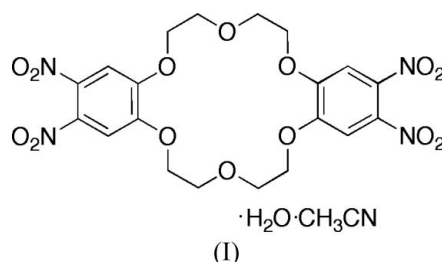


4',4'',5',5''-Tetranitro-2,3:11,12-dibenzo-  
1,4,7,10,13,16-hexaoxacyclooctadeca-2,11-diene  
acetonitrile solvate monohydrateMatthew J. Belousoff, Steven J.  
Langford,\* Melissa J. Latter and  
Vei L. LauSchool of Chemistry, Monash University,  
Wellington Rd, Clayton, 3800, Victoria,  
AustraliaCorrespondence e-mail:  
Steven.Langford@sci.monash.edu.au

## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.050  
 $wR$  factor = 0.119  
Data-to-parameter ratio = 16.0For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The structure of the title compound,  $\text{C}_{20}\text{H}_{20}\text{N}_4\text{O}_{14} \cdot \text{H}_2\text{O} \cdot \text{CH}_3\text{CN}$ , dimerizes in the solid state to form an open-ended box which holds two  $\text{H}_2\text{O}$  and two  $\text{CH}_3\text{CN}$  molecules.Received 30 August 2005  
Accepted 12 September 2005  
Online 17 September 2005

## Comment

The complexation ability of crown ethers has been well studied with regard to alkali metal ions and ammonium derivatized guests (Gokel *et al.*, 2004). Functionalized dibenzocrown ethers offer a versatile means for incorporation into supramolecular constructs, allowing potential applications in artificial photosynthesis and molecular devices to be developed (Langford *et al.*, 2002). We report here the crystal structure of the tetranitrodibenzocrown ether, (I), an important precursor to a family of antipodally arranged bichromophoric systems.The synthesis of (I) was achieved using a previously described method (Duggan *et al.*, 2001). Slow evaporation of a solution in  $\text{CH}_3\text{CN}$  and  $\text{H}_2\text{O}$  (1:1  $v/v$ ) produced crystals suitable for X-ray analysis. The crystal structure reveals a single crown ether in the asymmetric unit (Fig. 1), cocrystallized with one molecule of  $\text{CH}_3\text{CN}$  and one of  $\text{H}_2\text{O}$ . Both solvent molecules participate in hydrogen bonding; the  $\text{H}_2\text{O}$  molecule bridges (I) and  $\text{CH}_3\text{CN}$ , with  $D-A$  distances of 2.314 (17) Å and 2.105 (16) Å, respectively (see Table 1).Molecules of (I) dimerize in the solid state to form open-ended boxes in which two molecules of  $\text{H}_2\text{O}$  and two molecules of  $\text{CH}_3\text{CN}$  are housed, as shown in Fig. 2. Edge-on stacking of (I) forms channels (*ca* 8 Å wide and 15 Å long) which are filled by the included solvent molecules. The stacking is stabilized by  $\pi-\pi$  interactions between crown ethers with a mean plane separation distance of 3.61 Å.

## Experimental

The title compound was synthesized by a literature procedure (Duggan *et al.*, 2001). Yellow crystals suitable for X-ray analysis were grown by slow evaporation of a  $\text{CH}_3\text{CN}$  and  $\text{H}_2\text{O}$  (1:1  $v/v$ ) solution of the compound.

Crystal data

C<sub>20</sub>H<sub>20</sub>N<sub>4</sub>O<sub>14</sub>·C<sub>2</sub>H<sub>3</sub>N·H<sub>2</sub>O  
*M<sub>r</sub>* = 599.47  
 Triclinic, *P*1̄  
*a* = 10.766 (2) Å  
*b* = 11.648 (2) Å  
*c* = 11.813 (2) Å  
 α = 69.89 (3)°  
 β = 74.07 (3)°  
 γ = 75.84 (3)°  
*V* = 1319.0 (5) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.509 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 25863 reflections  
 θ = 4–25°  
 μ = 0.13 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Square prism, yellow  
 0.2 × 0.17 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer  
 Thick-slice φ and ω scans  
 25863 measured reflections  
 6200 independent reflections  
 3503 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.089  
 θ<sub>max</sub> = 27.9°  
*h* = -12 → 14  
*k* = -15 → 15  
*l* = -15 → 15

