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1,4-Dimethoxy-13b,13c-diphenyl-6*H*,13*H*-5,7,12,13b,13c,14-hexahydro-5a,6a,12a,13a-tetraazabenz[5,6]azuleno-[2,1,8-*ija*]benz[*f*]azulene-6,13-dione chloroform solvate

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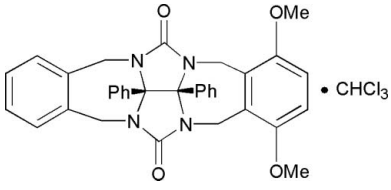
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.006 Å; R factor = 0.063; wR factor = 0.154; data-to-parameter ratio = 13.6.

The title compound, $C_{34}H_{30}N_4O_4\cdot CHCl_3$, is an example of a diphenylglycoluril-derived molecular clip. It forms dimeric aggregates in the crystal structure through intermolecular C—H···O interactions. A tape-like supramolecular packing motif occurs in the crystal structure *via* intermolecular C—H···O contacts between adjacent dimers. The $CHCl_3$ solvent molecules occupy voids in the crystal structure.

Related literature

For related literature, see: Chen *et al.* (2007); Hof *et al.* (2002); Hu *et al.*, (2007); Sijbesma *et al.*, (1993). For analysis, see: Spek (2003). For synthesis, see: Wang *et al.* (2006).



Experimental

Crystal data

$C_{34}H_{30}N_4O_4\cdot CHCl_3$
M_r = 677.99
Triclinic, $P\overline{1}$
a = 11.0635 (17) Å
b = 12.5197 (19) Å
c = 14.174 (2) Å
α = 64.652 (3)°
β = 87.539 (3)°
γ = 68.087 (3)°
V = 1630.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 292 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
8674 measured reflections
5658 independent reflections
3004 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.154
S = 0.96
5658 reflections
417 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C29—H29···O2^{i}	0.93	2.52	3.399 (4)	158
C4—H4···O3^{ii}	0.93	2.55	3.396 (5)	151

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

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2212).

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supplementary materials

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1,4-Dimethoxy-13b,13c-diphenyl-6*H*,13*H*-5,7,12,13b,13c,14-hexahydro-5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-6,13-dione chloroform solvate

Y.-Z. Wang, Z.-G. Wang and L. Li

Comment

Molecular clips based on the glycoluril skeleton, which have a well defined geometry due to the rigidity of the fused rings, have been prepared for a wide variety of supramolecular applications, including molecular recognition (Hu *et al.*, 2007), molecular assemblies (Hof *et al.*, 2002), crystal engineering (Wang *et al.*, 2006; Chen *et al.*, 2007), *etc*.

The title compound (I), Fig. 1, based on the concave molecule diphenylglycoluril, which has two aromatic side-walls of the cleft and two urea carbonyl groups, possesses potential binding features to dihydroxy-substituted aromatic molecules by π-π stacking and hydrogen bonds (Sijbesma *et al.*, 1993). Compound (I) forms dimeric aggregates through intermolecular C29—H29···O2 interactions with centrosymmetrically related molecules (Fig. 2 & Table 1). A tape-like packing motif is formed *via* complementary intermolecular C4—H4···O3 hydrogen bonds between adjacent dimers (Fig. 3 & Table 1). The solvent $CHCl_3$ molecules occupy voids in the crystal structure.

Experimental

Compound (I) was synthesized according to literature procedure (Wang *et al.*, 2006) in 42% isolated yield. Crystals appropriate for data collection were obtained by slow evaporation of CH_3OH:MeOH=20:1 (*v*/*v*) solution held at room temperature.

Refinement

All H-atoms were included in the riding model approximation with with N—H = 0.86Å and C—H = 0.93 − 98 Å, and with U_{iso}(H) = 1.2U_{eq}(C, N) and U_{iso}(methyl-H) = 1.5U_{eq}(methyl-C).

Figures

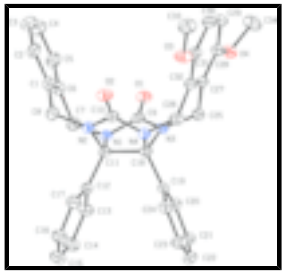


Fig. 1. The molecular structure of (I), showing the atom-labelling scheme and displacement ellipsoids at the 20° probability level. All hydrogen atoms and solvent $CHCl_3$ molecules are omitted for clarity.

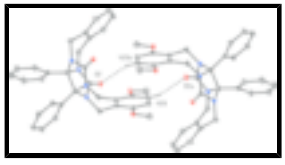


Fig. 2. Dimeric aggregates in (I) mediated by C—H···O interactions, shown as dashed bonds. Solvent $CHCl_3$ molecules and most hydrogen atoms are omitted for clarity.

