Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethyl 2,6-bis(4-ethynylphenyl)-4,8-dioxoperhydro-2,3a,4a,6,7a,8a-hexaazacyclopenta[*def*]fluorene-8b,8c-dicarboxylate

Sheng-Li Hu,*‡ Shuai Wang and Liping Cao

Key Laboratory of Pesticides & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: hushengli168@126.com

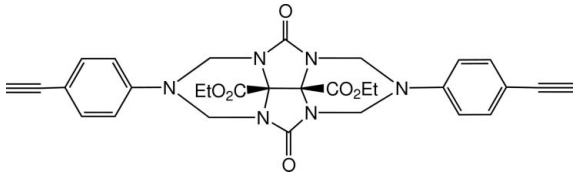
Received 24 October 2007; accepted 30 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.053; wR factor = 0.147; data-to-parameter ratio = 13.3.

The molecule of the title compound, $C_{30}H_{28}N_6O_6$, a glycoluril derivative, lies on a twofold rotation axis with two ethyl acetate groups bonded to the convex face of the glycoluril system. The dihedral angle between the imidazolone rings is 73.12 (3)°. Two symmetry-equivalent six-membered triazine rings are fused to the glycouril unit to form rigid side walls of a molecular clip. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds. The ethyl ester group is disordered over two sites of occupancy 0.539 (7) and 0.461 (7).

Related literature

For related literature, see: Yin *et al.* (2006); Rebek (2005); Rowan *et al.* (1999); Witt *et al.* (2000).



Experimental

Crystal data

$C_{30}H_{28}N_6O_6$
M_r = 568.58
Monoclinic, $C2/c$
a = 16.0226 (10) Å
b = 14.0617 (9) Å
c = 13.7870 (9) Å
β = 115.523 (1)°
V = 2803.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
12898 measured reflections
3057 independent reflections
1952 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.147
S = 1.05
3057 reflections
229 parameters
11 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O3^{i}	0.93	2.35	3.226 (7)	158

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

We are grateful to the Central China Normal University for financial support and to Professor Wu An-Xin for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2535).

References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Rebek, J. Jr (2005). *Angew. Chem. Int. Ed.* **44**, 2068–2078.
Rowan, A. E., Elemans, J. A. A. W. & Notle, R. J. M. (1999). *Acc. Chem. Res.* **32**, 995–1006.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Witt, D., Lagona, J., Damkaci, F., Fettinger, J. C. & Isaacs, L. (2000). *Org. Lett.* **2**, 755–758.
Yin, G., Wang, Z., Chen, Y., Wu, A. & Pan, Y. (2006). *Synlett*, pp. 49–52.

‡ Also at Hubei Key Laboratory of Bioanalytical Techniques, Hubei Normal University, Huangshi 435002, People's Republic of China.

  doi:10.1107/S160053680705458X

supplementary materials

Acta Cryst. (2007). E**63**, o4542 [**doi:10.1107/S160053680705458X**]

Diethyl 2,6-bis(4-ethynylphenyl)-4,8-dioxoperhydro-2,3a,4a,6,7a,8a-hexaaza-cyclopenta[*def*]fluorene-8b,8c-dicarboxylate

S.-L. Hu, S. Wang and L. Cao

Comment

Glycoluryl derivatives have been employed in many applications, including polymer cross-linking, explosives, stabilization of organic compounds against photo-degradation, textile waste, stream purfication, and comblinational chemistry (Witt *et al.*, 2000). They are also used as building blocks for self assembly, molecular recognition, and catalysis (Rebek, 2005; Rowan *et al.*, 1999). In this paper we report the crystal structure of the title glycoluryl derivative, (I)(Fig. 1), in which the dihedral angle between the imidazolone rings of the glycouril unit is 73.12 (3) ° and the dihedral angle between two phenyl ring is 16.82 (4)°. The molecule lies on a crystallographic twofold axis. In the crystal structure, molecules are connected by wek intermolecular C—H···O hydrogen bonds (Fig. 2).

