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3,6,14,17-Tetraoxa-23,24-diazatricyclo[17.3.1.1^{8,12}]-tetracosa-1(23),8,10,12(24),19,21-hexaene (bis-pyridino-18-crown-6)

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The title compound, $C_{18}H_{22}N_2O_4$, lies about a centre of inversion. The four ether O atoms lie in a plane, with the two strictly parallel pyridine rings located on either side of this plane; their N atoms point in opposite directions.

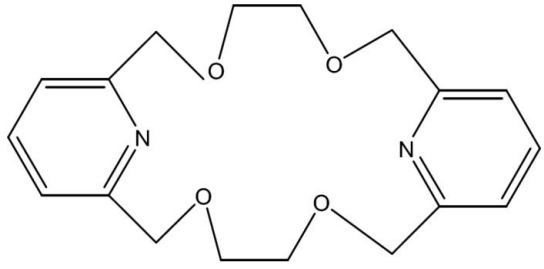
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.092
Data-to-parameter ratio = 13.3

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

Comment

There has been a growing interest in the synthesis of macrocyclic polyethers, especially those containing one or more pyridine units. Pyridine crown ethers coordinate readily with transition metal cations (Cane & Buchwald, 1977; Lamb *et al.*, 1980; Tummler *et al.*, 1977) and neutral molecules (van Staveren *et al.*, 1984; Grootenhuis *et al.*, 1986), as the pyridine N atom is a good electron donor. Although the title compound, bis-pyridino-18-crown-6, (I), has been synthesized previously (Newcomb *et al.*, 1977), its crystal structure has not been investigated to date and is reported here (Fig. 1).



(I)

The molecule lies on a centre of inversion at the centre of the 18-crown ring. The four ether O atoms lie in a plane. The two strictly parallel pyridine rings are located on either side of this plane with their N atoms pointing in opposite directions. The angle between the O1/O2/O1*A*/O2*A* plane and those of the pyridine rings is 82.06 (4)°. The cavity of the crown ether is distorted to be thinner and longer than that observed for tetramethyl-bis-pyridino-18-crown-6, while the average C—O bond length (1.424 Å) is similar (Bradshaw *et al.*, 1990).

Experimental

Bis-pyridino-18-crown-6 was synthesized according to the method of Bradshaw *et al.* (1990). Colourless single crystals of (I) were obtained from CH_2Cl_2 and petroleum ether (1:1 *v*/*v*) after 2 d (m.p. 418–420 K).

Crystal data

$C_{18}H_{22}N_2O_4$
M_r = 330.38
Orthorhombic, *Pbca*
a = 14.797 (4) Å
b = 7.2616 (19) Å
c = 15.191 (4) Å
V = 1632.2 (7) Å^3
Z = 4
D_x = 1.344 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3983 reflections
θ = 2.7–25.0°
μ = 0.10 mm^{-1}
T = 298 (2) K
Block, colourless
0.53 × 0.48 × 0.41 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.962
7873 measured reflections
1449 independent reflections
1198 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -15 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -18 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.092
S = 1.07
1449 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.3434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

All H atoms were refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H, and C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2 atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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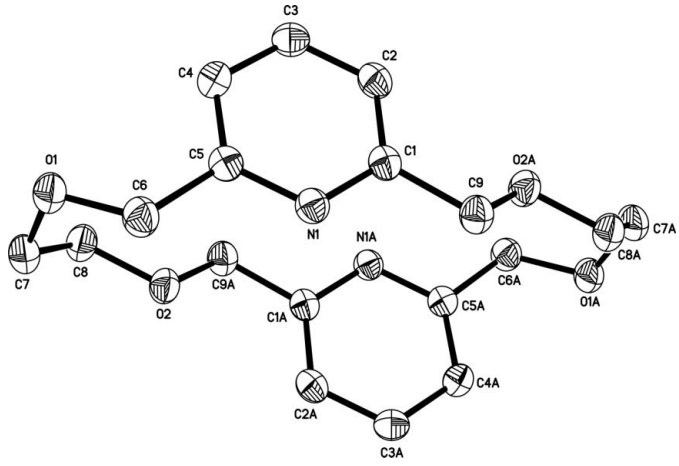

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The suffix *A* indicates the symmetry operation (1 − x, −y, 1 − z). H atoms have been omitted.

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