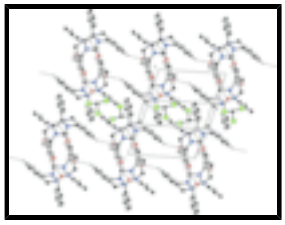


Fig. 3. Crystal packing in (I). The C—H···O interactions are shown as dashed bonds. Hydrogen atoms are omitted for clarity.

1,4-Dimethoxy-13*b*,l3c-diphenyl-6H,13*H*-5,7,12,13*b*,13*c*,14-Hexahydro- 5a,6a,l2a,13*a*-tetraazabenz[5,6]azuleno[2,1,8-ija]benz[*f*]azulene-6,13-dione chloroform solvate

Crystal data

$C_{34}H_{30}N_4O_4{\cdot}CHCl_3$	$Z = 2$
$M_r = 677.99$	$F_{000} = 704$
Triclinic, $P\overline{1}$	$D_x = 1.381$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.0635$ (17) Å	Cell parameters from 1381 reflections
$b = 12.5197$ (19) Å	$\theta = 2.1$–24.8°
$c = 14.174$ (2) Å	$\mu = 0.33$ mm^{-1}
$\alpha = 64.652$ (3)°	$T = 292$ (2) K
$\beta = 87.539$ (3)°	Block, colorless
$\gamma = 68.087$ (3)°	0.40 × 0.30 × 0.20 mm
$V = 1630.0$ (4) Å^3	

Data collection

Bruker SMART 4K CCD area detector diffractometer	3004 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.058$
Monochromator: graphite	$\theta_{max} = 25.0^\circ$
$T = 292(2)$ K	$\theta_{min} = 1.9^\circ$
φ and ω scans	$h = -13\rightarrow11$
Absorption correction: none	$k = -14\rightarrow14$
8674 measured reflections	$l = -14\rightarrow16$
5658 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
$R[F^2 > 2\sigma(F^2)] = 0.063$	H-atom parameters constrained
$wR(F^2) = 0.154$	?
$S = 0.96$	$(\Delta/\sigma)_{max} = <0.001$
5658 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
417 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}

Primary atom site location: structure-invariant direct methods

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	−0.1406 (3)	0.6430 (3)	0.2839 (3)	0.0540 (10)
C2	−0.2244 (4)	0.7493 (4)	0.2984 (3)	0.0687 (12)
H2	−0.1961	0.7707	0.3462	0.082*
C3	−0.3486 (4)	0.8218 (4)	0.2420 (4)	0.0821 (14)
H3	−0.4044	0.8915	0.2523	0.098*
C4	−0.3906 (4)	0.7918 (4)	0.1707 (4)	0.0844 (14)
H4	−0.4749	0.8409	0.1331	0.101*
C5	−0.3085 (4)	0.6895 (4)	0.1545 (3)	0.0713 (12)
H5	−0.3374	0.6709	0.1049	0.086*
C6	−0.1831 (4)	0.6132 (3)	0.2109 (3)	0.0544 (10)
C7	−0.0980 (3)	0.4973 (3)	0.1953 (3)	0.0565 (10)
H7A	−0.0833	0.4206	0.2606	0.068*
H7B	−0.1456	0.4928	0.1420	0.068*
C8	−0.0070 (4)	0.5620 (3)	0.3503 (3)	0.0555 (10)
H8A	0.0039	0.5998	0.3948	0.067*
H8B	−0.0042	0.4770	0.3961	0.067*
C9	0.0355 (3)	0.5824 (3)	0.0637 (3)	0.0420 (8)
C10	0.1732 (3)	0.6231 (3)	0.2662 (3)	0.0452 (9)
C11	0.1362 (3)	0.4639 (3)	0.2390 (2)	0.0406 (8)
C12	0.1906 (3)	0.3242 (3)	0.3196 (3)	0.0436 (9)
C13	0.1770 (4)	0.2316 (3)	0.2978 (3)	0.0598 (11)
H13	0.1294	0.2554	0.2345	0.072*
C14	0.2344 (4)	0.1027 (4)	0.3703 (4)	0.0774 (13)
H14	0.2245	0.0404	0.3559	0.093*
C15	0.3064 (5)	0.0674 (4)	0.4640 (4)	0.0823 (14)
H15	0.3451	−0.0189	0.5123	0.099*
C16	0.3207 (4)	0.1582 (4)	0.4856 (3)	0.0733 (13)
H16	0.3696	0.1342	0.5483	0.088*
C17	0.2622 (4)	0.2863 (4)	0.4142 (3)	0.0594 (11)
H17	0.2711	0.3482	0.4298	0.071*
C18	0.2372 (3)	0.5091 (3)	0.1657 (2)	0.0401 (8)