Experimental

The title compound was synthesized in analogy to the literature procedure of Yin *et al.* (2006), Crystals appropriate for data collection were obtained by slow evaporation from a methanol-chloroform solution (1:20 V/V) of (I).

Refinement

The H atoms were constrained to an ideal geometry and constrained to ride on their parent atoms as follows: methylene H with d(C—H)=0.97 Å and U_{iso}(H) = 1.2U_{eq}(C); methine H with d(C—H)=0.98 Å and U_{iso}(H) = 1.2U_{eq}(C); aromatic H with d(C—H)=0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The unique ethyl acetate group is disorder over two sites; the site-occupancy factors for the two orientations were refined using the *DFIX* instruction in *SHELXTL* (Sheldrick, 2000) giving 0.539 (7) and 0.461 (7) for the major and minor components, respectively.

Figures

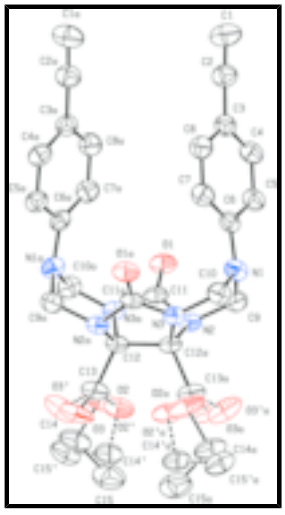


Fig. 1. The molecular structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms atoms shown as circles of arbitrary radii.

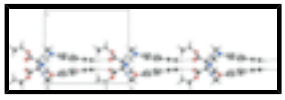


Fig. 2. The molecular packing of viewed along the *b* axis. Hydrogen bonds are shown as dashed lines

Diethyl 2,6-bis(4-ethynylphenyl)-4,8-dioxoperhydro- 2,3a,4a,6,7a,8a-hexaaza-cyclopenta[def]fluorene-8 b,8c-dicarboxylate

Crystal data

$C_{30}H_{28}N_6O_6$	F_{000} = 1192
M_r = 568.58	D_x = 1.347 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3199 reflections
a = 16.0226 (10) Å	θ = 2.2–22.7º
b = 14.0617 (9) Å	μ = 0.10 mm^{-1}
c = 13.7870 (9) Å	*T* = 294 (2) K
β = 115.523 (1)º	Plate, colorless
V = 2803.1 (3) Å^3	0.30 × 0.20 × 0.04 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1952 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.055
Monochromator: graphite	θ_{max} = 27.0º
T = 294(2) K	θ_{min} = 2.0º
/f and /w scans	*h* = −20→20
Absorption correction: none	*k* = −17→17
12898 measured reflections	*l* = −16→17

3057 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
$R[F^2 > 2\sigma(F^2)] = 0.053$	H-atom parameters constrained
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
3057 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
229 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
11 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 $F^2 > 2$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	0.1661 (2)	1.5590 (2)	0.3141 (2)	0.1019 (9)	
H1	0.1691	1.6246	0.3076	0.122*	
C2	0.16248 (15)	1.47641 (18)	0.32223 (18)	0.0773 (6)	
C3	0.16116 (13)	1.37528 (14)	0.33657 (15)	0.0638 (5)	
C4	0.22584 (14)	1.31674 (15)	0.32419 (17)	0.0687 (6)	
H4	0.2685	1.3429	0.3027	0.082*	
C5	0.22749 (13)	1.22119 (14)	0.34323 (17)	0.0668 (5)	
H5	0.2722	1.1837	0.3357	0.080*	
C6	0.16372 (12)	1.17853 (13)	0.37371 (14)	0.0581 (5)	
C7	0.09803 (13)	1.23665 (15)	0.38301 (17)	0.0692 (6)	
H7	0.0535	1.2102	0.4013	0.083*	
C8	0.09754 (14)	1.33303 (16)	0.36569 (18)	0.0715 (6)	
H8	0.0532	1.3707	0.3738	0.086*	
C9	0.19180 (13)	1.01352 (14)	0.33255 (17)	0.0676 (5)	
H9A	0.2329	0.9646	0.3778	0.081*	
H9B	0.2233	1.0459	0.2958	0.081*	

