0.3364	0.3252	0.122*	0.36 (2)
H15E	0.6404	0.3577	0.3140	0.122*	0.36 (2)
H15F	0.5884	0.3403	0.2510	0.122*	0.36 (2)
C16	0.37500 (11)	0.56958 (12)	0.20477 (10)	0.0319 (4)	
C17	0.45154 (11)	0.60894 (14)	0.18663 (11)	0.0385 (5)	
C18	0.54944 (13)	0.69342 (17)	0.23643 (15)	0.0600 (7)	
H18A	0.5502	0.7369	0.2006	0.072*	
H18B	0.5875	0.6505	0.2265	0.072*	
C19	0.56359 (17)	0.7339 (2)	0.30383 (16)	0.0811 (10)	
H19A	0.5264	0.7774	0.3124	0.122*	
H19B	0.6124	0.7600	0.3039	0.122*	
H19C	0.5611	0.6905	0.3389	0.122*	
C20	0.31026 (12)	0.54585 (14)	0.09478 (10)	0.0404 (5)	
H20A	0.2874	0.5001	0.0681	0.048*	
H20B	0.3463	0.5756	0.0661	0.048*	
C21	0.28691 (12)	0.67668 (12)	0.15651 (10)	0.0361 (5)	
H21A	0.3239	0.7061	0.1286	0.043*	
H21B	0.2491	0.7184	0.1700	0.043*	
C22	0.19878 (11)	0.63132 (14)	0.06778 (10)	0.0365 (5)	
C23	0.15080 (13)	0.56850 (16)	0.04217 (11)	0.0483 (6)	
H23	0.1561	0.5111	0.0555	0.058*	
C24	0.09497 (14)	0.59180 (19)	−0.00343 (13)	0.0583 (7)	
H24	0.0621	0.5500	−0.0199	0.070*	
C25	0.08790 (13)	0.67635 (19)	−0.02449 (12)	0.0563 (7)	
H25	0.0507	0.6909	−0.0557	0.068*	
C26	0.13505 (12)	0.73984 (17)	−0.00012 (12)	0.0483 (6)	
C27	0.19051 (12)	0.71622 (14)	0.04632 (11)	0.0406 (5)	
H27	0.2227	0.7584	0.0633	0.049*	
C28	0.12859 (16)	0.8322 (2)	−0.02293 (17)	0.0790 (9)	
H28A	0.1743	0.8496	−0.0448	0.118*	
H28B	0.1195	0.8685	0.0160	0.118*	
H28C	0.0878	0.8376	−0.0546	0.118*	
N1	0.24122 (9)	0.44287 (10)	0.33290 (9)	0.0364 (4)	
N2	0.33984 (9)	0.43436 (10)	0.25214 (8)	0.0325 (4)	
N3	0.31940 (9)	0.56596 (10)	0.31470 (8)	0.0320 (4)	
N4	0.34792 (9)	0.50907 (10)	0.15396 (8)	0.0343 (4)	
N5	0.32233 (9)	0.63989 (10)	0.21670 (8)	0.0306 (4)	
N6	0.25262 (10)	0.60692 (10)	0.11782 (8)	0.0364 (4)	
O1	0.27578 (9)	0.38472 (9)	0.15775 (8)	0.0469 (4)	
O2	0.22632 (8)	0.66624 (9)	0.29289 (8)	0.0422 (4)	