C19	0.3615 (3)	0.4004 (3)	0.1684 (2)	0.0411 (8)
C20	0.3570 (3)	0.3353 (3)	0.1123 (3)	0.0519 (10)
H20	0.2804	0.3632	0.0685	0.062*
C21	0.4653 (4)	0.2286 (3)	0.1202 (3)	0.0627 (11)
H21	0.4615	0.1854	0.0817	0.075*
C22	0.5776 (4)	0.1873 (4)	0.1850 (3)	0.0664 (12)
H22	0.6502	0.1155	0.1907	0.080*
C23	0.5843 (4)	0.2507 (4)	0.2414 (3)	0.0684 (12)
H23	0.6612	0.2225	0.2850	0.082*
C24	0.4759 (3)	0.3570 (3)	0.2333 (3)	0.0570 (10)
H24	0.4802	0.3997	0.2722	0.068*
C25	0.2006 (3)	0.6642 (3)	−0.0291 (3)	0.0489 (9)
H25A	0.1522	0.6795	−0.0922	0.059*
H25B	0.2931	0.6181	−0.0294	0.059*
C26	0.3252 (3)	0.6787 (3)	0.1428 (3)	0.0521 (10)
H26A	0.4006	0.6331	0.1177	0.063*
H26B	0.3578	0.7036	0.1893	0.063*
C27	0.1817 (3)	0.7925 (3)	−0.0334 (3)	0.0484 (9)
C28	0.1085 (3)	0.9054 (3)	−0.1229 (3)	0.0502 (9)
C29	0.0892 (4)	1.0230 (3)	−0.1281 (3)	0.0596 (10)
H29	0.0395	1.0979	−0.1871	0.071*
C30	0.1428 (4)	1.0303 (3)	−0.0468 (3)	0.0611 (11)
H30	0.1286	1.1105	−0.0512	0.073*
C31	0.2175 (3)	0.9207 (3)	0.0415 (3)	0.0513 (10)
C32	0.2371 (3)	0.7995 (3)	0.0491 (3)	0.0473 (9)
C33	0.2590 (4)	1.0407 (4)	0.1199 (4)	0.0815 (14)
H33A	0.2920	1.0864	0.0584	0.122*
H33B	0.3065	1.0272	0.1818	0.122*
H33C	0.1672	1.0900	0.1158	0.122*
C34	0.0162 (5)	0.9993 (4)	−0.3025 (3)	0.0882 (15)
H34A	−0.0609	1.0639	−0.2970	0.132*
H34B	−0.0053	0.9755	−0.3536	0.132*
H34C	0.0832	1.0327	−0.3243	0.132*
C35	0.2834 (5)	0.6779 (4)	0.4960 (3)	0.0879 (15)
H35	0.2463	0.6870	0.4302	0.105*
Cl1	0.3533 (2)	0.51797 (14)	0.58193 (17)	0.1871 (10)
Cl2	0.40018 (18)	0.74442 (14)	0.46850 (15)	0.1407 (7)
Cl3	0.15721 (19)	0.75913 (16)	0.54826 (12)	0.1523 (7)
N1	0.0289 (3)	0.4966 (2)	0.1636 (2)	0.0416 (7)
N2	0.1022 (3)	0.5492 (2)	0.2900 (2)	0.0452 (7)
N3	0.1577 (3)	0.5838 (2)	0.0626 (2)	0.0405 (7)
N4	0.2639 (3)	0.5902 (2)	0.2043 (2)	0.0434 (7)
O1	−0.0526 (2)	0.6432 (2)	−0.01028 (18)	0.0539 (7)
O2	0.1596 (2)	0.7038 (2)	0.29762 (19)	0.0608 (7)
O3	0.2754 (3)	0.9211 (2)	0.1251 (2)	0.0706 (8)
O4	0.0629 (3)	0.8893 (2)	−0.2027 (2)	0.0675 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.048 (2)	0.054 (2)	0.057 (2)	−0.0174 (19)	0.0174 (19)	−0.025 (2)
C2	0.068 (3)	0.067 (3)	0.080 (3)	−0.025 (2)	0.031 (2)	−0.043 (2)
C3	0.063 (3)	0.065 (3)	0.105 (4)	−0.007 (2)	0.024 (3)	−0.042 (3)
C4	0.050 (3)	0.078 (3)	0.100 (4)	−0.006 (2)	0.014 (3)	−0.034 (3)
C5	0.049 (3)	0.081 (3)	0.078 (3)	−0.018 (2)	0.009 (2)	−0.036 (3)
C6	0.049 (2)	0.056 (2)	0.057 (2)	−0.0184 (19)	0.0111 (19)	−0.026 (2)
C7	0.049 (2)	0.059 (2)	0.062 (3)	−0.0190 (19)	0.0070 (19)	−0.028 (2)
C8	0.068 (3)	0.057 (2)	0.048 (2)	−0.022 (2)	0.014 (2)	−0.031 (2)