C10	0.05180 (13)	1.02199 (11)	0.16204 (14)	0.0521 (4)	
C11	−0.11956 (15)	1.03730 (15)	0.04899 (15)	0.0683 (6)	
H11A	−0.1018	1.0856	0.0112	0.082*	
H11B	−0.1581	0.9911	−0.0034	0.082*	
C12	−0.04897 (13)	0.92670 (11)	0.20293 (14)	0.0525 (4)	
C13	−0.08516 (16)	0.82917 (14)	0.15162 (19)	0.0730 (6)	
N1	0.17227 (11)	1.08128 (11)	0.39963 (13)	0.0654 (5)	
N2	0.10735 (10)	0.96859 (9)	0.25254 (12)	0.0535 (4)	
N3	−0.03683 (10)	0.99023 (9)	0.12713 (11)	0.0528 (4)	
O1	0.07893 (9)	1.08330 (8)	0.12080 (11)	0.0660 (4)	
C14	−0.0932 (6)	0.7206 (4)	0.0159 (6)	0.101 (2)	0.539 (7)
H14A	−0.1119	0.7359	−0.0592	0.121*	0.539 (7)
H14B	−0.1472	0.6988	0.0242	0.121*	0.539 (7)
C15	−0.0228 (4)	0.6458 (4)	0.0495 (6)	0.118 (3)	0.539 (7)
H15A	0.0320	0.6694	0.0460	0.176*	0.539 (7)
H15B	−0.0459	0.5920	0.0027	0.176*	0.539 (7)
H15C	−0.0085	0.6271	0.1219	0.176*	0.539 (7)
O2	−0.0548 (5)	0.8049 (4)	0.0821 (6)	0.107 (3)	0.539 (7)
O3	−0.1358 (6)	0.7852 (5)	0.1785 (7)	0.097 (2)	0.539 (7)
C14'	−0.0331 (5)	0.6950 (4)	0.0815 (5)	0.0825 (19)	0.461 (7)
H14C	0.0255	0.6739	0.0841	0.099*	0.461 (7)
H14D	−0.0503	0.6516	0.1245	0.099*	0.461 (7)
C15'	−0.1034 (6)	0.6932 (6)	−0.0291 (5)	0.097 (3)	0.461 (7)
H15D	−0.1621	0.7105	−0.0312	0.145*	0.461 (7)
H15E	−0.1073	0.6304	−0.0580	0.145*	0.461 (7)
H15F	−0.0873	0.7376	−0.0712	0.145*	0.461 (7)
O2'	−0.0227 (4)	0.7905 (4)	0.1261 (6)	0.084 (2)	0.461 (7)
O3'	−0.1586 (6)	0.7929 (7)	0.1352 (9)	0.109 (4)	0.461 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.127 (2)	0.0702 (16)	0.137 (2)	0.0139 (15)	0.085 (2)	0.0180 (16)
C2	0.0842 (14)	0.0712 (16)	0.0856 (16)	0.0025 (12)	0.0453 (13)	0.0057 (12)
C3	0.0683 (12)	0.0612 (12)	0.0584 (12)	−0.0044 (9)	0.0239 (10)	0.0000 (10)
C4	0.0735 (12)	0.0670 (13)	0.0736 (14)	−0.0136 (10)	0.0392 (11)	−0.0070 (10)
C5	0.0659 (11)	0.0654 (13)	0.0750 (14)	−0.0075 (10)	0.0360 (11)	−0.0087 (10)
C6	0.0621 (11)	0.0605 (12)	0.0471 (11)	−0.0111 (9)	0.0192 (9)	−0.0062 (9)
C7	0.0709 (12)	0.0709 (14)	0.0740 (14)	−0.0071 (10)	0.0390 (11)	−0.0002 (11)
C8	0.0704 (12)	0.0725 (14)	0.0779 (14)	0.0019 (10)	0.0380 (12)	−0.0010 (11)
C9	0.0668 (12)	0.0601 (12)	0.0805 (14)	0.0043 (9)	0.0362 (11)	0.0062 (11)
C10	0.0793 (12)	0.0375 (9)	0.0562 (11)	0.0047 (8)	0.0449 (10)	−0.0012 (8)
C11	0.0872 (14)	0.0668 (13)	0.0500 (11)	0.0066 (10)	0.0287 (11)	−0.0030 (9)
C12	0.0731 (10)	0.0372 (9)	0.0579 (11)	−0.0034 (8)	0.0385 (9)	−0.0049 (7)
C13	0.1027 (17)	0.0463 (11)	0.0882 (16)	−0.0129 (11)	0.0583 (15)	−0.0129 (11)
N1	0.0766 (10)	0.0620 (10)	0.0602 (10)	−0.0104 (8)	0.0319 (9)	−0.0036 (8)
N2	0.0677 (9)	0.0414 (8)	0.0615 (10)	0.0025 (7)	0.0375 (8)	0.0033 (7)
N3	0.0726 (10)	0.0453 (8)	0.0467 (8)	0.0002 (7)	0.0315 (8)	−0.0002 (6)

