

The highly saddled octaethyltetraphenylporphyrin-[H₄OETPP] dichloride toluene 1.33-solvate

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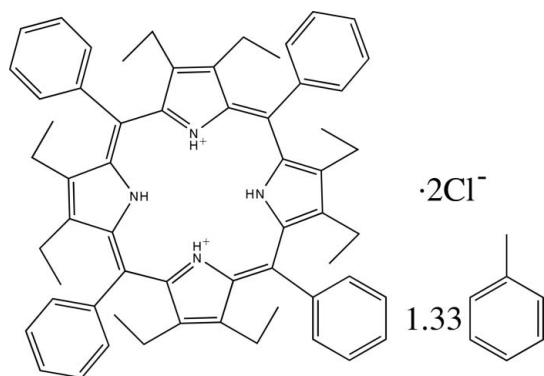
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.046; wR factor = 0.139; data-to-parameter ratio = 16.3.

The title compound, 2,3,7,8,12,13,17,18-octaethyl-5,10,15,20-tetraphenylporphyrin dichloride toluene 1.33-solvate, $\text{C}_{60}\text{H}_{64}\text{N}_4^{2+} \cdot 2\text{Cl}^- \cdot 1.33\text{C}_7\text{H}_8$, displays a highly S_4 -saddled porphyrin core with the $C\beta$ atoms displaced 1.23 (9) Å out of the N_4 plane. All N atoms are protonated and form hydrogen bonds [$\text{N} \cdots \text{Cl} = 3.206$ (1) Å] with chloride anions. An S_4 symmetry axis passes through the porphyrin center. The phenyl group of the porphyrin is disordered over two sites, with occupancies approximately 0.6:0.4.

Related literature

For related literature, see: Adler *et al.* (1970); Barkigia *et al.* (1988, 1995); Regev *et al.* (1994); Senge (1992); Senge & Kalisch (1999); Senge *et al.* (1994).



Experimental

Crystal data

$\text{C}_{60}\text{H}_{64}\text{N}_4^{2+} \cdot 2\text{Cl}^- \cdot 1.33\text{C}_7\text{H}_8$
 $M_r = 1034.90$
 Cubic, $I\bar{4}3d$
 $a = 25.8135$ (2) Å
 $V = 17200.5$ (2) Å³

$Z = 12$
 Mo $K\alpha$ radiation
 $\mu = 0.16$ mm⁻¹
 $T = 100$ (2) K
 $0.42 \times 0.32 \times 0.29$ mm

Data collection

Bruker APEX II CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.936$, $T_{\max} = 0.955$

176336 measured reflections
 4414 independent reflections
 3859 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.139$
 $S = 1.15$
 4414 reflections
 271 parameters
 42 restraints

H-atom parameters constrained
 $\Delta\rho_{\max} = 0.42$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³
 Absolute structure: Flack (1983),
 2073 Friedel pairs
 Flack parameter: -0.07 (7)

Data collection: APEX2 (Bruker–Nonius, 2004); cell refinement: APEX2 and SAINT (Bruker–Nonius, 2004); data reduction: SAINT and XPREP (Sheldrick, 2005); program(s) used to solve structure: XS (Sheldrick, 2001); program(s) used to refine structure: XL (Sheldrick, 2001); molecular graphics: XP (Sheldrick, 1998) and PLATON (Spek, 2003); 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: PK2027).

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supplementary materials

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The highly saddled octaethyltetraphenylporphyrin-[H₄OETPP] dichloride toluene 1.33-solvate

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Comment

Different macrocycle conformations are believed to play an important role in a variety of protein complexes. (Senge 1992; Barkigia *et al.* 1988) Porphyrins bearing large and sterically interacting substituents at the porphyrin periphery have been synthesized successfully to yield tetrapyrroles with significantly distorted macrocycles. For one such porphyrin, octaethyltetraphenylporphyrin (OETPP), its free base and diacid salts have been reported (Regev *et al.* 1994; Barkigia *et al.* 1995; Senge *et al.* 1994; Senge & Kalisch, 1999). We report herein the molecular structure of another diacid salt of OETPP as a different solvate, [H₄OETPP]·2 C₁(C₇H₈)_{1.33}.