O3	0.45827 (9)	0.52344 (12)	0.36771 (8)	0.0553 (4)	
O4	0.49553 (8)	0.46721 (11)	0.26752 (8)	0.0544 (4)	
O5	0.48131 (9)	0.60350 (13)	0.13262 (9)	0.0622 (5)	
O6	0.47578 (8)	0.65276 (10)	0.24036 (8)	0.0481 (4)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.079 (2)	0.0484 (14)	0.0691 (18)	−0.0121 (13)	0.0201 (15)	0.0033 (13)
C2	0.0509 (14)	0.0436 (12)	0.0441 (13)	−0.0091 (11)	0.0080 (11)	−0.0035 (10)
C3	0.0470 (14)	0.0571 (15)	0.0664 (17)	−0.0101 (12)	0.0222 (12)	−0.0003 (13)
C4	0.0407 (13)	0.0597 (16)	0.0733 (18)	0.0061 (12)	0.0193 (13)	−0.0025 (13)
C5	0.0411 (13)	0.0434 (12)	0.0550 (14)	0.0039 (10)	0.0081 (11)	0.0017 (11)
C6	0.0352 (11)	0.0377 (11)	0.0350 (11)	−0.0029 (9)	0.0031 (9)	−0.0022 (9)
C7	0.0400 (12)	0.0414 (12)	0.0405 (12)	−0.0012 (10)	0.0054 (10)	−0.0034 (10)
C8	0.0405 (12)	0.0324 (10)	0.0405 (12)	0.0018 (9)	0.0068 (9)	0.0019 (9)
C9	0.0391 (12)	0.0369 (11)	0.0355 (11)	−0.0003 (9)	0.0057 (9)	−0.0014 (9)
C10	0.0323 (11)	0.0315 (10)	0.0409 (11)	0.0071 (9)	0.0044 (9)	−0.0050 (9)
C11	0.0277 (10)	0.0282 (9)	0.0384 (11)	−0.0023 (8)	−0.0018 (8)	−0.0061 (8)
C12	0.0309 (10)	0.0316 (10)	0.0342 (10)	0.0021 (8)	0.0019 (8)	−0.0002 (8)
C13	0.0335 (11)	0.0417 (12)	0.0406 (12)	0.0010 (9)	−0.0009 (9)	0.0101 (10)
C14	0.0326 (13)	0.0772 (18)	0.0723 (18)	0.0126 (12)	−0.0030 (12)	0.0087 (15)
C15	0.049 (3)	0.071 (3)	0.124 (8)	0.005 (2)	−0.023 (5)	0.025 (5)
C15'	0.049 (3)	0.071 (3)	0.124 (8)	0.005 (2)	−0.023 (5)	0.025 (5)
C16	0.0287 (10)	0.0337 (10)	0.0333 (10)	0.0029 (8)	0.0017 (8)	0.0002 (8)
C17	0.0297 (11)	0.0422 (12)	0.0435 (12)	0.0025 (9)	0.0025 (9)	0.0089 (10)
C18	0.0301 (12)	0.0647 (16)	0.085 (2)	−0.0147 (11)	−0.0006 (12)	0.0153 (15)
C19	0.0648 (19)	0.083 (2)	0.095 (2)	−0.0328 (17)	−0.0254 (17)	0.0057 (18)