O1	0.0950 (10)	0.0526 (8)	0.0732 (9)	0.0010 (6)	0.0577 (8)	0.0087 (6)
C14	0.153 (7)	0.064 (4)	0.090 (6)	−0.026 (4)	0.057 (6)	−0.038 (4)
C15	0.140 (5)	0.085 (4)	0.148 (6)	−0.007 (4)	0.081 (5)	−0.030 (4)
O2	0.190 (6)	0.073 (3)	0.103 (5)	−0.056 (4)	0.106 (5)	−0.048 (3)
O3	0.163 (5)	0.051 (2)	0.115 (6)	−0.044 (2)	0.096 (4)	−0.038 (3)
C14'	0.111 (5)	0.057 (4)	0.086 (5)	−0.002 (4)	0.048 (4)	−0.025 (3)
C15'	0.129 (6)	0.078 (6)	0.085 (5)	−0.032 (5)	0.047 (4)	−0.023 (4)
O2'	0.125 (4)	0.055 (2)	0.094 (5)	−0.006 (2)	0.069 (4)	−0.030 (3)
O3'	0.141 (5)	0.101 (5)	0.128 (8)	−0.063 (4)	0.100 (6)	−0.057 (5)

Geometric parameters (Å, °)

C1—C2	1.170 (3)	C12—N2^{i}	1.447 (2)
C1—H1	0.9300	C12—N3	1.450 (2)
C2—C3	1.437 (3)	C12—C13	1.538 (3)
C3—C8	1.381 (3)	C12—C12^{i}	1.547 (4)
C3—C4	1.390 (3)	C13—O3	1.200 (5)
C4—C5	1.367 (3)	C13—O3'	1.211 (6)
C4—H4	0.9300	C13—O2	1.294 (4)
C5—C6	1.397 (2)	C13—O2'	1.314 (5)
C5—H5	0.9300	N1—C11^{i}	1.454 (2)
C6—C7	1.381 (3)	N2—C12^{i}	1.447 (2)
C6—N1	1.405 (2)	C14—O2	1.460 (6)
C7—C8	1.376 (3)	C14—C15	1.463 (7)
C7—H7	0.9300	C14—H14A	0.9700
C8—H8	0.9300	C14—H14B	0.9700
C9—N1	1.453 (2)	C15—H15A	0.9600
C9—N2	1.470 (2)	C15—H15B	0.9600
C9—H9A	0.9700	C15—H15C	0.9600
C9—H9B	0.9700	C14'—C15'	1.455 (7)
C10—O1	1.2118 (19)	C14'—O2'	1.456 (6)
C10—N3	1.363 (2)	C14'—H14C	0.9700
C10—N2	1.399 (2)	C14'—H14D	0.9700
C11—N1^{i}	1.454 (2)	C15'—H15D	0.9600
C11—N3	1.458 (2)	C15'—H15E	0.9600
C11—H11A	0.9700	C15'—H15F	0.9600
C11—H11B	0.9700		
C2—C1—H1	180.0	N3—C12—C12^{i}	101.66 (14)
C1—C2—C3	177.6 (3)	C13—C12—C12^{i}	115.55 (12)
C8—C3—C4	117.63 (19)	O3—C13—O2	127.8 (4)
C8—C3—C2	121.48 (18)	O3'—C13—O2	113.3 (5)
C4—C3—C2	120.86 (17)	O3—C13—O2'	124.3 (5)
C5—C4—C3	120.80 (18)	O3'—C13—O2'	124.5 (5)
C5—C4—H4	119.6	O3—C13—C12	119.3 (3)
C3—C4—H4	119.6	O3'—C13—C12	127.3 (4)
C4—C5—C6	121.58 (18)	O2—C13—C12	112.9 (3)
C4—C5—H5	119.2	O2'—C13—C12	108.2 (3)