Figure 1 shows the thermal-ellipsoid plot of the cation, H₄OETPP²⁺. This porphyrin has an S₄ symmetry axis through the porphyrin center. The asymmetric unit is one-fourth porphyrin. The neighboring pyrrole units are displaced pairwise above and below the mean plane. One of the most important features is the severe saddling of the porphyrin core. The displacements of the porphyrin core are also shown in Figure 1. The average displacement of the β-carbons from the four nitrogen mean plane is 1.23 (9) Å, which is similar to those in other diacid salts of OETPP (Barkigia *et al.* 1995; Senge *et al.* 1994; Senge & Kalisch, 1999). Saddling is also shown by the tilt of pyrrole rings away from porphyrin mean plane. In the title compound, the average of the dihedral angle between pyrrole and porphyrin mean plane is 38.25 (4)°.

The four pyrrole N atoms are protonated, they show alternating up- and down- displacements. As shown in Figure 2, two chloride anions are held at both sides of the porphyrin plane by hydrogen-bonds to pyrrole nitrogen. The Cl···N distance is 3.206 (1) Å, which is comparable with 3.15 and 3.23 Å in [H₄OETPP]·2 C₁-CH₂Cl₂ (Senge & Kalisch, 1999).

Experimental

During the metallation of H₂OETPP (Adler *et al.* 1970), the reaction solution was washed by 2 mol/L HCl solution. The resulting material was crystallized in toluene, which yielded [H₄OETPP]·2 C₁ crystals as an impurity.

Refinement

The structures were solved by direct methods using XS (Sheldrick, 2001) and refined against F² using XL (Sheldrick, 2001); subsequent difference Fourier syntheses led to the location of all the remaining non-hydrogen atoms. For the structure refinement all data were used, including negative intensities. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were idealized with the standard *SHELXTL* idealization methods. The program *SADABS* was applied for the absorption correction. The phenyl group of the porphyrin molecule is disordered over two positions. The final refinement gave the occupancy for the major component as 0.58 (4). The toluene solvate is disordered around a threefold axis, its occupancy is fixed as 0.33333 and the phenyl ring was refined as a rigid group. Anisotropic displacement parameters for atoms of these groups were refined by similar U^{ij} restraints (SIMU). Rounding errors create differences between reported formula and that calculated by checkCIF.

Figures

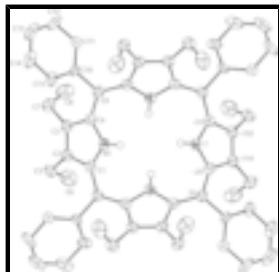


Fig. 1. The molecular structure showing 50% probability displacement ellipsoids. Only the major component of the phenyl group is displayed. The displacements of the atoms of the porphyrin core from the mean plane defined by the four nitrogen porphyrin atoms are also given in 0.01 Å.

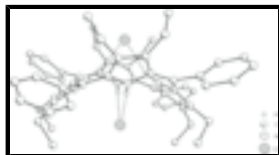


Fig. 2. Crystal structure of the title compound with hydrogen bonds as shown in dashed lines.

2,3,7,8,12,13,17,18-octaethyl-5,10,15,20-tetraphenylporphyrin dichloride toluene 1.33-solvate

Crystal data

$C_{60}H_{64}N_4^{2+} \cdot 2Cl^- \cdot 1.33C_7H_8$

$M_r = 1034.90$

Cubic, $I\bar{4}3d$

Hall symbol: I -4bd 2c 3

$a = 25.8135$ (2) Å

$b = 25.8135$ (2) Å

$c = 25.8135$ (2) Å

$\alpha = 90^\circ$

$\beta = 90^\circ$

$\gamma = 90^\circ$

$V = 17200.5$ (2) Å³

$Z = 12$

$F_{000} = 6632$

$D_x = 1.199$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9107 reflections