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.050  
*wR* (*F*<sup>2</sup>) = 0.119  
*S* = 1.00  
 6200 reflections  
 388 parameters

H atoms treated by a mixture of independent and constrained refinement  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0527*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.33 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.32 e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> – <i>H</i> ··· <i>A</i>	<i>D</i> – <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> – <i>H</i> ··· <i>A</i>
O1W–H1W···O13 <sup>i</sup>	0.84 (1)	2.31 (2)	3.141 (3)	169 (7)
O1W–H2W···N5	0.84 (1)	2.11 (2)	2.932 (3)	168 (5)

Symmetry code: (i) -*x*, -*y* + 1, -*z* + 1.

All H atoms were placed in idealized positions except for the water H atoms. O1W–H1W and O1W–H2W distances were restrained to 0.83 (1) Å.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the Australian Research Council, Discovery Project Scheme through awards to SJL (DP0343096 & DP0556313). VLL thanks the Australian government for an Australian Postgraduate Research award.

References

Duggan, S. A., Fallon, G., Langford, S. J., Lau, V-L., Satchell, J. F., Paddon-Row, M. N. (2001). *J. Org. Chem.* **66**, 4419–4426.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

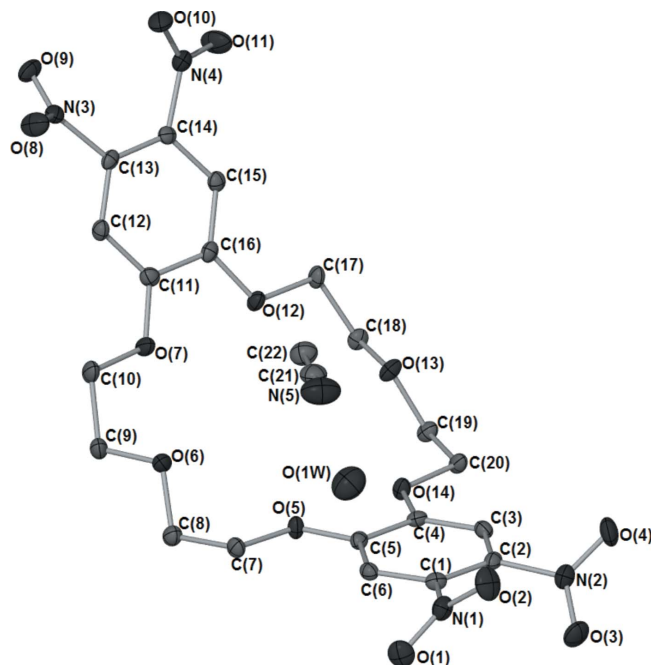


Figure 1  
 ORTEP-3 (Farrugia, 1997) representation of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

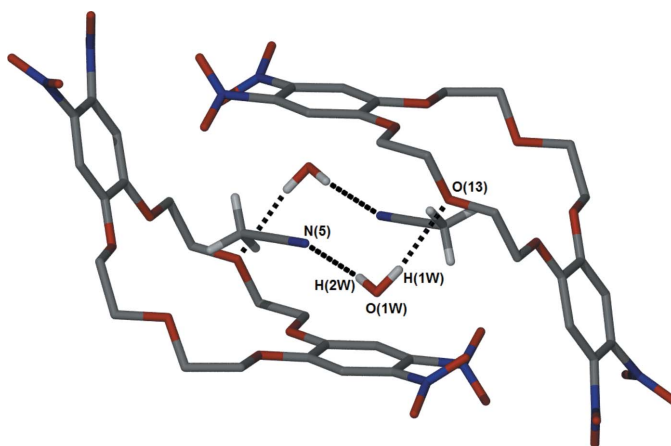


Figure 2  
 Solid-state dimer formed by (I), with included solvent, showing the extent of hydrogen bonding (dashed lines). H atoms on the crown ether have been omitted for clarity.

Gokel, G. W., Leevy, W. M. & Weber, M. E. (2004). *Chem. Rev.* **104**, 2723–2750.  
 Langford, S. J., Lau, V-L., Lee, M. A. P., Lygris, E. (2002). *J. Porph. Phthal.* **6**, 748–756.  
 Nonius (2002). *COLLECT*. Nonius BV, Delft, The Netherlands.  
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.