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

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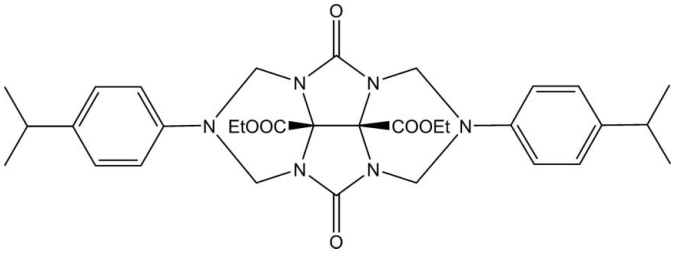
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; R factor = 0.059; wR factor = 0.167; data-to-parameter ratio = 13.3.

In the title compound, $C_{32}H_{40}N_6O_6$, the dihedral angles between the two fused five-membered rings in the glycoluril unit and between the two benzene rings are 72.5 (4) and 84.4 (1)°, respectively.

Related literature

For related literature, see: Freeman *et al.* (1981); Rowan *et al.* (1999); Hof *et al.* (2002); Lagona *et al.* (2003); Lee *et al.* (2003). Details of the synthesis are given by Yin *et al.* (2006).



Experimental

Crystal data

$C_{32}H_{40}N_6O_6$
M_r = 604.70
Triclinic, $P\overline{1}$
a = 10.109 (1) Å
b = 12.076 (2) Å
c = 13.356 (2) Å
α = 94.923 (2)°
β = 107.235 (2)°
γ = 91.014 (2)°
V = 1549.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
7805 measured reflections
5378 independent reflections
4588 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.167
S = 1.04
5378 reflections
403 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2201).

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supplementary materials

Acta Cryst. (2007). E**63**, o3615 [**doi:10.1107/S160053680703440X**]

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

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Comment

Since Mock and co-workers first characterized the chemical nature and structure of cucurbit[6]uril (Freeman *et al.*, 1981), many receptors based on glycoluril have been reported, including Nolte's molecular clips and molecular baskets (Rowan *et al.*, 1999), Rebek's molecular capsules (Hof *et al.*, 2002), CB[*n*] homologues (n = 5, 7 and 8; Lee *et al.*, 2003) and CB[*n*] (n = 5–7) derivatives (Lee *et al.*, 2003; Lagona *et al.*, 2003). The title compound (Fig. 1) is a new type of receptor based on glycoluril.

Experimental

The title compound was synthesized according to the literature procedure (Yin *et al.*, 2006). Crystals suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane-methanol (2:1) solution under ambient conditions.

Refinement

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) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C) was applied. The displacement parameters of atom C2 were restrained to appoximate isotropic behaviour.

Figures

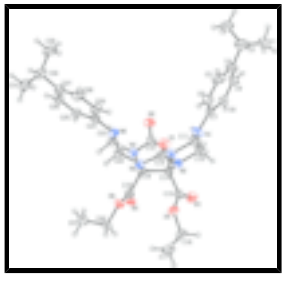


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

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

Crystal data

$C_{32}H_{40}N_6O_6$	$Z = 2$
$M_r = 604.70$	$F_{000} = 644$
Triclinic, $P\overline{1}$	$D_x = 1.296$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.1090 (13) Å	Cell parameters from 4147 reflections
b = 12.0760 (16) Å	θ = 2.4–28.2º
c = 13.3560 (18) Å	μ = 0.09 mm^{-1}
α = 94.923 (2)º	T = 292 (2) K
β = 107.235 (2)º	Block, colorless
γ = 91.014 (2)º	0.30 × 0.20 × 0.10 mm
V = 1549.9 (4) Å^3	