$\theta = 2.2$ – 27.0°

$\mu = 0.16$ mm⁻¹

$T = 100$ (2) K

Block, red

$0.42 \times 0.32 \times 0.29$ mm

Data collection

Bruker APEX II CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.33 pixels mm⁻¹

$T = 100$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2004)

$T_{\min} = 0.936$, $T_{\max} = 0.955$

176336 measured reflections

4414 independent reflections

3859 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\text{max}} = 30.6^\circ$

$\theta_{\text{min}} = 1.9^\circ$

$h = -36 \rightarrow 36$

$k = -36 \rightarrow 36$

$l = -36 \rightarrow 29$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 9.3007P]$
$wR(F^2) = 0.139$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{\max} = 0.003$
4414 reflections	$\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$
271 parameters	$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$
42 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 2073 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.07 (7)$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	0.5000	0.2500	0.02930 (2)	0.03694 (15)	
N1	0.53228 (5)	0.32591 (5)	0.12214 (5)	0.0251 (2)	
H1A	0.5196	0.3011	0.1025	0.030*	
C1	0.50375 (6)	0.36471 (6)	0.14469 (6)	0.0249 (3)	
C2	0.53873 (6)	0.39489 (6)	0.17608 (6)	0.0270 (3)	
C3	0.58772 (6)	0.37522 (6)	0.16901 (6)	0.0274 (3)	
C4	0.58334 (6)	0.33159 (6)	0.13465 (6)	0.0256 (3)	
C5	0.52423 (8)	0.43372 (7)	0.21691 (7)	0.0337 (3)	
H5A	0.5000	0.4594	0.2019	0.040*	
H5B	0.5557	0.4524	0.2284	0.040*	
C6	0.49915 (12)	0.40811 (10)	0.26289 (9)	0.0524 (6)	
H6A	0.4912	0.4343	0.2892	0.079*	
H6B	0.5228	0.3823	0.2775	0.079*	
H6C	0.4670	0.3911	0.2519	0.079*	
C7	0.63502 (7)	0.39025 (8)	0.19900 (8)	0.0370 (4)	
H7A	0.6305	0.4258	0.2128	0.044*	

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H7B	0.6654	0.3904	0.1755	0.044*	
C8	0.64522 (10)	0.35331 (11)	0.24360 (10)	0.0535 (6)	
H8A	0.6745	0.3661	0.2642	0.080*	