C9	0.044 (2)	0.038 (2)	0.046 (2)	−0.0108 (17)	0.0027 (18)	−0.0245 (19)
C10	0.047 (2)	0.040 (2)	0.042 (2)	−0.0078 (17)	−0.0065 (17)	−0.0199 (18)
C11	0.042 (2)	0.0355 (19)	0.040 (2)	−0.0080 (15)	0.0004 (16)	−0.0190 (17)
C12	0.048 (2)	0.039 (2)	0.043 (2)	−0.0152 (16)	0.0091 (17)	−0.0195 (18)
C13	0.068 (3)	0.045 (2)	0.062 (3)	−0.018 (2)	0.001 (2)	−0.023 (2)
C14	0.102 (4)	0.047 (3)	0.084 (3)	−0.028 (2)	0.012 (3)	−0.030 (3)
C15	0.098 (4)	0.047 (3)	0.066 (3)	−0.011 (2)	0.008 (3)	−0.008 (2)
C16	0.082 (3)	0.057 (3)	0.052 (3)	−0.007 (2)	0.000 (2)	−0.016 (2)
C17	0.068 (3)	0.050 (2)	0.053 (3)	−0.015 (2)	0.002 (2)	−0.023 (2)
C18	0.042 (2)	0.0372 (19)	0.041 (2)	−0.0112 (16)	0.0001 (16)	−0.0197 (17)
C19	0.041 (2)	0.0365 (19)	0.040 (2)	−0.0117 (16)	0.0062 (16)	−0.0145 (17)
C20	0.046 (2)	0.048 (2)	0.061 (2)	−0.0104 (18)	0.0046 (18)	−0.031 (2)
C21	0.061 (3)	0.054 (2)	0.075 (3)	−0.015 (2)	0.014 (2)	−0.038 (2)
C22	0.052 (3)	0.050 (2)	0.083 (3)	−0.009 (2)	0.020 (2)	−0.027 (2)
C23	0.045 (2)	0.060 (3)	0.079 (3)	−0.004 (2)	−0.004 (2)	−0.026 (2)
C24	0.055 (2)	0.050 (2)	0.060 (3)	−0.013 (2)	−0.001 (2)	−0.024 (2)
C25	0.053 (2)	0.042 (2)	0.041 (2)	−0.0122 (17)	0.0035 (17)	−0.0150 (18)
C26	0.049 (2)	0.044 (2)	0.064 (3)	−0.0183 (18)	0.0001 (19)	−0.024 (2)
C27	0.040 (2)	0.036 (2)	0.059 (2)	−0.0094 (16)	0.0103 (18)	−0.0176 (19)
C28	0.047 (2)	0.043 (2)	0.050 (2)	−0.0130 (18)	0.0029 (18)	−0.016 (2)
C29	0.053 (2)	0.038 (2)	0.066 (3)	−0.0108 (18)	0.008 (2)	−0.011 (2)
C30	0.050 (2)	0.037 (2)	0.087 (3)	−0.0137 (19)	0.009 (2)	−0.023 (2)
C31	0.042 (2)	0.040 (2)	0.073 (3)	−0.0161 (18)	0.0055 (19)	−0.025 (2)
C32	0.040 (2)	0.037 (2)	0.061 (3)	−0.0127 (16)	0.0070 (18)	−0.0203 (19)
C33	0.088 (3)	0.061 (3)	0.116 (4)	−0.032 (2)	0.012 (3)	−0.054 (3)
C34	0.101 (4)	0.070 (3)	0.058 (3)	−0.019 (3)	−0.019 (2)	−0.006 (3)
C35	0.125 (4)	0.081 (3)	0.070 (3)	−0.053 (3)	0.001 (3)	−0.033 (3)
Cl1	0.246 (2)	0.0731 (11)	0.1969 (19)	−0.0633 (12)	0.0846 (17)	−0.0248 (12)
Cl2	0.1482 (15)	0.0909 (10)	0.1857 (17)	−0.0635 (10)	0.0059 (12)	−0.0484 (11)
Cl3	0.1754 (17)	0.1303 (13)	0.0933 (11)	−0.0199 (12)	0.0216 (11)	−0.0338 (10)
N1	0.0408 (17)	0.0408 (17)	0.0416 (18)	−0.0125 (13)	0.0013 (13)	−0.0194 (15)
N2	0.0488 (18)	0.0443 (18)	0.0478 (18)	−0.0146 (14)	0.0071 (14)	−0.0284 (15)
N3	0.0421 (17)	0.0361 (16)	0.0374 (17)	−0.0116 (13)	−0.0025 (13)	−0.0136 (14)
N4	0.0451 (17)	0.0398 (17)	0.0474 (18)	−0.0141 (14)	−0.0007 (14)	−0.0227 (15)
O1	0.0511 (15)	0.0494 (15)	0.0527 (16)	−0.0088 (12)	−0.0118 (13)	−0.0228 (13)
O2	0.0724 (18)	0.0496 (15)	0.0684 (18)	−0.0155 (13)	−0.0013 (13)	−0.0393 (14)