Data collection

Bruker SMART CCD diffractometer	4588 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.021
Monochromator: graphite	θ_{max} = 25.0º
T = 292(2) K	θ_{min} = 1.6º
φ and ω scans	h = −9→12
Absorption correction: none	k = −14→12
7805 measured reflections	l = −15→15
5378 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
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H-atom parameters constrained
$wR(F^2)$ = 0.167	$w = 1/[\sigma^2(F_o^2) + (0.0849P)^2 + 0.7695P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
5378 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
403 parameters	$\Delta\rho_{min}$ = −0.64 e Å^{-3}
6 restraints	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}
C1	0.8029 (4)	0.3378 (4)	0.7675 (3)	0.0918 (12)
H1A	0.9015	0.3516	0.7942	0.138*
H1B	0.7736	0.2881	0.8099	0.138*
H1C	0.7576	0.4068	0.7699	0.138*
C2	0.7693 (4)	0.2918 (6)	0.6675 (4)	0.143 (2)
H2	0.7862	0.2168	0.6898	0.171*
C3	0.8587 (4)	0.2801 (5)	0.6040 (4)	0.129 (2)
H3A	0.8314	0.3281	0.5484	0.193*
H3B	0.8533	0.2044	0.5743	0.193*
H3C	0.9522	0.3000	0.6462	0.193*
C4	0.6189 (3)	0.2613 (5)	0.6094 (3)	0.0954 (15)
C5	0.5381 (4)	0.3374 (4)	0.5519 (3)	0.0897 (13)
H5	0.5773	0.4078	0.5524	0.108*
C6	0.4001 (3)	0.3147 (3)	0.4927 (2)	0.0655 (8)
H6	0.3498	0.3685	0.4537	0.079*
C7	0.3385 (2)	0.2112 (2)	0.49238 (18)	0.0459 (6)
C8	0.4174 (3)	0.1347 (3)	0.5515 (2)	0.0564 (7)
H8	0.3783	0.0651	0.5539	0.068*
C9	0.5555 (3)	0.1612 (4)	0.6078 (2)	0.0783 (10)
H9	0.6067	0.1074	0.6462	0.094*
C10	0.0931 (3)	0.2609 (2)	0.4350 (2)	0.0525 (6)
H10A	0.1322	0.3359	0.4589	0.063*
H10B	0.0495	0.2368	0.4855	0.063*
C11	0.1464 (3)	0.0729 (2)	0.3981 (2)	0.0495 (6)
H11A	0.1020	0.0500	0.4487	0.059*
H11B	0.2203	0.0232	0.3969	0.059*
C12	0.0131 (2)	0.33010 (19)	0.26148 (19)	0.0416 (5)
C13	0.0956 (2)	0.05948 (17)	0.20743 (18)	0.0391 (5)
C14	−0.0621 (2)	0.15017 (18)	0.27892 (17)	0.0376 (5)
C15	−0.1849 (2)	0.1101 (2)	0.31536 (18)	0.0451 (6)
C16	−0.4158 (3)	0.1497 (3)	0.3121 (2)	0.0598 (7)
H16A	−0.4009	0.1655	0.3871	0.072*
H16B	−0.4420	0.0714	0.2928	0.072*
C17	−0.5262 (3)	0.2183 (3)	0.2529 (3)	0.0813 (10)
H17A	−0.4988	0.2956	0.2719	0.122*
H17B	−0.6102	0.2028	0.2697	0.122*
H17C	−0.5414	0.2010	0.1788	0.122*
C18	−0.0971 (2)	0.17054 (17)	0.15953 (17)	0.0358 (5)
C19	−0.2451 (2)	0.13019 (19)	0.09281 (18)	0.0398 (5)
C20	−0.4616 (3)	0.1772 (2)	−0.0195 (2)	0.0538 (6)
H20A	−0.5201	0.2404	−0.0233	0.065*
H20B	−0.4998	0.1192	0.0111	0.065*
C21	−0.4605 (3)	0.1357 (3)	−0.1274 (2)	0.0693 (8)
H21A	−0.4227	0.1932	−0.1577	0.104*
H21B	−0.5535	0.1153	−0.1703	0.104*