H8B	0.6534	0.3189	0.2299	0.080*	
H8C	0.6143	0.3511	0.2656	0.080*	
C9	0.62278 (6)	0.29898 (6)	0.11615 (6)	0.0274 (3)	
C10A	0.6751 (4)	0.3241 (7)	0.1054 (7)	0.026 (2)	0.58 (4)
C11A	0.7208 (5)	0.3005 (5)	0.1244 (5)	0.0376 (17)	0.58 (4)
H11A	0.7192	0.2695	0.1443	0.045*	0.58 (4)
C12A	0.7684 (4)	0.3239 (6)	0.1133 (4)	0.048 (3)	0.58 (4)
H12A	0.7994	0.3093	0.1269	0.057*	0.58 (4)
C13A	0.7710 (4)	0.3681 (6)	0.0827 (5)	0.052 (2)	0.58 (4)
H13A	0.8036	0.3831	0.0747	0.062*	0.58 (4)
C14A	0.7261 (5)	0.3903 (4)	0.0640 (4)	0.0441 (18)	0.58 (4)
H14A	0.7281	0.4205	0.0431	0.053*	0.58 (4)
C15A	0.6778 (6)	0.3686 (6)	0.0754 (5)	0.0331 (18)	0.58 (4)
H15A	0.6470	0.3843	0.0628	0.040*	0.58 (4)
C10B	0.6743 (7)	0.3166 (10)	0.1126 (10)	0.039 (5)	0.42 (4)
C11B	0.7144 (7)	0.2923 (9)	0.1366 (9)	0.050 (3)	0.42 (4)
H11B	0.7078	0.2648	0.1601	0.060*	0.42 (4)
C12B	0.7645 (7)	0.3075 (9)	0.1268 (11)	0.060 (4)	0.42 (4)
H12B	0.7918	0.2880	0.1419	0.072*	0.42 (4)
C13B	0.7765 (5)	0.3469 (12)	0.0981 (10)	0.067 (7)	0.42 (4)
H13B	0.8118	0.3558	0.0925	0.081*	0.42 (4)
C14B	0.7372 (9)	0.3762 (11)	0.0757 (7)	0.066 (5)	0.42 (4)
H14B	0.7447	0.4064	0.0561	0.079*	0.42 (4)
C15B	0.6847 (9)	0.3592 (10)	0.0833 (9)	0.046 (4)	0.42 (4)
H15B	0.6571	0.3779	0.0678	0.055*	0.42 (4)
C21	0.6692 (6)	0.1960 (7)	0.2840 (5)	0.080 (4)	0.33
C22	0.6698 (7)	0.2339 (6)	0.3206 (6)	0.092 (4)	0.33
H22A	0.6514	0.2649	0.3132	0.111*	0.33
C23	0.6957 (6)	0.2311 (6)	0.3687 (5)	0.080 (3)	0.33
H23A	0.6928	0.2582	0.3935	0.096*	0.33
C24	0.7247 (5)	0.1886 (6)	0.3782 (5)	0.089 (3)	0.33
H24A	0.7439	0.1857	0.4095	0.107*	0.33
C25	0.7261 (6)	0.1458 (7)	0.3383 (6)	0.094 (4)	0.33
H25A	0.7455	0.1151	0.3443	0.113*	0.33
C26	0.6988 (7)	0.1521 (6)	0.2935 (5)	0.079 (3)	0.33
H26A	0.7002	0.1257	0.2679	0.095*	0.33
C27	0.6384 (7)	0.1992 (6)	0.2345 (6)	0.111 (6)	0.33
H27A	0.6200	0.1665	0.2289	0.166*	0.33
H27B	0.6619	0.2058	0.2054	0.166*	0.33
H27C	0.6133	0.2276	0.2371	0.166*	0.33