O3	0.0702 (19)	0.0482 (16)	0.098 (2)	−0.0208 (13)	−0.0085 (16)	−0.0365 (16)
O4	0.0759 (19)	0.0501 (16)	0.0529 (17)	−0.0155 (14)	−0.0068 (14)	−0.0088 (14)

Geometric parameters (Å, °)

C1—C6	1.391 (5)	C19—C20	1.375 (4)
C1—C2	1.403 (5)	C19—C24	1.379 (4)
C1—C8	1.510 (5)	C20—C21	1.386 (4)
C2—C3	1.376 (6)	C20—H20	0.9300
C2—H2	0.9300	C21—C22	1.367 (5)
C3—C4	1.368 (6)	C21—H21	0.9300
C3—H3	0.9300	C22—C23	1.366 (5)
C4—C5	1.373 (5)	C22—H22	0.9300
C4—H4	0.9300	C23—C24	1.383 (5)
C5—C6	1.389 (5)	C23—H23	0.9300
C5—H5	0.9300	C24—H24	0.9300
C6—C7	1.510 (5)	C25—N3	1.454 (4)
C7—N1	1.454 (4)	C25—C27	1.514 (4)
C7—H7A	0.9700	C25—H25A	0.9700
C7—H7B	0.9700	C25—H25B	0.9700
C8—N2	1.450 (4)	C26—N4	1.459 (4)
C8—H8A	0.9700	C26—C32	1.514 (4)
C8—H8B	0.9700	C26—H26A	0.9700
C9—O1	1.217 (4)	C26—H26B	0.9700
C9—N3	1.358 (4)	C27—C32	1.394 (5)
C9—N1	1.382 (4)	C27—C28	1.400 (4)
C10—O2	1.229 (4)	C28—C29	1.375 (5)
C10—N2	1.356 (4)	C28—O4	1.377 (4)
C10—N4	1.368 (4)	C29—C30	1.370 (5)
C11—N1	1.445 (4)	C29—H29	0.9300
C11—N2	1.464 (4)	C30—C31	1.380 (5)
C11—C12	1.514 (4)	C30—H30	0.9300
C11—C18	1.587 (4)	C31—O3	1.374 (4)
C12—C13	1.380 (4)	C31—C32	1.406 (4)
C12—C17	1.384 (4)	C33—O3	1.408 (4)
C13—C14	1.391 (5)	C33—H33A	0.9600
C13—H13	0.9300	C33—H33B	0.9600
C14—C15	1.385 (6)	C33—H33C	0.9600
C14—H14	0.9300	C34—O4	1.425 (4)
C15—C16	1.360 (6)	C34—H34A	0.9600
C15—H15	0.9300	C34—H34B	0.9600
C16—C17	1.381 (5)	C34—H34C	0.9600
C16—H16	0.9300	C35—Cl1	1.711 (4)
C17—H17	0.9300	C35—Cl2	1.731 (5)
C18—N3	1.452 (4)	C35—Cl3	1.740 (5)
C18—N4	1.460 (4)	C35—H35	0.9800
C18—C19	1.519 (4)		
C6—C1—C2	119.6 (4)	C22—C21—H21	120.2
C6—C1—C8	121.7 (3)	C20—C21—H21	120.2

C2—C1—C8	118.8 (3)	C23—C22—C21	120.6 (3)
C3—C2—C1	120.0 (4)	C23—C22—H22	119.7
C3—C2—H2	120.0	C21—C22—H22	119.7
C1—C2—H2	120.0	C22—C23—C24	119.6 (4)
C4—C3—C2	120.4 (4)	C22—C23—H23	120.2
C4—C3—H3	119.8	C24—C23—H23	120.2
C2—C3—H3	119.8	C19—C24—C23	120.8 (3)
C3—C4—C5	120.1 (4)	C19—C24—H24	119.6
C3—C4—H4	119.9	C23—C24—H24	119.6
C5—C4—H4	119.9	N3—C25—C27	113.9 (3)
C4—C5—C6	121.1 (4)	N3—C25—H25A	108.8
C4—C5—H5	119.4	C27—C25—H25A	108.8
C6—C5—H5	119.4	N3—C25—H25B	108.8
C5—C6—C1	118.8 (3)	C27—C25—H25B	108.8
C5—C6—C7	119.7 (3)	H25A—C25—H25B	107.7
C1—C6—C7	121.4 (3)	N4—C26—C32	115.6 (3)
N1—C7—C6	114.7 (3)	N4—C26—H26A	108.4
N1—C7—H7A	108.6	C32—C26—H26A	108.4
C6—C7—H7A	108.6	N4—C26—H26B	108.4
N1—C7—H7B	108.6	C32—C26—H26B	108.4
C6—C7—H7B	108.6	H26A—C26—H26B	107.4
H7A—C7—H7B	107.6	C32—C27—C28	119.9 (3)
N2—C8—C1	114.3 (3)	C32—C27—C25	121.2 (3)
N2—C8—H8A	108.7	C28—C27—C25	119.0 (3)
C1—C8—H8A	108.7	C29—C28—O4	124.1 (3)
N2—C8—H8B	108.7	C29—C28—C27	119.9 (4)
C1—C8—H8B	108.7	O4—C28—C27	115.9 (3)
H8A—C8—H8B	107.6	C30—C29—C28	120.4 (4)
O1—C9—N3	126.6 (3)	C30—C29—H29	119.8
O1—C9—N1	125.4 (3)	C28—C29—H29	119.8
N3—C9—N1	108.0 (3)	C29—C30—C31	121.1 (3)
O2—C10—N2	125.1 (3)	C29—C30—H30	119.5
O2—C10—N4	126.0 (4)	C31—C30—H30	119.5
N2—C10—N4	108.8 (3)	O3—C31—C30	124.1 (3)
N1—C11—N2	112.3 (2)	O3—C31—C32	116.4 (3)
N1—C11—C12	113.4 (3)	C30—C31—C32	119.5 (4)
N2—C11—C12	111.2 (3)	C27—C32—C31	119.2 (3)
N1—C11—C18	102.7 (3)	C27—C32—C26	120.8 (3)
N2—C11—C18	101.2 (3)	C31—C32—C26	119.9 (3)
C12—C11—C18	115.2 (2)	O3—C33—H33A	109.5
C13—C12—C17	118.9 (3)	O3—C33—H33B	109.5
C13—C12—C11	120.8 (3)	H33A—C33—H33B	109.5
C17—C12—C11	120.1 (3)	O3—C33—H33C	109.5
C12—C13—C14	120.0 (4)	H33A—C33—H33C	109.5
C12—C13—H13	120.0	H33B—C33—H33C	109.5
C14—C13—H13	120.0	O4—C34—H34A	109.5
C15—C14—C13	119.9 (4)	O4—C34—H34B	109.5
C15—C14—H14	120.0	H34A—C34—H34B	109.5
C13—C14—H14	120.0	O4—C34—H34C	109.5

