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[5,15-Bis(4-pyridyl)-2,8,12,18-tetra-*n*-butyl-3,7,13,17-tetramethylporphyrinato]zinc(II) chloroform solvate

Kirstie Y. Wild, Bruno Therrien, Paul G. Plieger, David L. Officer and Anthony K. Burrell*

Department of Chemistry, Massey University, Private Bag 11222, Palmerston North, New Zealand

Correspondence e-mail:
a.k.burrell@massey.ac.nz

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The crystal structure of the title compound, [Zn($C_{50}H_{58}N_6$)]·$CHCl_3$, reveals a polymeric chain in a zigzag conformation, with a single pyridyl group from each porphyrin making the connection.

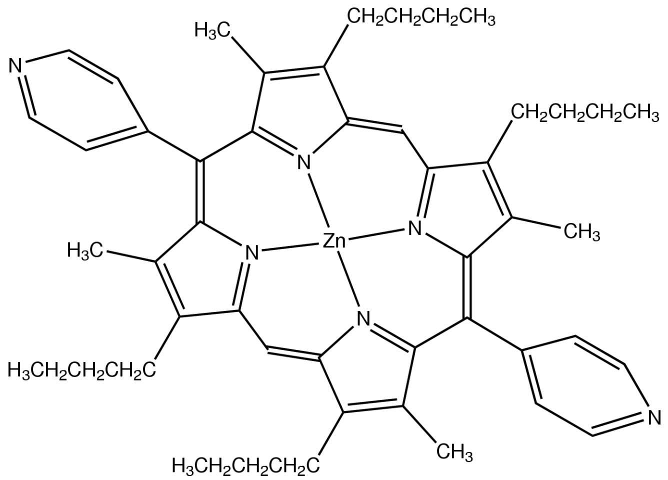
Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.065
wR factor = 0.169
Data-to-parameter ratio = 18.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The formation of porphyrin assemblies using coordination chemistry has been an active area of research for the last few years. The formation of coordination polymers using zinc porphyrins with pyridyl substituents was pioneered by the Fleischer group (Shachter *et al.*, 1988). Recently, we have explored the use of pyridyl porphyrins to prepare porphyrin assemblies (Burrell *et al.*, 1998). In general, the number of pyridyl groups on the porphyrin has little effect on the nature of the solid-state structure of the polymer obtained upon insertion of zinc. Thus, the monopyridyl porphyrin (Shachter *et al.*, 1988) has essentially the same structure as the tetrapyridyl analogue (Krupitsky *et al.*, 1994).



(I)

In the title compound, (I), the porphyrin has β-alkyl groups which have a different steric requirement than phenyl or pyridyl groups (Fig. 1). However, the same structural motif is conserved with a single pyridyl group being utilized in formation of the coordination polymer (Fig. 2). The linear chains are stacked one upon another with the closest approach being approximately 4 Å. The sheets that result from this stacking are separated by channels that contain the chloroform solvate.

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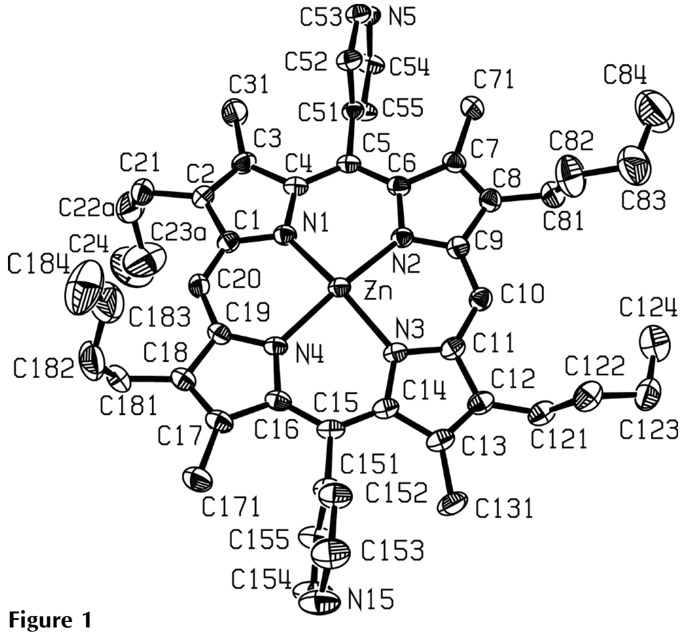

Figure 1
ORTEP-3 (Farrugia, 1997) view of (I) showing displacement ellipsoids drawn at the 50% probability level. The minor disorder component is not shown.