H21C	−0.4046	0.0719	−0.1238	0.104*
C22	−0.0256 (3)	0.32243 (19)	0.0719 (2)	0.0435 (5)
H22A	0.0058	0.4002	0.0855	0.052*
H22B	−0.1055	0.3145	0.0095	0.052*
C23	0.0401 (2)	0.13656 (18)	0.03493 (18)	0.0411 (5)
H23A	−0.0386	0.1248	−0.0281	0.049*
H23B	0.1144	0.0917	0.0244	0.049*
C24	0.1447 (2)	0.29381 (19)	−0.02230 (19)	0.0416 (5)
C25	0.1541 (3)	0.2275 (2)	−0.1101 (2)	0.0478 (6)
H25	0.1197	0.1541	−0.1217	0.057*
C26	0.2139 (3)	0.2695 (2)	−0.1802 (2)	0.0515 (6)
H26	0.2199	0.2230	−0.2378	0.062*
C27	0.2645 (3)	0.3770 (2)	−0.1678 (2)	0.0548 (7)
C28	0.2548 (3)	0.4435 (2)	−0.0809 (3)	0.0649 (8)
H28	0.2876	0.5173	−0.0708	0.078*
C29	0.1969 (3)	0.4025 (2)	−0.0084 (2)	0.0560 (7)
H29	0.1933	0.4486	0.0501	0.067*
C30	0.3317 (3)	0.4201 (3)	−0.2465 (3)	0.0685 (8)
H30	0.3409	0.3554	−0.2931	0.082*
C31	0.2418 (4)	0.4985 (3)	−0.3153 (3)	0.0831 (10)
H31A	0.2367	0.5663	−0.2736	0.125*
H31B	0.1504	0.4648	−0.3460	0.125*
H31C	0.2807	0.5146	−0.3701	0.125*
C32	0.4765 (4)	0.4688 (4)	−0.1925 (3)	0.0910 (12)
H32A	0.5184	0.4890	−0.2445	0.136*
H32B	0.5308	0.4146	−0.1518	0.136*
H32C	0.4723	0.5336	−0.1469	0.136*
N1	0.2021 (2)	0.18694 (18)	0.42762 (18)	0.0561 (6)
N2	−0.00950 (19)	0.25972 (16)	0.33196 (15)	0.0406 (4)
N3	0.04502 (19)	0.06902 (15)	0.29320 (15)	0.0395 (4)
N4	0.00119 (19)	0.10323 (15)	0.12450 (14)	0.0374 (4)
N5	−0.06466 (19)	0.28778 (15)	0.16120 (15)	0.0380 (4)
N6	0.0858 (2)	0.25419 (15)	0.05369 (15)	0.0411 (5)
O1	0.0824 (2)	0.41615 (14)	0.28468 (16)	0.0589 (5)
O2	0.20021 (18)	0.01458 (15)	0.20352 (15)	0.0539 (5)
O3	−0.1824 (2)	0.0326 (2)	0.36445 (18)	0.0741 (6)
O4	−0.28856 (17)	0.17690 (15)	0.28589 (15)	0.0526 (5)
O5	−0.28085 (18)	0.03424 (15)	0.08548 (16)	0.0595 (5)
O6	−0.32039 (16)	0.21045 (14)	0.04754 (13)	0.0477 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0514 (18)	0.145 (4)	0.066 (2)	−0.018 (2)	0.0034 (15)	−0.001 (2)
C2	0.052 (2)	0.236 (5)	0.121 (3)	−0.030 (3)	0.025 (2)	−0.082 (3)
C3	0.0457 (19)	0.235 (6)	0.100 (3)	−0.029 (3)	0.031 (2)	−0.043 (3)
C4	0.0376 (17)	0.165 (4)	0.071 (2)	−0.016 (2)	0.0142 (16)	−0.047 (3)
C5	0.070 (2)	0.103 (3)	0.095 (3)	−0.048 (2)	0.041 (2)	−0.042 (2)

