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(9,10-Triptyceno)benzo-28-crown-8

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Key indicators

Single-crystal X-ray study
T = 233 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.134
Data-to-parameter ratio = 12.4

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

The structure of the title compound, $C_{40}H_{44}O_8$, differs from previously determined structures of triptyceno–crown ether molecules as its longer crown ether chain does not congest a triptycene cavity.

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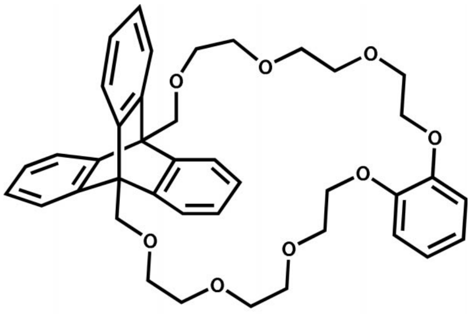
Comment

We have studied triptycene-substituted crown ether rings as molecular gearing systems (Gakh *et al.*, 1997; Bryan *et al.*, 1999) and as potential recognition agents for cations with a low charge-to-size ratio (Gakh *et al.*, 1995). Since the steric demands of inserting the relatively large triptycene group into 18-crown-6 or 21-crown-7 resulted in an elliptically shaped cavity, poorly suited for binding spherical cations (Gakh *et al.*, 1995; Bryan *et al.*, 1999), we decided to investigate the title compound, (I).


(I)

The title compound can be thought of as the result of inserting a triptycene group into benzo-24-crown-8. The crown ring does not collapse in on itself to the extent normally observed for large crown ether molecules (Bryan *et al.*, 2000, and references therein). In contrast to the previously determined structures of triptyceno-22-crown-6 (Bryan, *et al.*, 1999) and triptyceno-25-crown-7 (Gakh *et al.*, 1995), the crown ether does not fill the space between two triptycene arene rings, but is elongated away from it. As a result, the inter-arene angles of the triptycene group are all close to 120°, whereas the smaller crown segments force a larger inter-arene angle between the two rings occupied by the crown. The inter-arene angles for the triptycene group in the title compound are: $A1—A2$ = 121.7 (1), $A1—A3$ = 119.4 (1), and $A2—A3$ = 118.9 (1)°, where $A1$ = C3–8, $A2$ = C9–14, and $A3$ = C15–20.

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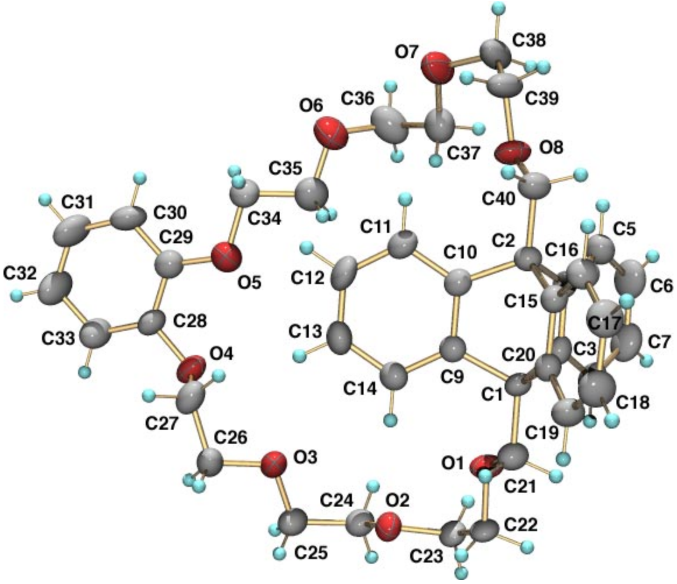

Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids. For clarity, the minor disorder component is not shown.

Experimental

The compound was prepared as previously described (Gakh *et al.*, 1997). Crystals were grown from 2-methoxyethanol. Similarly sized crystals were also found to grow from 2-propanol.