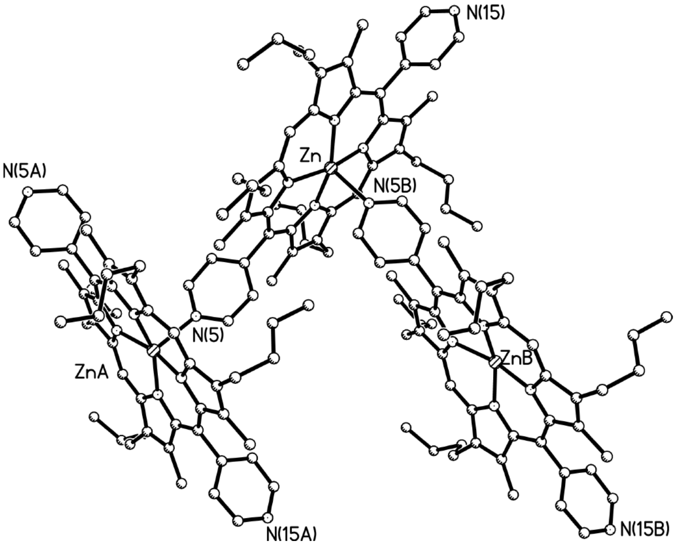

Figure 2
The polymer conformation where Zn*A* is generated by $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$ and Zn*B* is generated by $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Experimental

The 5,15-bis(4-pyridyl)porphyrin was synthesized according to a previously reported method (Forsyth *et al.*, 1995). Zinc insertion: zinc acetate (4.61 mg, 21.0 µ*M*), dissolved in MeOH (3 ml), was added to 5,15-bis(4-pyridyl)porphyrin (15.5 mg, 20.8 µ*M*) in CH_2Cl_2 (20 ml). The mixture was refluxed for 20 min, at which point thin-layer chromatography (1% $MeOH/CH_2Cl_2$) showed the reaction was complete. The solvent was removed and the residue was dissolved in CH_2Cl_2 (10 ml), washed with water (10 ml) and dried ($MgSO_4$). Quantitative yield. Crystals were grown by the slow evaporation of a 1:1 solvent mixture of $CHCl_3$/MeOH. ^{1}H NMR (400 MHz, $CDCl_3$): 9.95 (*s*, 2H), 7.06 (*s br*, 4H), 3.78 (*t*, 8H), 1.98 (*quint*, 8H), 1.61 (*m*, 8H), 1.62 (*s br*, 4H), 1.24 (*s*, 12H), 1.00 (*t*, 12H).

Crystal data

$[Zn(C_{50}H_{58}N_6)]\cdot CHCl_3$
$M_r = 927.76$
Monoclinic, $P2_1/n$
$a = 17.8146$ (2) Å
$b = 13.1650$ (2) Å
$c = 20.2171$ (4) Å
$\beta = 90.84$ (1)°
$V = 4741.00$ (13) Å^3
$Z = 4$

$D_x = 1.300$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6496 reflections
$\theta = 1.5$–28.3°
$\mu = 0.73$ mm^{-1}
$T = 203$ (2) K
Plate, red
$0.30 \times 0.22 \times 0.02$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.800$, $T_{\max} = 0.986$
10 404 measured reflections

10 404 independent reflections
6454 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5°$
$h = -23 \rightarrow 23$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.169$
$S = 1.08$
10404 reflections
564 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 7.9269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -1.03$ e Å^{-3}

One *n*-butyl chain was found disordered (59/41 occupation factors). The *n*-butyl C atoms (C22 and C23) were refined with two split positions.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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