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0396 (3)	0.0432 (3)	0.0280 (3)	-0.0041 (3)	0.000	0.000

N1	0.0245 (6)	0.0252 (6)	0.0257 (6)	-0.0014 (5)	-0.0018 (5)	-0.0030 (5)
C1	0.0275 (7)	0.0229 (6)	0.0242 (6)	0.0000 (5)	-0.0019 (5)	-0.0001 (5)
C2	0.0319 (7)	0.0240 (7)	0.0250 (7)	-0.0005 (6)	-0.0039 (5)	-0.0011 (5)
C3	0.0295 (7)	0.0265 (7)	0.0263 (7)	-0.0026 (6)	-0.0029 (6)	-0.0039 (6)
C4	0.0252 (7)	0.0257 (7)	0.0260 (7)	-0.0037 (5)	-0.0014 (5)	-0.0015 (6)
C5	0.0365 (8)	0.0303 (8)	0.0343 (8)	0.0060 (6)	-0.0060 (7)	-0.0076 (6)
C6	0.0664 (14)	0.0536 (12)	0.0371 (10)	0.0086 (11)	0.0127 (10)	-0.0089 (9)
C7	0.0304 (8)	0.0403 (9)	0.0402 (9)	-0.0037 (7)	-0.0043 (7)	-0.0164 (8)
C8	0.0437 (11)	0.0739 (15)	0.0429 (11)	0.0034 (11)	-0.0187 (9)	-0.0073 (11)
C9	0.0253 (7)	0.0291 (7)	0.0279 (7)	-0.0028 (6)	-0.0018 (6)	-0.0040 (5)
C10A	0.017 (3)	0.029 (5)	0.033 (3)	-0.006 (2)	0.0045 (16)	-0.006 (3)
C11A	0.025 (3)	0.045 (3)	0.043 (4)	-0.003 (2)	0.005 (2)	-0.019 (3)
C12A	0.021 (3)	0.064 (8)	0.058 (5)	-0.018 (4)	0.010 (3)	-0.027 (5)
C13A	0.036 (3)	0.067 (5)	0.052 (4)	-0.028 (3)	0.021 (3)	-0.031 (3)
C14A	0.051 (4)	0.049 (3)	0.033 (3)	-0.025 (3)	0.014 (3)	-0.014 (2)
C15A	0.040 (4)	0.030 (3)	0.030 (3)	-0.011 (3)	0.013 (3)	-0.001 (2)
C10B	0.048 (6)	0.027 (6)	0.041 (8)	-0.013 (3)	0.009 (3)	-0.012 (6)
C11B	0.022 (4)	0.074 (9)	0.055 (8)	-0.002 (4)	-0.003 (5)	-0.033 (6)
C12B	0.030 (4)	0.063 (8)	0.086 (11)	-0.013 (5)	0.004 (5)	-0.036 (7)
C13B	0.031 (4)	0.097 (16)	0.073 (12)	-0.033 (8)	0.018 (6)	-0.049 (11)
C14B	0.069 (11)	0.089 (11)	0.041 (6)	-0.049 (9)	0.022 (6)	-0.022 (6)
C15B	0.039 (4)	0.049 (10)	0.049 (8)	-0.009 (5)	0.006 (5)	-0.011 (6)
C21	0.085 (8)	0.108 (10)	0.048 (5)	0.047 (8)	-0.011 (5)	-0.018 (5)
C22	0.115 (11)	0.102 (10)	0.060 (7)	0.023 (9)	-0.013 (7)	-0.004 (7)
C23	0.080 (8)	0.088 (8)	0.072 (7)	0.031 (6)	-0.014 (5)	-0.019 (6)
C24	0.088 (7)	0.108 (9)	0.071 (6)	0.012 (7)	0.007 (6)	0.025 (6)
C25	0.113 (10)	0.089 (9)	0.081 (8)	-0.029 (8)	-0.041 (7)	0.026 (7)
C26	0.098 (9)	0.075 (7)	0.065 (6)	-0.018 (7)	-0.037 (6)	-0.004 (5)
C27	0.165 (16)	0.101 (10)	0.067 (7)	0.043 (10)	-0.017 (9)	-0.016 (7)

Geometric parameters (Å, °)

N1—C4	1.365 (2)	C13A—C14A	1.379 (14)
N1—C1	1.3729 (19)	C13A—H13A	0.9500
N1—H1A	0.8800	C14A—C15A	1.398 (19)
C1—C9 ⁱ	1.405 (2)	C14A—H14A	0.9500
C1—C2	1.442 (2)	C15A—H15A	0.9500
C2—C3	1.375 (2)	C10B—C15B	1.36 (3)
C2—C5	1.502 (2)	C10B—C11B	1.36 (3)
C3—C4	1.438 (2)	C11B—C12B	1.374 (18)
C3—C7	1.497 (2)	C11B—H11B	0.9500
C4—C9	1.405 (2)	C12B—C13B	1.296 (18)
C5—C6	1.505 (3)	C12B—H12B	0.9500
C5—H5A	0.9900	C13B—C14B	1.39 (2)
C5—H5B	0.9900	C13B—H13B	0.9500
C6—H6A	0.9800	C14B—C15B	1.44 (3)
C6—H6B	0.9800	C14B—H14B	0.9500
C6—H6C	0.9800	C15B—H15B	0.9500
C7—C8	1.518 (3)	C21—C22	1.360 (19)