C16—C15—C14	120.2 (4)	H34A—C34—H34C	109.5
C16—C15—H15	119.9	H34B—C34—H34C	109.5
C14—C15—H15	119.9	Cl1—C35—Cl2	110.7 (3)
C15—C16—C17	119.8 (4)	Cl1—C35—Cl3	110.3 (3)
C15—C16—H16	120.1	Cl2—C35—Cl3	110.0 (2)
C17—C16—H16	120.1	Cl1—C35—H35	108.6
C16—C17—C12	121.1 (4)	Cl2—C35—H35	108.6
C16—C17—H17	119.4	Cl3—C35—H35	108.6
C12—C17—H17	119.4	C9—N1—C11	111.5 (3)
N3—C18—N4	112.0 (2)	C9—N1—C7	119.9 (3)
N3—C18—C19	112.2 (2)	C11—N1—C7	121.2 (3)
N4—C18—C19	112.6 (3)	C10—N2—C8	124.0 (3)
N3—C18—C11	101.7 (2)	C10—N2—C11	113.5 (3)
N4—C18—C11	102.8 (2)	C8—N2—C11	122.1 (3)
C19—C18—C11	114.7 (3)	C9—N3—C18	113.5 (3)
C20—C19—C24	118.6 (3)	C9—N3—C25	123.8 (3)
C20—C19—C18	119.2 (3)	C18—N3—C25	122.3 (3)
C24—C19—C18	121.9 (3)	C10—N4—C26	119.2 (3)
C19—C20—C21	120.8 (3)	C10—N4—C18	111.5 (3)
C19—C20—H20	119.6	C26—N4—C18	121.5 (3)
C21—C20—H20	119.6	C31—O3—C33	118.0 (3)
C22—C21—C20	119.6 (3)	C28—O4—C34	116.5 (3)
C6—C1—C2—C3	1.1 (6)	C28—C27—C32—C31	0.6 (5)
C8—C1—C2—C3	−177.5 (4)	C25—C27—C32—C31	179.7 (3)
C1—C2—C3—C4	−0.7 (7)	C28—C27—C32—C26	−175.3 (3)
C2—C3—C4—C5	−0.4 (7)	C25—C27—C32—C26	3.8 (5)
C3—C4—C5—C6	1.2 (7)	O3—C31—C32—C27	−179.6 (3)
C4—C5—C6—C1	−0.8 (6)	C30—C31—C32—C27	0.6 (5)
C4—C5—C6—C7	176.8 (4)	O3—C31—C32—C26	−3.7 (5)
C2—C1—C6—C5	−0.4 (6)	C30—C31—C32—C26	176.5 (3)
C8—C1—C6—C5	178.2 (4)	N4—C26—C32—C27	−61.4 (4)
C2—C1—C6—C7	−177.9 (3)	N4—C26—C32—C31	122.8 (3)
C8—C1—C6—C7	0.6 (6)	O1—C9—N1—C11	−166.3 (3)
C5—C6—C7—N1	123.4 (4)	N3—C9—N1—C11	15.2 (3)
C1—C6—C7—N1	−59.1 (5)	O1—C9—N1—C7	−16.0 (5)
C6—C1—C8—N2	57.4 (5)	N3—C9—N1—C7	165.5 (3)
C2—C1—C8—N2	−124.0 (4)	N2—C11—N1—C9	91.2 (3)
N1—C11—C12—C13	24.4 (4)	C12—C11—N1—C9	−141.7 (3)
N2—C11—C12—C13	152.1 (3)	C18—C11—N1—C9	−16.7 (3)
C18—C11—C12—C13	−93.5 (4)	N2—C11—N1—C7	−58.7 (4)
N1—C11—C12—C17	−159.7 (3)	C12—C11—N1—C7	68.4 (3)
N2—C11—C12—C17	−32.1 (4)	C18—C11—N1—C7	−166.6 (3)
C18—C11—C12—C17	82.3 (4)	C6—C7—N1—C9	−70.3 (4)
C17—C12—C13—C14	0.2 (5)	C6—C7—N1—C11	77.1 (4)
C11—C12—C13—C14	176.1 (3)	O2—C10—N2—C8	4.8 (5)
C12—C13—C14—C15	−0.7 (6)	N4—C10—N2—C8	−176.9 (3)
C13—C14—C15—C16	0.3 (7)	O2—C10—N2—C11	178.1 (3)
C14—C15—C16—C17	0.5 (7)	N4—C10—N2—C11	−3.6 (3)
C15—C16—C17—C12	−1.0 (6)	C1—C8—N2—C10	96.5 (4)