C6	0.0671 (19)	0.0655 (18)	0.0625 (17)	−0.0134 (15)	0.0211 (15)	−0.0052 (14)
C7	0.0398 (13)	0.0605 (15)	0.0355 (12)	−0.0056 (11)	0.0097 (10)	0.0002 (11)
C8	0.0439 (14)	0.0768 (19)	0.0442 (14)	0.0007 (13)	0.0056 (11)	0.0102 (13)
C9	0.0429 (16)	0.133 (3)	0.0486 (16)	0.0147 (19)	0.0012 (13)	−0.0077 (18)
C10	0.0482 (14)	0.0548 (15)	0.0460 (14)	0.0009 (12)	0.0033 (11)	−0.0043 (11)
C11	0.0428 (13)	0.0501 (14)	0.0467 (14)	0.0016 (11)	−0.0007 (11)	0.0070 (11)
C12	0.0353 (12)	0.0362 (12)	0.0535 (14)	0.0011 (9)	0.0153 (10)	−0.0007 (10)
C13	0.0375 (12)	0.0292 (11)	0.0483 (13)	−0.0020 (9)	0.0093 (10)	0.0032 (9)
C14	0.0329 (11)	0.0383 (12)	0.0397 (12)	−0.0023 (9)	0.0077 (9)	0.0046 (9)
C15	0.0408 (13)	0.0539 (14)	0.0396 (12)	−0.0062 (11)	0.0101 (10)	0.0073 (11)
C16	0.0450 (15)	0.0777 (19)	0.0634 (17)	−0.0083 (13)	0.0272 (13)	0.0064 (14)
C17	0.0485 (17)	0.112 (3)	0.095 (2)	0.0093 (17)	0.0355 (17)	0.023 (2)
C18	0.0325 (11)	0.0338 (11)	0.0412 (12)	0.0000 (9)	0.0109 (9)	0.0049 (9)
C19	0.0363 (12)	0.0400 (13)	0.0421 (12)	−0.0016 (10)	0.0097 (10)	0.0059 (10)
C20	0.0325 (12)	0.0692 (17)	0.0543 (15)	0.0043 (11)	0.0040 (11)	0.0081 (13)
C21	0.0507 (16)	0.093 (2)	0.0564 (17)	0.0038 (15)	0.0060 (13)	0.0002 (15)
C22	0.0465 (13)	0.0369 (12)	0.0523 (14)	0.0040 (10)	0.0213 (11)	0.0092 (10)
C23	0.0439 (13)	0.0367 (12)	0.0447 (12)	0.0009 (10)	0.0163 (10)	0.0036 (10)
C24	0.0348 (11)	0.0451 (13)	0.0465 (13)	0.0034 (10)	0.0129 (10)	0.0096 (10)
C25	0.0444 (13)	0.0497 (14)	0.0525 (14)	0.0040 (11)	0.0189 (11)	0.0057 (11)
C26	0.0446 (14)	0.0618 (16)	0.0515 (14)	0.0084 (12)	0.0185 (11)	0.0085 (12)
C27	0.0482 (14)	0.0679 (18)	0.0564 (15)	0.0121 (13)	0.0245 (12)	0.0165 (13)
C28	0.0658 (18)	0.0506 (16)	0.088 (2)	−0.0080 (13)	0.0361 (16)	0.0157 (15)
C29	0.0621 (17)	0.0506 (15)	0.0635 (17)	−0.0056 (12)	0.0325 (14)	0.0020 (12)
C30	0.073 (2)	0.0697 (19)	0.080 (2)	0.0096 (15)	0.0453 (17)	0.0198 (16)
C31	0.083 (2)	0.102 (3)	0.082 (2)	0.019 (2)	0.0419 (19)	0.039 (2)
C32	0.064 (2)	0.112 (3)	0.117 (3)	0.0039 (19)	0.049 (2)	0.043 (2)
N1	0.0418 (12)	0.0503 (13)	0.0613 (13)	0.0017 (9)	−0.0049 (10)	−0.0028 (10)
N2	0.0351 (10)	0.0412 (10)	0.0427 (10)	0.0003 (8)	0.0088 (8)	−0.0011 (8)
N3	0.0354 (10)	0.0380 (10)	0.0418 (10)	0.0008 (8)	0.0055 (8)	0.0065 (8)
N4	0.0372 (10)	0.0340 (9)	0.0417 (10)	0.0015 (8)	0.0124 (8)	0.0049 (8)
N5	0.0387 (10)	0.0323 (9)	0.0448 (10)	−0.0007 (8)	0.0152 (8)	0.0037 (8)
N6	0.0437 (11)	0.0361 (10)	0.0482 (11)	0.0016 (8)	0.0202 (9)	0.0065 (8)
O1	0.0587 (11)	0.0408 (10)	0.0735 (12)	−0.0139 (8)	0.0180 (9)	−0.0048 (9)
O2	0.0454 (10)	0.0512 (10)	0.0668 (12)	0.0148 (8)	0.0173 (9)	0.0094 (8)
O3	0.0567 (12)	0.0890 (15)	0.0856 (15)	0.0010 (11)	0.0237 (11)	0.0491 (13)
O4	0.0395 (9)	0.0599 (11)	0.0657 (11)	0.0007 (8)	0.0249 (8)	0.0139 (9)
O5	0.0446 (10)	0.0460 (11)	0.0761 (13)	−0.0100 (8)	−0.0008 (9)	0.0102 (9)
O6	0.0360 (9)	0.0505 (10)	0.0518 (10)	0.0038 (7)	0.0039 (7)	0.0105 (8)

Geometric parameters (Å, °)

C1—C2	1.344 (6)	C17—H17A	0.960
C1—H1A	0.960	C17—H17B	0.960
C1—H1B	0.960	C17—H17C	0.960
C1—H1C	0.960	C18—N5	1.444 (3)
C2—C3	1.412 (6)	C18—N4	1.451 (3)
C2—C4	1.510 (5)	C18—C19	1.542 (3)
C2—H2	0.980	C19—O5	1.195 (3)