C20	0.0481 (13)	0.0387 (11)	0.0343 (11)	0.0038 (10)	−0.0025 (10)	−0.0021 (9)
C21	0.0410 (12)	0.0308 (10)	0.0366 (11)	0.0016 (9)	−0.0065 (9)	0.0013 (9)
C22	0.0347 (11)	0.0458 (12)	0.0290 (10)	0.0006 (9)	0.0001 (9)	−0.0021 (9)
C23	0.0498 (14)	0.0517 (13)	0.0433 (13)	−0.0055 (11)	−0.0019 (11)	−0.0088 (11)
C24	0.0467 (14)	0.0812 (19)	0.0470 (14)	−0.0108 (13)	−0.0086 (11)	−0.0224 (14)
C25	0.0401 (13)	0.089 (2)	0.0400 (13)	0.0070 (13)	−0.0089 (10)	−0.0029 (13)
C26	0.0364 (12)	0.0676 (16)	0.0410 (12)	0.0061 (11)	0.0006 (10)	0.0076 (11)
C27	0.0340 (11)	0.0479 (12)	0.0398 (12)	0.0005 (10)	−0.0027 (9)	0.0037 (10)
C28	0.0587 (18)	0.088 (2)	0.091 (2)	0.0069 (16)	−0.0165 (16)	0.0415 (18)
N1	0.0352 (9)	0.0334 (9)	0.0407 (10)	0.0001 (7)	0.0089 (8)	−0.0012 (8)
N2	0.0324 (9)	0.0301 (8)	0.0349 (9)	0.0022 (7)	0.0049 (7)	−0.0001 (7)
N3	0.0303 (9)	0.0325 (8)	0.0332 (9)	0.0010 (7)	0.0027 (7)	0.0006 (7)
N4	0.0381 (9)	0.0319 (8)	0.0330 (9)	0.0031 (7)	0.0011 (7)	−0.0012 (7)
N5	0.0295 (8)	0.0296 (8)	0.0328 (9)	0.0012 (7)	−0.0029 (7)	−0.0016 (7)
N6	0.0422 (10)	0.0325 (9)	0.0344 (9)	0.0030 (7)	−0.0079 (8)	−0.0035 (7)
O1	0.0533 (10)	0.0368 (8)	0.0507 (9)	−0.0042 (7)	−0.0017 (8)	−0.0092 (7)
O2	0.0359 (8)	0.0403 (8)	0.0505 (9)	0.0094 (7)	0.0026 (7)	−0.0070 (7)
O3	0.0412 (9)	0.0844 (12)	0.0402 (9)	−0.0005 (8)	−0.0064 (7)	0.0054 (9)
O4	0.0344 (8)	0.0740 (11)	0.0546 (10)	0.0209 (8)	−0.0046 (7)	−0.0056 (9)
O5	0.0478 (10)	0.0900 (14)	0.0487 (10)	−0.0068 (9)	0.0168 (8)	0.0049 (9)
O6	0.0317 (8)	0.0581 (10)	0.0544 (10)	−0.0146 (7)	0.0027 (7)	−0.0002 (8)

Geometric parameters (Å, °)

C1—C2	1.508 (3)	C15—H15A	0.9600

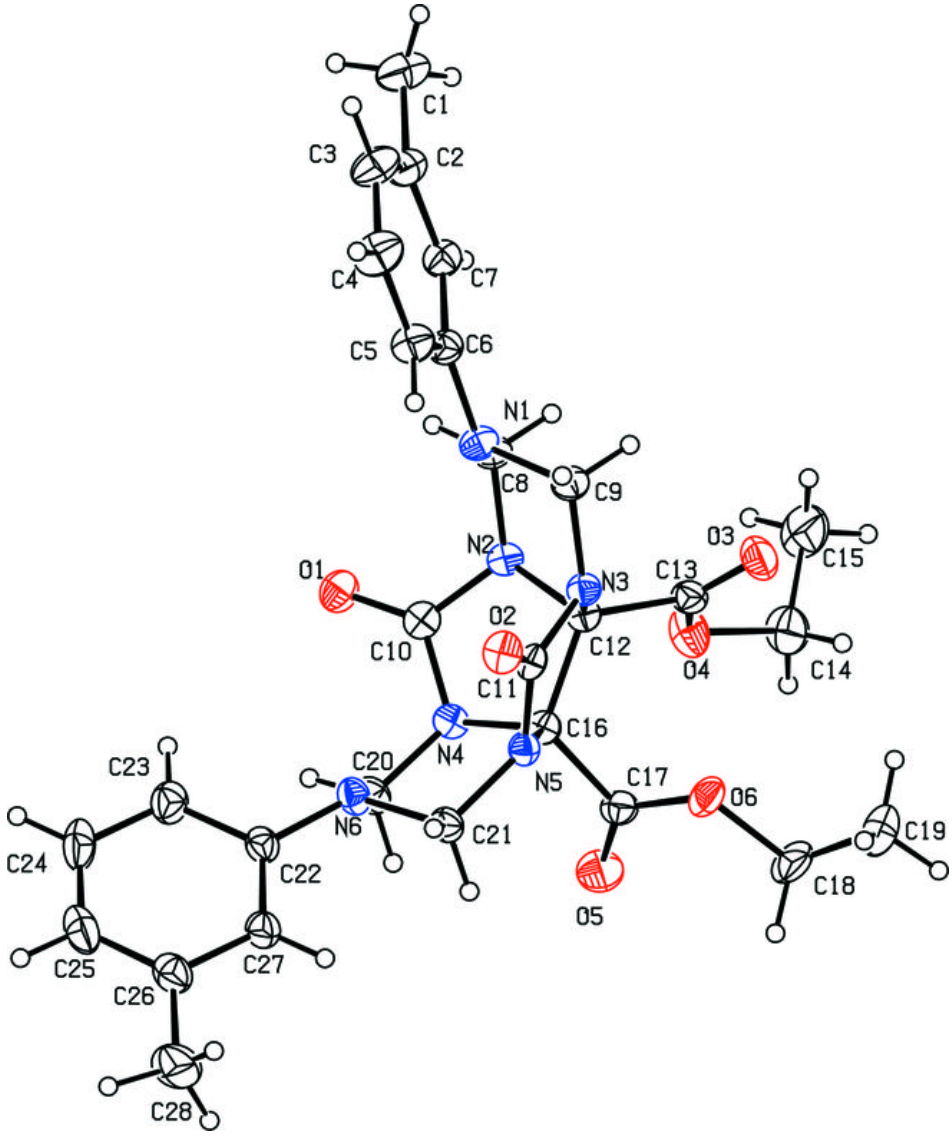
C1—H1A	0.9600	C15—H15B	0.9600
C1—H1B	0.9600	C15—H15C	0.9600
C1—H1C	0.9600	C15'—H15D	0.9600
C2—C3	1.377 (3)	C15'—H15E	0.9600