C6—C5—H5	119.2	C6—N1—C9	120.04 (15)
C7—C6—C5	117.35 (18)	C6—N1—C11^{i}	121.63 (16)
C7—C6—N1	123.15 (16)	C9—N1—C11^{i}	111.16 (15)
C5—C6—N1	119.38 (17)	C10—N2—C12^{i}	108.53 (14)
C8—C7—C6	120.95 (17)	C10—N2—C9	117.71 (15)
C8—C7—H7	119.5	C12^{i}—N2—C9	114.26 (14)
C6—C7—H7	119.5	C10—N3—C12	112.96 (14)
C7—C8—C3	121.65 (19)	C10—N3—C11	126.10 (15)
C7—C8—H8	119.2	C12—N3—C11	116.78 (15)
C3—C8—H8	119.2	O2—C14—C15	109.0 (6)
N1—C9—N2	112.24 (14)	O2—C14—H14A	109.9
N1—C9—H9A	109.2	C15—C14—H14A	109.9
N2—C9—H9A	109.2	O2—C14—H14B	109.9
N1—C9—H9B	109.2	C15—C14—H14B	109.9
N2—C9—H9B	109.2	H14A—C14—H14B	108.3
H9A—C9—H9B	107.9	C13—O2—C14	119.1 (5)
O1—C10—N3	126.66 (17)	C15'—C14'—O2'	110.8 (6)
O1—C10—N2	125.56 (17)	C15'—C14'—H14C	109.5
N3—C10—N2	107.74 (14)	O2'—C14'—H14C	109.5
N1^{i}—C11—N3	111.55 (14)	C15'—C14'—H14D	109.5
N1^{i}—C11—H11A	109.3	O2'—C14'—H14D	109.5
N3—C11—H11A	109.3	H14C—C14'—H14D	108.1
N1^{i}—C11—H11B	109.3	C14'—C15'—H15D	109.5
N3—C11—H11B	109.3	C14'—C15'—H15E	109.5
H11A—C11—H11B	108.0	H15D—C15'—H15E	109.5
N2^{i}—C12—N3	111.53 (13)	C14'—C15'—H15F	109.5
N2^{i}—C12—C13	111.95 (14)	H15D—C15'—H15F	109.5
N3—C12—C13	111.03 (14)	H15E—C15'—H15F	109.5
N2^{i}—C12—C12^{i}	104.58 (15)	C13—O2'—C14'	122.2 (5)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O3^{ii}	0.93	2.35	3.226 (7)	158