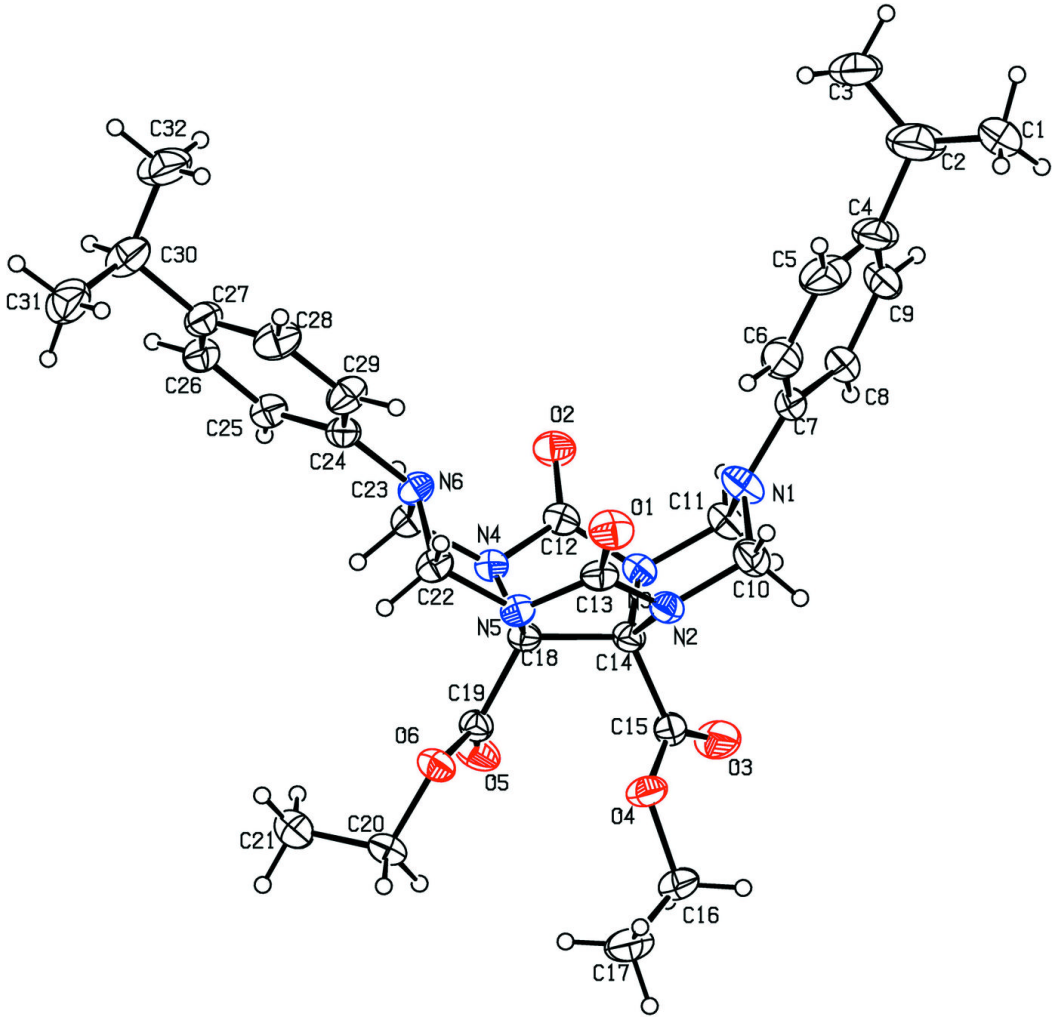
C3—H3A	0.960	C19—O6	1.318 (3)
C3—H3B	0.960	C20—O6	1.470 (3)
C3—H3C	0.960	C20—C21	1.487 (4)
C4—C9	1.354 (6)	C20—H20A	0.970
C4—C5	1.371 (6)	C20—H20B	0.970
C5—C6	1.393 (5)	C21—H21A	0.960
C5—H5	0.930	C21—H21B	0.960
C6—C7	1.385 (4)	C21—H21C	0.960
C6—H6	0.930	C22—N5	1.453 (3)
C7—C8	1.376 (4)	C22—N6	1.472 (3)
C7—N1	1.403 (3)	C22—H22A	0.970
C8—C9	1.391 (4)	C22—H22B	0.970
C8—H8	0.930	C23—N4	1.450 (3)
C9—H9	0.930	C23—N6	1.463 (3)
C10—N1	1.451 (3)	C23—H23A	0.970
C10—N2	1.456 (3)	C23—H23B	0.970
C10—H10A	0.970	C24—C29	1.382 (4)
C10—H10B	0.970	C24—C25	1.390 (4)
C11—N1	1.455 (3)	C24—N6	1.430 (3)
C11—N3	1.467 (3)	C25—C26	1.380 (4)
C11—H11A	0.970	C25—H25	0.930
C11—H11B	0.970	C26—C27	1.365 (4)
C12—O1	1.207 (3)	C26—H26	0.930
C12—N5	1.385 (3)	C27—C28	1.383 (4)
C12—N2	1.385 (3)	C27—C30	1.531 (4)
C13—O2	1.210 (3)	C28—C29	1.391 (4)
C13—N4	1.381 (3)	C28—H28	0.930
C13—N3	1.383 (3)	C29—H29	0.930
C14—N3	1.455 (3)	C30—C31	1.500 (5)
C14—N2	1.458 (3)	C30—C32	1.509 (5)
C14—C15	1.546 (3)	C30—H30	0.980
C14—C18	1.570 (3)	C31—H31A	0.960
C15—O3	1.184 (3)	C31—H31B	0.960
C15—O4	1.321 (3)	C31—H31C	0.960
C16—O4	1.468 (3)	C32—H32A	0.960
C16—C17	1.475 (5)	C32—H32B	0.960
C16—H16A	0.970	C32—H32C	0.960
C16—H16B	0.970		
C2—C1—H1A	109.5	O5—C19—C18	120.2 (2)
C2—C1—H1B	109.5	O6—C19—C18	113.29 (18)
H1A—C1—H1B	109.5	O6—C20—C21	110.6 (2)
C2—C1—H1C	109.5	O6—C20—H20A	109.5
H1A—C1—H1C	109.5	C21—C20—H20A	109.5
H1B—C1—H1C	109.5	O6—C20—H20B	109.5
C1—C2—C3	126.4 (4)	C21—C20—H20B	109.5
C1—C2—C4	118.9 (4)	H20A—C20—H20B	108.1
C3—C2—C4	114.2 (4)	C20—C21—H21A	109.5
C1—C2—H2	92.3	C20—C21—H21B	109.5
C3—C2—H2	92.3	H21A—C21—H21B	109.5