C2—C7	1.395 (3)	C15'—H15F	0.9600
C3—C4	1.376 (3)	C16—N4	1.448 (2)
C3—H3	0.9300	C16—N5	1.456 (2)
C4—C5	1.374 (3)	C16—C17	1.538 (3)
C4—H4	0.9300	C17—O5	1.186 (3)
C5—C6	1.393 (3)	C17—O6	1.323 (3)
C5—H5	0.9300	C18—O6	1.460 (3)
C6—C7	1.391 (3)	C18—C19	1.481 (4)
C6—N1	1.429 (2)	C18—H18A	0.9700
C7—H7	0.9300	C18—H18B	0.9700
C8—N2	1.456 (2)	C19—H19A	0.9600
C8—N1	1.462 (2)	C19—H19B	0.9600
C8—H8A	0.9700	C19—H19C	0.9600
C8—H8B	0.9700	C20—N4	1.455 (3)
C9—N3	1.445 (2)	C20—N6	1.468 (3)
C9—N1	1.470 (2)	C20—H20A	0.9700
C9—H9A	0.9700	C20—H20B	0.9700
C9—H9B	0.9700	C21—N5	1.453 (2)
C10—O1	1.208 (2)	C21—N6	1.453 (2)
C10—N2	1.378 (3)	C21—H21A	0.9700
C10—N4	1.391 (3)	C21—H21B	0.9700
C11—O2	1.211 (2)	C22—C27	1.385 (3)
C11—N5	1.377 (2)	C22—C23	1.388 (3)
C11—N3	1.384 (2)	C22—N6	1.423 (3)
C12—N3	1.449 (2)	C23—C24	1.386 (3)
C12—N2	1.455 (2)	C23—H23	0.9300
C12—C13	1.543 (3)	C24—C25	1.376 (4)
C12—C16	1.565 (3)	C24—H24	0.9300
C13—O3	1.189 (3)	C25—C26	1.378 (3)
C13—O4	1.327 (3)	C25—H25	0.9300
C14—C15'	1.466 (12)	C26—C27	1.393 (3)
C14—O4	1.472 (3)	C26—C28	1.500 (4)
C14—C15	1.481 (7)	C27—H27	0.9300
C14—H14A	0.9700	C28—H28A	0.9600
C14—H14B	0.9700	C28—H28B	0.9600
C14—H14C	0.9700	C28—H28C	0.9600
C14—H14D	0.9700		
C2—C1—H1A	109.5	N4—C16—N5	112.10 (15)
C2—C1—H1B	109.5	N4—C16—C17	113.20 (16)
H1A—C1—H1B	109.5	N5—C16—C17	108.43 (15)
C2—C1—H1C	109.5	N4—C16—C12	103.70 (15)
H1A—C1—H1C	109.5	N5—C16—C12	102.95 (14)
H1B—C1—H1C	109.5	C17—C16—C12	116.04 (16)
C3—C2—C7	118.8 (2)	O5—C17—O6	126.7 (2)
C3—C2—C1	120.7 (2)	O5—C17—C16	125.2 (2)

C7—C2—C1	120.5 (2)	O6—C17—C16	108.05 (17)
C4—C3—C2	120.8 (2)	O6—C18—C19	106.8 (2)
C4—C3—H3	119.6	O6—C18—H18A	110.4
C2—C3—H3	119.6	C19—C18—H18A	110.4
C5—C4—C3	120.5 (2)	O6—C18—H18B	110.4
C5—C4—H4	119.7	C19—C18—H18B	110.4
C3—C4—H4	119.7	H18A—C18—H18B	108.6