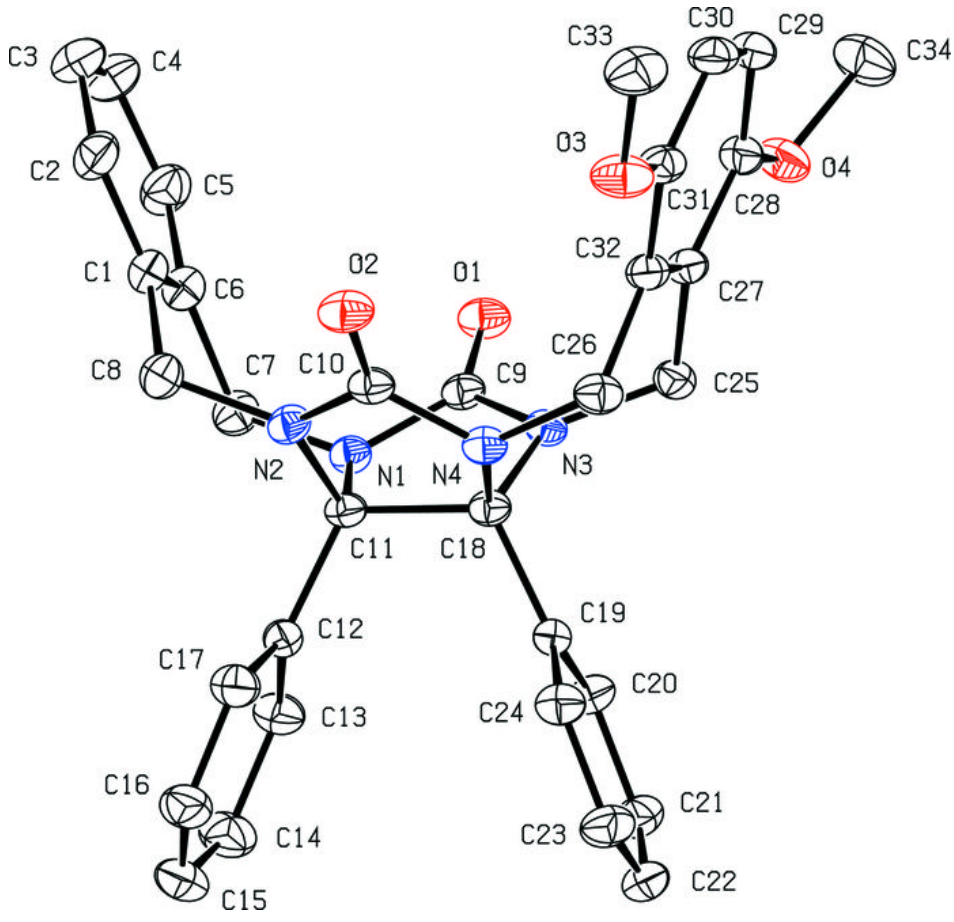
C13—C12—C17—C16	0.6 (6)	C1—C8—N2—C11	−76.3 (4)
C11—C12—C17—C16	−175.3 (3)	N1—C11—N2—C10	−114.4 (3)
N1—C11—C18—N3	11.7 (3)	C12—C11—N2—C10	117.4 (3)
N2—C11—C18—N3	−104.4 (2)	C18—C11—N2—C10	−5.5 (3)
C12—C11—C18—N3	135.5 (3)	N1—C11—N2—C8	59.1 (4)
N1—C11—C18—N4	127.8 (2)	C12—C11—N2—C8	−69.1 (4)
N2—C11—C18—N4	11.7 (3)	C18—C11—N2—C8	168.0 (3)
C12—C11—C18—N4	−108.4 (3)	O1—C9—N3—C18	175.1 (3)
N1—C11—C18—C19	−109.6 (3)	N1—C9—N3—C18	−6.5 (3)
N2—C11—C18—C19	134.3 (3)	O1—C9—N3—C25	2.4 (5)
C12—C11—C18—C19	14.2 (4)	N1—C9—N3—C25	−179.2 (2)
N3—C18—C19—C20	−37.1 (4)	N4—C18—N3—C9	−112.9 (3)
N4—C18—C19—C20	−164.6 (3)	C19—C18—N3—C9	119.3 (3)
C11—C18—C19—C20	78.3 (4)	C11—C18—N3—C9	−3.7 (3)
N3—C18—C19—C24	149.1 (3)	N4—C18—N3—C25	60.0 (4)
N4—C18—C19—C24	21.7 (5)	C19—C18—N3—C25	−67.8 (4)
C11—C18—C19—C24	−95.4 (4)	C11—C18—N3—C25	169.1 (2)
C24—C19—C20—C21	−0.4 (5)	C27—C25—N3—C9	92.8 (4)
C18—C19—C20—C21	−174.3 (3)	C27—C25—N3—C18	−79.3 (4)
C19—C20—C21—C22	0.3 (6)	O2—C10—N4—C26	−19.7 (5)
C20—C21—C22—C23	−0.3 (6)	N2—C10—N4—C26	162.0 (3)
C21—C22—C23—C24	0.4 (6)	O2—C10—N4—C18	−169.4 (3)
C20—C19—C24—C23	0.5 (6)	N2—C10—N4—C18	12.3 (3)
C18—C19—C24—C23	174.2 (3)	C32—C26—N4—C10	−71.8 (4)
C22—C23—C24—C19	−0.5 (6)	C32—C26—N4—C18	74.8 (4)
N3—C25—C27—C32	56.4 (4)	N3—C18—N4—C10	93.4 (3)
N3—C25—C27—C28	−124.5 (3)	C19—C18—N4—C10	−139.0 (3)
C32—C27—C28—C29	−1.4 (5)	C11—C18—N4—C10	−15.0 (3)
C25—C27—C28—C29	179.5 (3)	N3—C18—N4—C26	−55.5 (4)
C32—C27—C28—O4	176.9 (3)	C19—C18—N4—C26	72.1 (4)
C25—C27—C28—O4	−2.2 (5)	C11—C18—N4—C26	−164.0 (3)
O4—C28—C29—C30	−177.2 (3)	C30—C31—O3—C33	−0.9 (5)
C27—C28—C29—C30	0.9 (5)	C32—C31—O3—C33	179.4 (3)
C28—C29—C30—C31	0.3 (6)	C29—C28—O4—C34	12.6 (5)
C29—C30—C31—O3	179.1 (3)	C27—C28—O4—C34	−165.6 (3)
C29—C30—C31—C32	−1.1 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C29—H29···O2^{i}	0.93	2.52	3.399 (4)	158
C4—H4···O3^{ii}	0.93	2.55	3.396 (5)	151