C4—C2—H2	92.3	C20—C21—H21C	109.5
C2—C3—H3A	109.5	H21A—C21—H21C	109.5
C2—C3—H3B	109.5	H21B—C21—H21C	109.5
H3A—C3—H3B	109.5	N5—C22—N6	110.20 (18)
C2—C3—H3C	109.5	N5—C22—H22A	109.6
H3A—C3—H3C	109.5	N6—C22—H22A	109.6
H3B—C3—H3C	109.5	N5—C22—H22B	109.6
C9—C4—C5	115.8 (3)	N6—C22—H22B	109.6
C9—C4—C2	124.6 (5)	H22A—C22—H22B	108.1
C5—C4—C2	119.6 (5)	N4—C23—N6	109.62 (18)
C4—C5—C6	123.3 (4)	N4—C23—H23A	109.7
C4—C5—H5	118.4	N6—C23—H23A	109.7
C6—C5—H5	118.4	N4—C23—H23B	109.7
C7—C6—C5	119.4 (3)	N6—C23—H23B	109.7
C7—C6—H6	120.3	H23A—C23—H23B	108.2
C5—C6—H6	120.3	C29—C24—C25	117.6 (2)
C8—C7—C6	118.0 (3)	C29—C24—N6	119.3 (2)
C8—C7—N1	123.0 (2)	C25—C24—N6	123.1 (2)
C6—C7—N1	118.9 (2)	C26—C25—C24	120.7 (2)
C7—C8—C9	120.2 (3)	C26—C25—H25	119.6
C7—C8—H8	119.9	C24—C25—H25	119.6
C9—C8—H8	119.9	C27—C26—C25	122.1 (3)
C4—C9—C8	123.3 (4)	C27—C26—H26	118.9
C4—C9—H9	118.3	C25—C26—H26	118.9
C8—C9—H9	118.3	C26—C27—C28	117.4 (2)
N1—C10—N2	109.3 (2)	C26—C27—C30	120.7 (3)
N1—C10—H10A	109.8	C28—C27—C30	121.9 (3)
N2—C10—H10A	109.8	C27—C28—C29	121.4 (3)
N1—C10—H10B	109.8	C27—C28—H28	119.3
N2—C10—H10B	109.8	C29—C28—H28	119.3
H10A—C10—H10B	108.3	C24—C29—C28	120.7 (3)
N1—C11—N3	107.7 (2)	C24—C29—H29	119.7
N1—C11—H11A	110.2	C28—C29—H29	119.7
N3—C11—H11A	110.2	C31—C30—C32	112.6 (3)
N1—C11—H11B	110.2	C31—C30—C27	112.1 (2)
N3—C11—H11B	110.2	C32—C30—C27	111.7 (3)
H11A—C11—H11B	108.5	C31—C30—H30	106.6
O1—C12—N5	126.4 (2)	C32—C30—H30	106.6
O1—C12—N2	125.6 (2)	C27—C30—H30	106.6
N5—C12—N2	107.87 (18)	C30—C31—H31A	109.5
O2—C13—N4	125.6 (2)	C30—C31—H31B	109.5
O2—C13—N3	126.0 (2)	H31A—C31—H31B	109.5
N4—C13—N3	108.25 (19)	C30—C31—H31C	109.5
N3—C14—N2	112.76 (17)	H31A—C31—H31C	109.5
N3—C14—C15	111.56 (18)	H31B—C31—H31C	109.5
N2—C14—C15	109.84 (18)	C30—C32—H32A	109.5
N3—C14—C18	103.82 (17)	C30—C32—H32B	109.5
N2—C14—C18	102.48 (17)	H32A—C32—H32B	109.5
C15—C14—C18	116.05 (18)	C30—C32—H32C	109.5

