

1,8-Anthraquinono-18-crown-5 monohydrate

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Key indicators

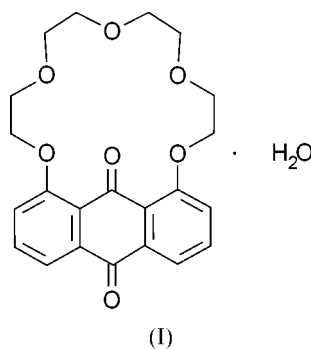
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.059
 wR factor = 0.218
Data-to-parameter ratio = 14.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The monohydrate of 1,8-anthraquinono-18-crown-5, $\text{C}_{22}\text{H}_{22}\text{O}_7 \cdot \text{H}_2\text{O}$, crystallizes in the space group $P\bar{1}$ with one complex molecule in the asymmetric unit, encapsulating the water molecule. The crystal structure is stabilized by hydrogen-bonding and aromatic π - π stacking interactions.

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Comment

Known crystal structures involving 1,8-anthraquinono-18-crown-5, (II), are the host-guest complexes of (II) with hydronium perchlorate (Young *et al.*, 1997), nitric acid trihydrate and sulfonic acid monohydrate (Young & Sykes, 1998). The striking feature of all these complexes is that, irrespective of the different modes of intermolecular interactions given by the variable content of water of crystallization and the presence of strong inorganic acids, the impact of the guest components on the crown ether conformation is only weak. Surprisingly, this is also shown in the present monohydrate of (II) lacking the acid component. In the monohydrate of (II), hereafter (I), the asymmetric unit contains a crown ether that encapsulates the water molecule.



The crown ether has non-crystallographic mirror symmetry (point group C_s) with atoms O1, O2, C6, C13 and O5 lying on the symmetry plane of the molecule. The O-C-C-O torsion angles within the polyether ring [range $64.8(3)$ – $79.9(3)^\circ$] are *gauche* with alternating signs, whereas the torsion angles of the C-O-C-C sequences [range $164.4(3)$ – $178.5(2)^\circ$] exhibit an *anti* conformation. Additionally, the anthraquinone unit shows a roof-like shape, in which the two aromatic rings adopt a dihedral angle of $21.5(4)^\circ$. The carbonyl atoms O1 and O2 are strongly bent out of the anthraquinone plane, which can be deduced from the dihedral angle between the planes of the C5-C6-C7-O1 and C12-C13-C14-O2 fragments [$42.2(3)^\circ$]. The water molecule is associated, *via* strong hydrogen bonds, with O4 and O6 of the crown ether. The host-guest aggregates within the crystal structure are associated *via* bifurcated C-H...O hydrogen

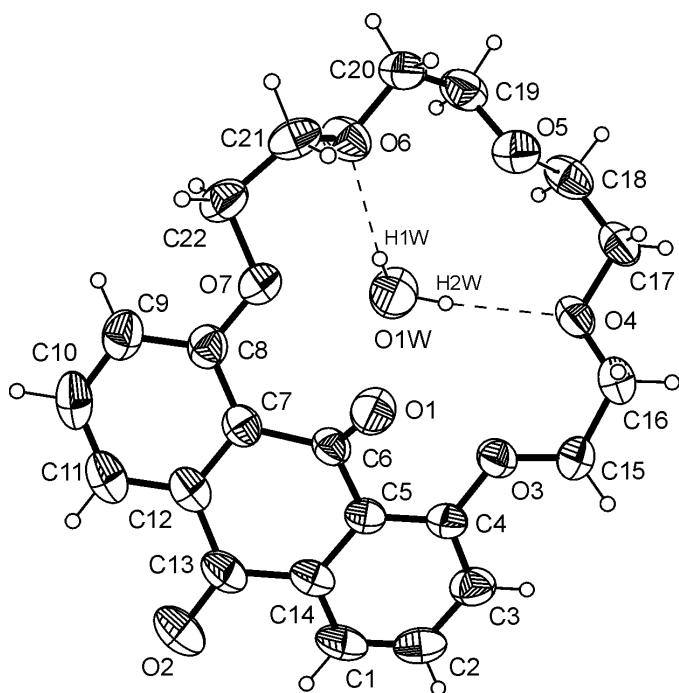


Figure 1
Perspective view of (I), showing 50% probability displacement ellipsoids for the non-H atoms. Hydrogen bonds are shown as dashed lines.