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

Fig. 1


C3
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C11
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Fig. 2

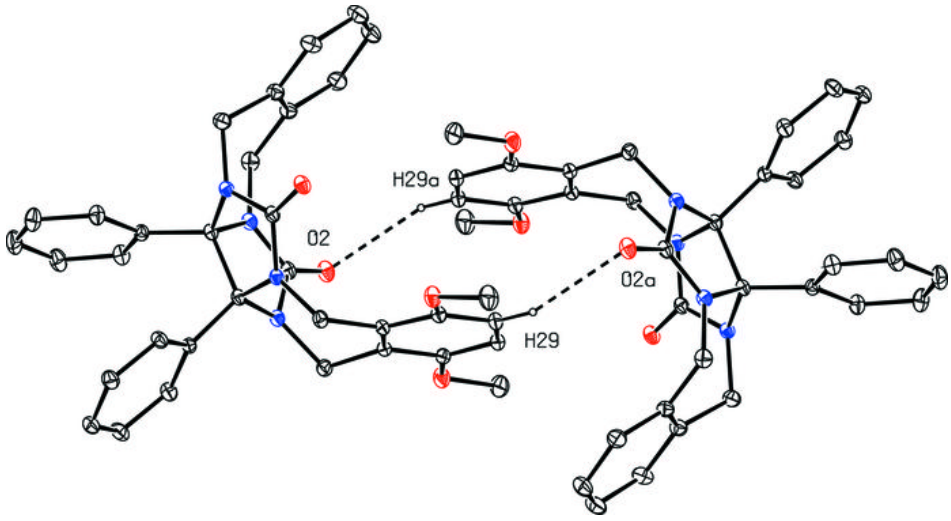

Fig. 3