O3—C15—O4	126.4 (2)	H32A—C32—H32C	109.5
O3—C15—C14	124.0 (2)	H32B—C32—H32C	109.5
O4—C15—C14	109.55 (19)	C7—N1—C10	119.9 (2)
O4—C16—C17	108.0 (2)	C7—N1—C11	121.5 (2)
O4—C16—H16A	110.1	C10—N1—C11	110.4 (2)
C17—C16—H16A	110.1	C12—N2—C10	118.6 (2)
O4—C16—H16B	110.1	C12—N2—C14	110.93 (18)
C17—C16—H16B	110.1	C10—N2—C14	115.98 (19)
H16A—C16—H16B	108.4	C13—N3—C14	110.46 (18)
C16—C17—H17A	109.5	C13—N3—C11	117.33 (19)
C16—C17—H17B	109.5	C14—N3—C11	116.62 (19)
H17A—C17—H17B	109.5	C13—N4—C23	121.57 (19)
C16—C17—H17C	109.5	C13—N4—C18	111.81 (18)
H17A—C17—H17C	109.5	C23—N4—C18	117.17 (17)
H17B—C17—H17C	109.5	C12—N5—C18	110.17 (18)
N5—C18—N4	111.19 (17)	C12—N5—C22	118.39 (19)
N5—C18—C19	115.38 (18)	C18—N5—C22	116.62 (17)
N4—C18—C19	108.58 (17)	C24—N6—C23	115.17 (18)
N5—C18—C14	104.16 (17)	C24—N6—C22	113.58 (18)
N4—C18—C14	102.78 (17)	C23—N6—C22	109.95 (18)
C19—C18—C14	114.03 (17)	C15—O4—C16	117.0 (2)
O5—C19—O6	126.5 (2)	C19—O6—C20	116.12 (19)
C1—C2—C4—C9	−90.4 (7)	N5—C12—N2—C14	20.4 (2)
C3—C2—C4—C9	96.9 (7)	N1—C10—N2—C12	−85.2 (3)
C1—C2—C4—C5	90.8 (7)	N1—C10—N2—C14	50.6 (3)
C3—C2—C4—C5	−81.9 (6)	N3—C14—N2—C12	100.0 (2)
C9—C4—C5—C6	−1.5 (5)	C15—C14—N2—C12	−134.86 (19)
C2—C4—C5—C6	177.4 (3)	C18—C14—N2—C12	−10.9 (2)
C4—C5—C6—C7	1.3 (5)	N3—C14—N2—C10	−39.1 (3)
C5—C6—C7—C8	0.1 (4)	C15—C14—N2—C10	86.0 (2)
C5—C6—C7—N1	−176.3 (3)	C18—C14—N2—C10	−150.0 (2)
C6—C7—C8—C9	−1.2 (4)	O2—C13—N3—C14	166.1 (2)
N1—C7—C8—C9	175.0 (3)	N4—C13—N3—C14	−17.9 (2)
C5—C4—C9—C8	0.3 (5)	O2—C13—N3—C11	29.1 (3)
C2—C4—C9—C8	−178.5 (3)	N4—C13—N3—C11	−154.93 (19)
C7—C8—C9—C4	1.0 (5)	N2—C14—N3—C13	−97.2 (2)
N3—C14—C15—O3	11.7 (3)	C15—C14—N3—C13	138.60 (19)
N2—C14—C15—O3	−114.1 (3)	C18—C14—N3—C13	12.9 (2)
C18—C14—C15—O3	130.3 (3)	N2—C14—N3—C11	40.1 (3)
N3—C14—C15—O4	−170.08 (19)	C15—C14—N3—C11	−84.1 (2)
N2—C14—C15—O4	64.1 (2)	C18—C14—N3—C11	150.24 (19)
C18—C14—C15—O4	−51.5 (3)	N1—C11—N3—C13	82.6 (3)
N3—C14—C18—N5	−119.46 (17)	N1—C11—N3—C14	−51.8 (3)
N2—C14—C18—N5	−1.9 (2)	O2—C13—N4—C23	−23.0 (3)
C15—C14—C18—N5	117.8 (2)	N3—C13—N4—C23	161.09 (18)
N3—C14—C18—N4	−3.4 (2)	O2—C13—N4—C18	−168.4 (2)
N2—C14—C18—N4	114.16 (17)	N3—C13—N4—C18	15.7 (2)
C15—C14—C18—N4	−126.2 (2)	N6—C23—N4—C13	−91.5 (2)
N3—C14—C18—C19	113.93 (19)	N6—C23—N4—C18	52.2 (3)