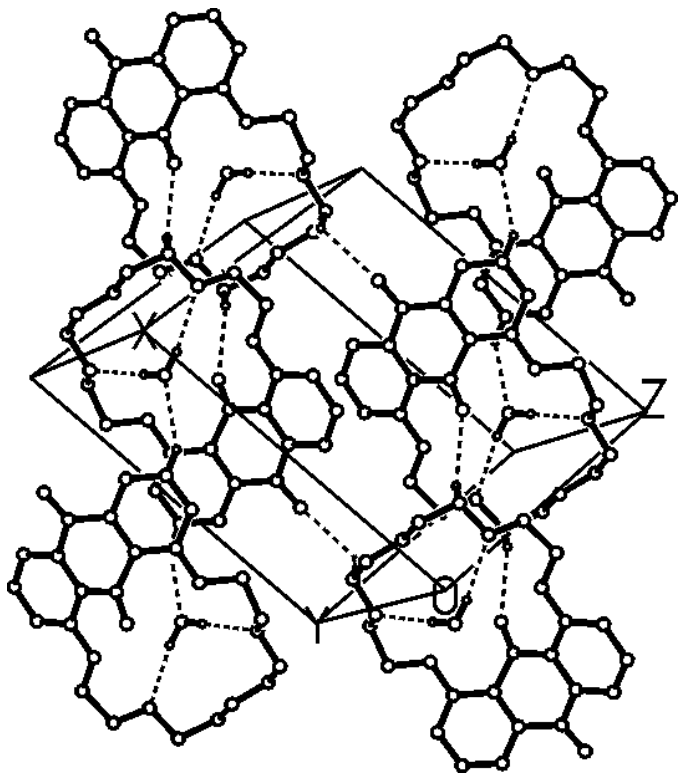


Figure 2
Packing diagram of (I). With the exception of water H atoms and the coordinating atom H2 of the anthraquinone unit, all H atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

bonds involving the water O atom, atom H2 of the anthraquinone unit and methylene atom H20B. As shown in the

packing diagram, the anthraquinone residues establish hydrophobic layers which are held together by face-to-face π -interactions [$Cg1 \cdots Cg1^i = 3.55 \text{ \AA}$ and $Cg2 \cdots Cg2^{ii} = 3.58 \text{ \AA}$; $Cg1$ and $Cg2$ are the centroids of rings C1–C5/C14 and C7–C12, respectively; symmetry codes: (i) $2 - x, 2 - y, 1 - z$; (ii) $1 - x, 1 - y, 1 - z$]. The non-polar layers are separated by the crown ether units. However, none of their O atoms participates in intermolecular hydrogen bonding.

Experimental

1,8-Anthraquinono-18-crown-5, (II), was synthesized according to the procedure described by Delgado *et al.* (1988) from 1,8-dichloroanthraquinone and tetraethyleneglycol in anhydrous tetrahydrofuran. Purification was carried out by column chromatography (silica gel, chloroform–methanol 5:1). Slow evaporation of the solvent yielded 45% of the monohydrate as yellow crystals.

Crystal data

$C_{22}H_{22}O_7 \cdot H_2O$	$Z = 2$
$M_r = 416.41$	$D_x = 1.376 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Cu $K\alpha$ radiation
$a = 7.441 (1) \text{ \AA}$	Cell parameters from 25 reflections
$b = 11.502 (2) \text{ \AA}$	$\theta = 7.9\text{--}22.6^\circ$
$c = 12.492 (2) \text{ \AA}$	$\mu = 0.88 \text{ mm}^{-1}$
$\alpha = 98.80 (3)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 106.00 (3)^\circ$	Irregular fragment, yellow
$\gamma = 95.47 (3)^\circ$	$0.25 \times 0.20 \times 0.20 \text{ mm}$
$V = 1004.9 (3) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\max} = 74.9^\circ$
ω - 2θ scans	$h = -9 \rightarrow 8$
Absorption correction: none	$k = -14 \rightarrow 14$
4376 measured reflections	$l = -15 \rightarrow 15$
4054 independent reflections	3 standard reflections every 250 reflections
2451 reflections with $I > 2\sigma(I)$	intensity decay: none
$R_{\text{int}} = 0.026$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.059$	$(\Delta/\sigma)_{\max} = 0.001$
$wR(F^2) = 0.218$	$\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$
$S = 1.00$	$\Delta\rho_{\min} = -0.32 \text{ e \AA}^{-3}$
4054 reflections	
279 parameters	

Table 1
Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1W-H1W \cdots O6$	0.88 (7)	2.30 (7)	3.127 (4)	157 (6)
$O1W-H2W \cdots O4$	0.89 (6)	2.21 (6)	3.095 (4)	170 (5)
$C2-H2 \cdots O1W^i$	0.93	2.56	3.308 (4)	138
$C20-H20B \cdots O1^{ii}$	0.97	2.54	3.255 (4)	131

Symmetry codes: (i) $2 - x, 2 - y, 1 - z$; (ii) $1 - x, 1 - y, -z$.

H atoms bonded to carbon were positioned geometrically and refined using a riding model, with C–H distances of 0.93 \AA for aromatic CH and 0.97 \AA for methylene CH. Their displacement parameter were set at $1.2U_{\text{eq}}$ of the parent atom. The water H atoms were refined isotropically.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997);

program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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