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C7—H7A	0.9900	C21—C26	1.39 (2)
C7—H7B	0.9900	C21—C27	1.506 (17)
C8—H8A	0.9800	C22—C23	1.411 (19)
C8—H8B	0.9800	C22—H22A	0.9500
C8—H8C	0.9800	C23—C24	1.352 (18)
C9—C1 ⁱⁱ	1.405 (2)	C23—H23A	0.9500
C9—C10B	1.41 (2)	C24—C25	1.51 (3)
C9—C10A	1.524 (13)	C24—H24A	0.9500
C10A—C15A	1.39 (2)	C25—C26	1.365 (17)
C10A—C11A	1.42 (2)	C25—H25A	0.9500
C11A—C12A	1.398 (12)	C26—H26A	0.9500
C11A—H11A	0.9500	C27—H27A	0.9800
C12A—C13A	1.390 (10)	C27—H27B	0.9800
C12A—H12A	0.9500	C27—H27C	0.9800
C4—N1—C1	109.84 (13)	C10A—C11A—H11A	120.8
C4—N1—H1A	125.1	C13A—C12A—C11A	120.9 (10)
C1—N1—H1A	125.1	C13A—C12A—H12A	119.5
N1—C1—C9 ⁱ	122.92 (14)	C11A—C12A—H12A	119.5
N1—C1—C2	107.24 (13)	C14A—C13A—C12A	120.0 (7)
C9 ⁱ —C1—C2	129.69 (14)	C14A—C13A—H13A	120.0
C3—C2—C1	107.58 (14)	C12A—C13A—H13A	120.0
C3—C2—C5	124.67 (15)	C13A—C14A—C15A	120.6 (8)
C1—C2—C5	126.78 (15)	C13A—C14A—H14A	119.7
C2—C3—C4	107.39 (14)	C15A—C14A—H14A	119.7
C2—C3—C7	125.87 (15)	C10A—C15A—C14A	119.6 (10)
C4—C3—C7	125.88 (15)	C10A—C15A—H15A	120.2
N1—C4—C9	123.72 (14)	C14A—C15A—H15A	120.2
N1—C4—C3	107.82 (13)	C15B—C10B—C11B	118.4 (19)
C9—C4—C3	128.46 (14)	C15B—C10B—C9	119.0 (19)
C2—C5—C6	111.57 (16)	C11B—C10B—C9	123 (2)
C2—C5—H5A	109.3	C10B—C11B—C12B	120.0 (18)
C6—C5—H5A	109.3	C10B—C11B—H11B	120.0
C2—C5—H5B	109.3	C12B—C11B—H11B	120.0
C6—C5—H5B	109.3	C13B—C12B—C11B	124 (2)
H5A—C5—H5B	108.0	C13B—C12B—H12B	118.2
C5—C6—H6A	109.5	C11B—C12B—H12B	118.2
C5—C6—H6B	109.5	C12B—C13B—C14B	119.3 (15)
H6A—C6—H6B	109.5	C12B—C13B—H13B	120.4
C5—C6—H6C	109.5	C14B—C13B—H13B	120.4
H6A—C6—H6C	109.5	C13B—C14B—C15B	117.7 (12)
H6B—C6—H6C	109.5	C13B—C14B—H14B	121.2
C3—C7—C8	111.77 (17)	C15B—C14B—H14B	121.2
C3—C7—H7A	109.3	C10B—C15B—C14B	120.6 (16)
C8—C7—H7A	109.3	C10B—C15B—H15B	119.7
C3—C7—H7B	109.3	C14B—C15B—H15B	119.7
C8—C7—H7B	109.3	C22—C21—C26	117.3 (11)
H7A—C7—H7B	107.9	C22—C21—C27	123.7 (16)
C7—C8—H8A	109.5	C26—C21—C27	119.0 (16)

C7—C8—H8B	109.5	C21—C22—C23	125.4 (16)
H8A—C8—H8B	109.5	C21—C22—H22A	117.3
C7—C8—H8C	109.5	C23—C22—H22A	117.3
H8A—C8—H8C	109.5	C24—C23—C22	117.6 (13)
H8B—C8—H8C	109.5	C24—C23—H23A	121.2
C4—C9—C1 ⁱⁱ	122.72 (14)	C22—C23—H23A	121.2
C4—C9—C10B	120.9 (9)	C23—C24—C25	118.9 (13)
C1 ⁱⁱ —C9—C10B	116.1 (10)	C23—C24—H24A	120.5
C4—C9—C10A	116.8 (6)	C25—C24—H24A	120.5
C1 ⁱⁱ —C9—C10A	120.4 (6)	C26—C25—C24	118.6 (16)
C15A—C10A—C11A	120.6 (11)	C26—C25—H25A	120.7
C15A—C10A—C9	119.8 (10)	C24—C25—H25A	120.7
C11A—C10A—C9	119.6 (13)	C25—C26—C21	122.0 (16)
C12A—C11A—C10A	118.4 (11)	C25—C26—H26A	119.0
C12A—C11A—H11A	120.8	C21—C26—H26A	119.0

Symmetry codes: (i) $-y+3/4, x-1/4, -z+1/4$; (ii) $y+1/4, -x+3/4, -z+1/4$.