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

Fig. 1

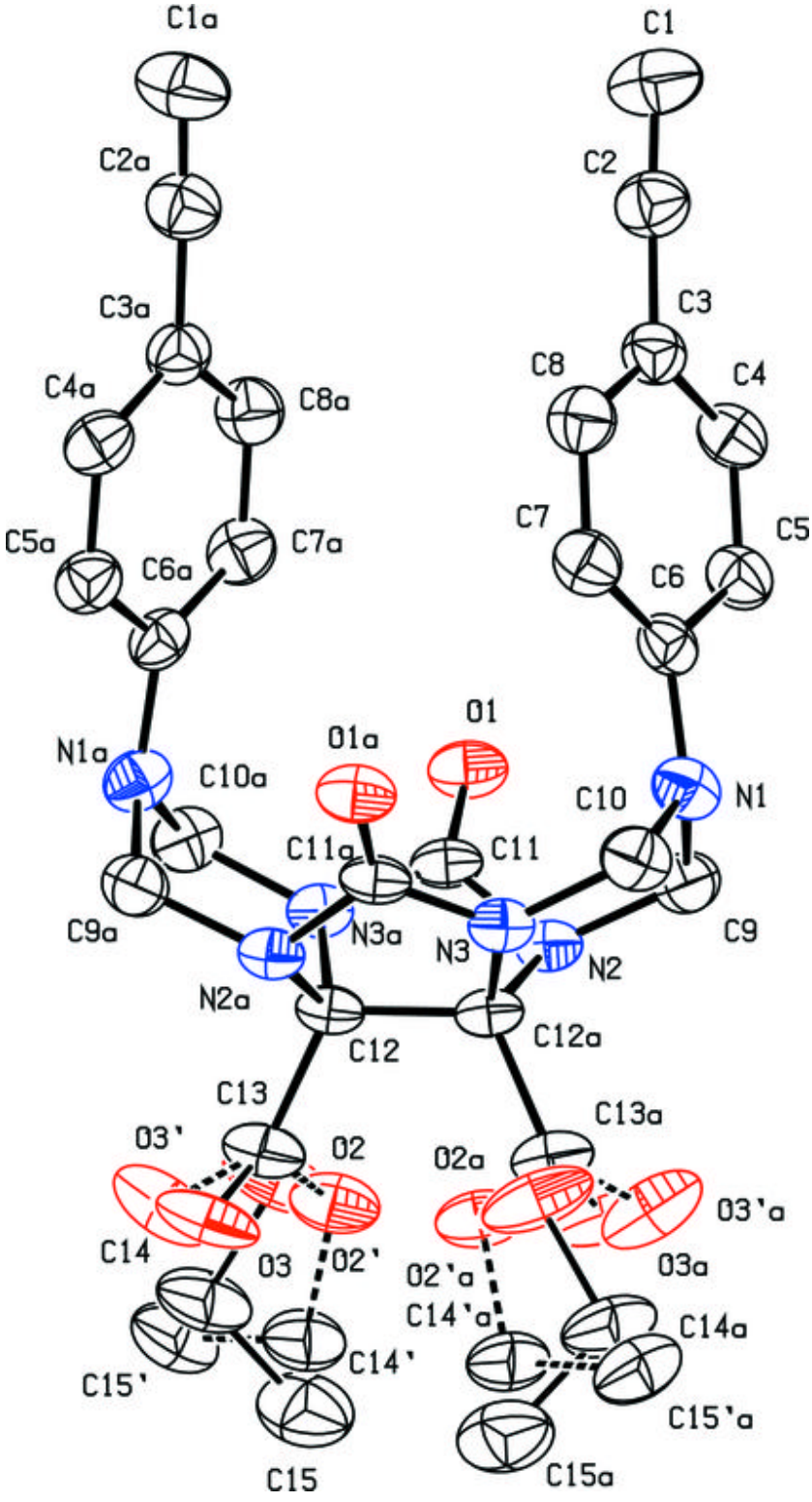

C1a
C1
C2a
C2
C3a
C3
C4a
C8a
C8
C4
C5a
C7a
C7
C5
C6a
C6
N1a
C10a
O1a
O1
C10
N1
C11a
C11
C9a
N3a
N3
N2
C9
N2a
C12
C12a
C13
C13a
O3'
O2
O2a
O3'a
C14
O3
O2'
O2'a
O3a
C14'a
C14a
C14'
C15'
C15'a
C15
C15a

Fig. 2