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The highly saddled diacid salt of octaethyltetraphenylporphyrin-[H₄OETPP] 2Cl

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Abstract

The title compound, [H₄OETPP] 2 Cl 1, displays a highly S₄-saddled porphyrin core with the β -carbon atoms displaced 1.23 (9) Å out of the four-nitrogen plane. All nitrogen atoms are protonated and form hydrogen bonds with chloride anions.

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); Sheldrick (2001).

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2/SAINT* (Bruker, 2004); data reduction: *SAINT/XPREF* (Bruker, 2004; Sheldrick, 2005); program(s) used to solve structure: *XS* (Sheldrick, 2001); program(s) used to refine structure: *XL* (Sheldrick, 2001); molecular graphics: *XP/PLATON* (Sheldrick, 1998; Spek, 2004); software used to prepare material for publication: *XCIF* (Sheldrick, 2001).

Crystal data

C₆₀H₆₄N₄ · 33(C₇H₈) 2Cl $\gamma = 90^\circ$
 $M_r = 1034.90$ $V = 17200.5 (2) \text{ \AA}^3$
 Cubic, $I\bar{4}_3d$ $Z = 12$
 $a = 25.8135 (2) \text{ \AA}$ $\text{Mo K}\alpha$
 $b = 25.8135 (2) \text{ \AA}$ $\mu = 0.16 \text{ mm}^{-1}$
 $c = 25.8135 (2) \text{ \AA}$ $T = 100 (2) \text{ K}$
 $\alpha = 90^\circ$ $0.42 \times 0.32 \times 0.29 \text{ mm}$
 $\beta = 90^\circ$

Data collection

CCD area detector diffractometer	4414 independent reflections
Absorption correction: multi-scan Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.	3859 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.936$, $T_{\max} = 0.955$	$R_{\text{int}} = 0.040$
176336 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.139$	$\Delta\rho_{\max} = 0.42 \text{ e } \text{\AA}^{-3}$
$S = 1.15$	$\Delta\rho_{\min} = 0.19 \text{ e } \text{\AA}^{-3}$
4414 reflections	Absolute structure: Flack H D (1983), 2073 Friedel pairs
271 parameters	Flack parameter: $-0.07(7)$
42 restraints	

Selected geometric parameters (Å)

N1—C4	1.365 (2)	C3—C4	1.438 (2)
N1—C1	1.3729 (19)	C3—C7	1.497 (2)
C1—C9 ⁱ	1.405 (2)	C4—C9	1.405 (2)
C1—C2	1.442 (2)	C5—C6	1.505 (3)
C2—C3	1.375 (2)	C7—C8	1.518 (3)
C2—C5	1.502 (2)		

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

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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References

- Adler AD, Longo FR, Kampus F, Kim J. J Inorg Nucl Chem. 1970; 32:2443–2445.
- Barkigia KM, Chantranupong L, Smith KM, Fajer J. J Am Chem Soc. 1988; 110:7566–7567.
- Barkigia KM, Fajer J, Berber MD, Smith KM. Acta Crystallogr Sect C:Cryst Struct Commun. 1995; 51:511–515.
- Bruker-Nonius AXS. *APEX2* and *SAINT*. Bruker-Nonius AXS; Madison, Wisconsin, USA: 2004.
- Regev A, Galili T, Medforth CJ, Smith KM, Barkigia KM, Fajer J, Levanon H. J Phys Chem. 1994; 98:2520–2526.
- Senge MO. J Photochem Photobiol B. 1992; 16:3–36.
- Senge MO, Forsyth TP, Nguyen LT, Smith KM. Angew Chem Int Ed. 1994; 33:2485–2487.
- Senge MO, Kalisch WW. Z Naturforsch B Chem Sci. 1999; 54:943–959.
- Sheldrick, GM. XP. Bruker-Nonius AXS; Madison, Wisconsin, USA: 1998.
- Sheldrick, GM. XCIF, XL and XS. Bruker-Nonius AXS; Madison, Wisconsin, USA: 2001.
- Sheldrick, GM. SADABS. University of Göttingen; Germany: 2004.
- Sheldrick, GM. XPREF. Bruker-Nonius AXS; Madison, Wisconsin, USA: 2005.
- Spek, AL. *PLATON*: A Multipurpose Crystallographic Tool. Utrecht University; Utrecht, The Netherlands: 2004.