Fig. 1

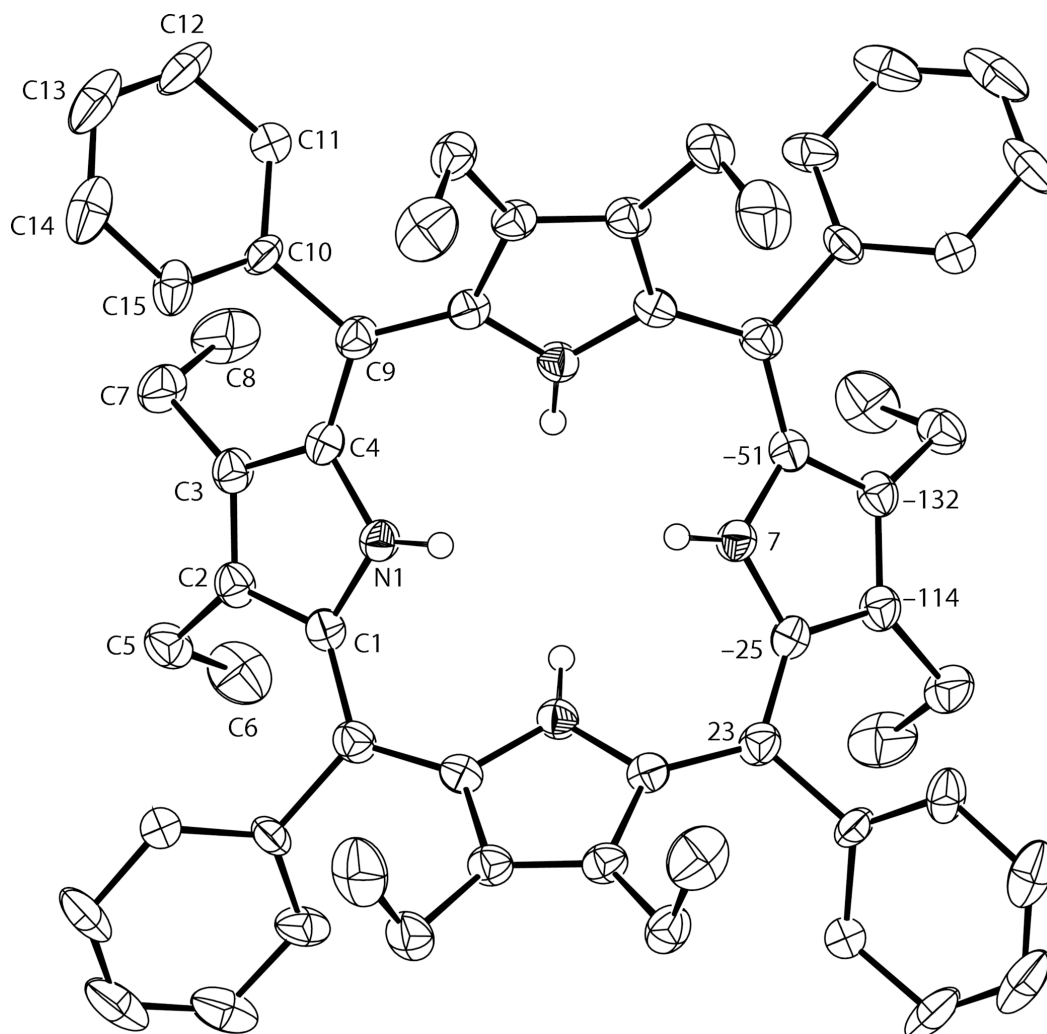


Fig. 2