N2—C14—C18—C19	−128.52 (18)	N5—C18—N4—C13	103.7 (2)
C15—C14—C18—C19	−8.8 (3)	C19—C18—N4—C13	−128.28 (19)
N5—C18—C19—O5	179.0 (2)	C14—C18—N4—C13	−7.2 (2)
N4—C18—C19—O5	53.4 (3)	N5—C18—N4—C23	−43.3 (3)
C14—C18—C19—O5	−60.5 (3)	C19—C18—N4—C23	84.7 (2)
N5—C18—C19—O6	−0.4 (3)	C14—C18—N4—C23	−154.23 (18)
N4—C18—C19—O6	−126.0 (2)	O1—C12—N5—C18	162.1 (2)
C14—C18—C19—O6	120.1 (2)	N2—C12—N5—C18	−21.7 (2)
C29—C24—C25—C26	0.2 (4)	O1—C12—N5—C22	24.3 (3)
N6—C24—C25—C26	−179.0 (2)	N2—C12—N5—C22	−159.50 (18)
C24—C25—C26—C27	−0.8 (4)	N4—C18—N5—C12	−95.9 (2)
C25—C26—C27—C28	0.5 (4)	C19—C18—N5—C12	139.94 (19)
C25—C26—C27—C30	179.2 (3)	C14—C18—N5—C12	14.2 (2)
C26—C27—C28—C29	0.6 (4)	N4—C18—N5—C22	42.8 (3)
C30—C27—C28—C29	−178.2 (3)	C19—C18—N5—C22	−81.4 (2)
C25—C24—C29—C28	0.9 (4)	C14—C18—N5—C22	152.83 (18)
N6—C24—C29—C28	−180.0 (3)	N6—C22—N5—C12	83.5 (2)
C27—C28—C29—C24	−1.3 (5)	N6—C22—N5—C18	−51.6 (3)
C26—C27—C30—C31	108.0 (3)	C29—C24—N6—C23	−176.9 (2)
C28—C27—C30—C31	−73.3 (4)	C25—C24—N6—C23	2.2 (3)
C26—C27—C30—C32	−124.5 (3)	C29—C24—N6—C22	55.0 (3)
C28—C27—C30—C32	54.2 (4)	C25—C24—N6—C22	−125.9 (2)
C8—C7—N1—C10	130.1 (3)	N4—C23—N6—C24	171.94 (18)
C6—C7—N1—C10	−53.8 (3)	N4—C23—N6—C22	−58.2 (2)
C8—C7—N1—C11	−15.4 (4)	N5—C22—N6—C24	−171.00 (19)
C6—C7—N1—C11	160.7 (3)	N5—C22—N6—C23	58.3 (2)
N2—C10—N1—C7	148.0 (2)	O3—C15—O4—C16	−3.1 (4)
N2—C10—N1—C11	−63.0 (3)	C14—C15—O4—C16	178.7 (2)
N3—C11—N1—C7	−148.9 (2)	C17—C16—O4—C15	−167.8 (3)
N3—C11—N1—C10	62.8 (3)	O5—C19—O6—C20	−0.9 (4)
O1—C12—N2—C10	−25.4 (3)	C18—C19—O6—C20	178.48 (19)
N5—C12—N2—C10	158.34 (19)	C21—C20—O6—C19	−84.4 (3)
O1—C12—N2—C14	−163.3 (2)		

Fig. 1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