C4—C5—C6	120.1 (2)	C18—C19—H19A	109.5
C4—C5—H5	119.9	C18—C19—H19B	109.5
C6—C5—H5	119.9	H19A—C19—H19B	109.5
C7—C6—C5	118.83 (19)	C18—C19—H19C	109.5
C7—C6—N1	123.38 (18)	H19A—C19—H19C	109.5
C5—C6—N1	117.78 (18)	H19B—C19—H19C	109.5
C6—C7—C2	120.9 (2)	N4—C20—N6	109.33 (16)
C6—C7—H7	119.5	N4—C20—H20A	109.8
C2—C7—H7	119.5	N6—C20—H20A	109.8
N2—C8—N1	108.58 (15)	N4—C20—H20B	109.8
N2—C8—H8A	110.0	N6—C20—H20B	109.8
N1—C8—H8A	110.0	H20A—C20—H20B	108.3
N2—C8—H8B	110.0	N5—C21—N6	108.40 (15)
N1—C8—H8B	110.0	N5—C21—H21A	110.0
H8A—C8—H8B	108.4	N6—C21—H21A	110.0
N3—C9—N1	109.90 (16)	N5—C21—H21B	110.0
N3—C9—H9A	109.7	N6—C21—H21B	110.0
N1—C9—H9A	109.7	H21A—C21—H21B	108.4
N3—C9—H9B	109.7	C27—C22—C23	119.1 (2)
N1—C9—H9B	109.7	C27—C22—N6	122.10 (19)
H9A—C9—H9B	108.2	C23—C22—N6	118.69 (19)
O1—C10—N2	125.82 (19)	C24—C23—C22	119.7 (2)
O1—C10—N4	125.72 (19)	C24—C23—H23	120.2
N2—C10—N4	108.38 (17)	C22—C23—H23	120.2
O2—C11—N5	126.11 (18)	C25—C24—C23	120.4 (2)
O2—C11—N3	125.87 (19)	C25—C24—H24	119.8
N5—C11—N3	107.94 (16)	C23—C24—H24	119.8
N3—C12—N2	111.61 (15)	C24—C25—C26	121.1 (2)
N3—C12—C13	111.51 (16)	C24—C25—H25	119.5
N2—C12—C13	110.57 (16)	C26—C25—H25	119.5
N3—C12—C16	103.62 (14)	C25—C26—C27	118.3 (2)
N2—C12—C16	103.42 (15)	C25—C26—C28	121.8 (2)
C13—C12—C16	115.71 (16)	C27—C26—C28	119.9 (2)
O3—C13—O4	126.3 (2)	C22—C27—C26	121.4 (2)
O3—C13—C12	124.1 (2)	C22—C27—H27	119.3
O4—C13—C12	109.68 (18)	C26—C27—H27	119.3
C15'—C14—O4	108.7 (8)	C26—C28—H28A	109.5
O4—C14—C15	111.7 (4)	C26—C28—H28B	109.5
C15'—C14—H14A	132.4	H28A—C28—H28B	109.5
O4—C14—H14A	109.3	C26—C28—H28C	109.5
C15—C14—H14A	109.3	H28A—C28—H28C	109.5
C15'—C14—H14B	85.1	H28B—C28—H28C	109.5

O4—C14—H14B	109.3	C6—N1—C8	117.50 (16)
C15—C14—H14B	109.3	C6—N1—C9	113.46 (15)
H14A—C14—H14B	107.9	C8—N1—C9	109.62 (16)
C15'—C14—H14C	110.1	C10—N2—C12	110.98 (15)
O4—C14—H14C	109.7	C10—N2—C8	120.71 (17)
C15—C14—H14C	129.4	C12—N2—C8	117.09 (15)
H14A—C14—H14C	82.5	C11—N3—C9	118.69 (16)
C15'—C14—H14D	109.7	C11—N3—C12	110.95 (15)
O4—C14—H14D	110.2	C9—N3—C12	116.54 (15)
C15—C14—H14D	83.1	C10—N4—C16	110.48 (15)
H14B—C14—H14D	129.8	C10—N4—C20	118.04 (17)
H14C—C14—H14D	108.3	C16—N4—C20	116.66 (15)
C14—C15—H15A	109.5	C11—N5—C21	120.77 (16)
C14—C15—H15B	109.5	C11—N5—C16	111.50 (15)
C14—C15—H15C	109.5	C21—N5—C16	116.32 (15)
C14—C15'—H15D	109.5	C22—N6—C21	116.54 (16)
C14—C15'—H15E	109.5	C22—N6—C20	115.66 (16)
H15D—C15'—H15E	109.5	C21—N6—C20	109.98 (16)
C14—C15'—H15F	109.5	C13—O4—C14	116.89 (19)
H15D—C15'—H15F	109.5	C17—O6—C18	118.24 (18)
H15E—C15'—H15F	109.5		
C7—C2—C3—C4	0.6 (4)	N3—C12—N2—C8	−42.0 (2)
C1—C2—C3—C4	179.8 (3)	C13—C12—N2—C8	82.7 (2)
C2—C3—C4—C5	−0.1 (4)	C16—C12—N2—C8	−152.79 (16)
C3—C4—C5—C6	−0.4 (4)	N1—C8—N2—C10	−88.0 (2)
C4—C5—C6—C7	0.2 (3)	N1—C8—N2—C12	52.4 (2)
C4—C5—C6—N1	179.1 (2)	O2—C11—N3—C9	26.0 (3)
C5—C6—C7—C2	0.4 (3)	N5—C11—N3—C9	−157.13 (16)
N1—C6—C7—C2	−178.43 (19)	O2—C11—N3—C12	165.04 (18)
C3—C2—C7—C6	−0.8 (3)	N5—C11—N3—C12	−18.1 (2)
C1—C2—C7—C6	−180.0 (2)	N1—C9—N3—C11	85.2 (2)
N3—C12—C13—O3	6.8 (3)	N1—C9—N3—C12	−51.6 (2)
N2—C12—C13—O3	−118.0 (2)	N2—C12—N3—C11	−98.72 (18)
C16—C12—C13—O3	124.9 (2)	C13—C12—N3—C11	137.07 (16)
N3—C12—C13—O4	−173.02 (16)	C16—C12—N3—C11	11.95 (19)
N2—C12—C13—O4	62.2 (2)	N2—C12—N3—C9	41.3 (2)
C16—C12—C13—O4	−54.9 (2)	C13—C12—N3—C9	−83.0 (2)
N3—C12—C16—N4	−118.72 (15)	C16—C12—N3—C9	151.93 (16)
N2—C12—C16—N4	−2.15 (18)	O1—C10—N4—C16	164.86 (19)
C13—C12—C16—N4	118.91 (18)	N2—C10—N4—C16	−18.2 (2)
N3—C12—C16—N5	−1.75 (18)	O1—C10—N4—C20	27.0 (3)
N2—C12—C16—N5	114.82 (15)	N2—C10—N4—C20	−156.09 (16)
C13—C12—C16—N5	−124.13 (17)	N5—C16—N4—C10	−98.17 (18)
N3—C12—C16—C17	116.50 (18)	C17—C16—N4—C10	138.78 (17)
N2—C12—C16—C17	−126.92 (17)	C12—C16—N4—C10	12.19 (19)
C13—C12—C16—C17	−5.9 (2)	N5—C16—N4—C20	40.3 (2)
N4—C16—C17—O5	12.0 (3)	C17—C16—N4—C20	−82.7 (2)
N5—C16—C17—O5	−113.0 (2)	C12—C16—N4—C20	150.68 (16)
C12—C16—C17—O5	131.8 (2)	N6—C20—N4—C10	85.2 (2)

N4—C16—C17—O6	−170.98 (16)	N6—C20—N4—C16	−50.1 (2)
N5—C16—C17—O6	64.0 (2)	O2—C11—N5—C21	−24.1 (3)
C12—C16—C17—O6	−51.2 (2)	N3—C11—N5—C21	159.02 (15)
C27—C22—C23—C24	−1.0 (3)	O2—C11—N5—C16	−166.26 (18)
N6—C22—C23—C24	176.1 (2)	N3—C11—N5—C16	16.9 (2)
C22—C23—C24—C25	1.4 (4)	N6—C21—N5—C11	−86.8 (2)
C23—C24—C25—C26	−1.1 (4)	N6—C21—N5—C16	53.6 (2)
C24—C25—C26—C27	0.3 (4)	N4—C16—N5—C11	101.83 (18)
C24—C25—C26—C28	179.5 (3)	C17—C16—N5—C11	−132.47 (17)
C23—C22—C27—C26	0.2 (3)	C12—C16—N5—C11	−9.00 (19)
N6—C22—C27—C26	−176.70 (19)	N4—C16—N5—C21	−42.1 (2)
C25—C26—C27—C22	0.1 (3)	C17—C16—N5—C21	83.6 (2)
C28—C26—C27—C22	−179.1 (2)	C12—C16—N5—C21	−152.95 (16)
C7—C6—N1—C8	11.7 (3)	C27—C22—N6—C21	13.4 (3)
C5—C6—N1—C8	−167.11 (19)	C23—C22—N6—C21	−163.54 (19)
C7—C6—N1—C9	−118.0 (2)	C27—C22—N6—C20	−118.2 (2)
C5—C6—N1—C9	63.2 (2)	C23—C22—N6—C20	64.9 (2)
N2—C8—N1—C6	168.14 (16)	N5—C21—N6—C22	163.66 (16)
N2—C8—N1—C9	−60.4 (2)	N5—C21—N6—C20	−62.2 (2)
N3—C9—N1—C6	−165.72 (16)	N4—C20—N6—C22	−164.63 (17)
N3—C9—N1—C8	60.7 (2)	N4—C20—N6—C21	60.8 (2)
O1—C10—N2—C12	−166.38 (19)	O3—C13—O4—C14	−5.7 (3)
N4—C10—N2—C12	16.7 (2)	C12—C13—O4—C14	174.17 (18)
O1—C10—N2—C8	−23.7 (3)	C15'—C14—O4—C13	116.8 (10)
N4—C10—N2—C8	159.39 (16)	C15—C14—O4—C13	87.0 (8)
N3—C12—N2—C10	102.14 (18)	O5—C17—O6—C18	−5.5 (3)
C13—C12—N2—C10	−133.11 (17)	C16—C17—O6—C18	177.54 (18)
C16—C12—N2—C10	−8.64 (19)	C19—C18—O6—C17	−177.4 (2)

Fig. 1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