Crystal data

$C_{40}H_{44}O_8$
$M_r = 652.8$
Monoclinic, $P2_1/c$
$a = 9.3198\ (17)$ Å
$b = 23.416\ (2)$ Å
$c = 15.838\ (3)$ Å
$\beta = 91.333\ (9)^\circ$
$V = 3455.4\ (10)$ Å^3
$Z = 4$

$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.1–12.2^\circ$
$\mu = 0.09$ mm^{-1}
$T = 233$ K
Plate, colorless
0.63 × 0.50 × 0.04 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
7830 measured reflections
6061 independent reflections
3227 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$

$\theta_{\rm max} = 25.0^\circ$
$h = 0 \rightarrow 11$
$k = -16 \rightarrow 27$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.134$
$S = 1.01$
6061 reflections
487 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C21	1.411 (3)	O6—C35	1.414 (9)
O1—C22	1.411 (3)	O6—C36	1.394 (7)
O2—C23	1.418 (3)	O6*A*—C35*A*	1.407 (15)
O2—C24	1.412 (3)	O6*A*—C36*A*	1.425 (14)
O3—C25	1.412 (3)	O7—C37	1.443 (16)
O3—C26	1.415 (3)	O7—C38	1.469 (16)
O4—C27	1.426 (3)	O7*A*—C38	1.33 (3)
O4—C28	1.384 (3)	O7*A*—C37*A*	1.43 (3)
O5—C29	1.362 (3)	O8—C39	1.420 (3)
O5—C34	1.475 (12)	O8—C40	1.422 (3)
O5—C34*A*	1.35 (3)		
C21—O1—C22	113.47 (18)	O4—C27—C26	109.7 (2)
C23—O2—C24	111.13 (19)	O4—C28—C29	120.8 (2)
C25—O3—C26	112.36 (19)	O4—C28—C33	118.9 (2)
C27—O4—C28	113.36 (19)	O5—C29—C30	124.9 (2)
C29—O5—C34	113.7 (5)	O5—C29—C28	116.3 (2)
C29—O5—C34*A*	128.4 (12)	O5—C34—C35	105.6 (9)
C35—O6—C36	113.6 (5)	O5—C34*A*—C35*A*	114 (2)
C35*A*—O6*A*—C36*A*	111.7 (10)	O6—C35—C34	107.2 (8)
C37—O7—C38	113.4 (9)	O6*A*—C35*A*—C34*A*	109.0 (15)
C37*A*—O7*A*—C38	115.8 (18)	O6—C36—C37	116.3 (5)
C39—O8—C40	111.68 (19)	O6*A*—C36*A*—C37*A*	109.9 (10)
O1—C21—C1	109.53 (19)	O7—C37—C36	107.9 (8)
O1—C22—C23	108.41 (19)	O7*A*—C37*A*—C36*A*	106.5 (16)
O2—C23—C22	109.29 (18)	O7—C38—C39	117.8 (7)
O2—C24—C25	110.3 (2)	O7*A*—C38—C39	108.5 (13)
O3—C25—C24	109.8 (2)	O8—C39—C38	108.9 (2)
O3—C26—C27	109.5 (2)	O8—C40—C2	110.16 (19)
C22—O1—C21—C1	175.73 (17)	C35—O6—C36—C37	−93.9 (7)
C21—O1—C22—C23	175.31 (18)	C38—O7—C37—C36	−165.9 (8)
C24—O2—C23—C22	173.31 (19)	C37—O7—C38—C39	−81.4 (11)
C23—O2—C24—C25	174.2 (2)	C40—O8—C39—C38	168.9 (2)
C26—O3—C25—C24	−174.6 (2)	C39—O8—C40—C2	167.38 (19)
C25—O3—C26—C27	−168.7 (2)	O1—C22—C23—O2	−76.4 (2)
C28—O4—C27—C26	153.3 (2)	O2—C24—C25—O3	74.4 (3)
C27—O4—C28—C29	89.3 (3)	O3—C26—C27—O4	75.3 (3)
C27—O4—C28—C33	−91.9 (3)	O4—C28—C29—O5	−2.1 (4)
C34—O5—C29—C28	174.9 (6)	O5—C34—C35—O6	178.7 (8)
C34—O5—C29—C30	−4.8 (7)	O6—C36—C37—O7	−73.0 (9)
C29—O5—C34—C35	−168.9 (7)	O7—C38—C39—O8	68.5 (7)
C36—O6—C35—C34	−151.2 (8)		

Part of the crown ring (between C34 and O7) is disordered, and was modeled over two sites (67:33). Chemically equivalent bond distances and angles between the two disorder components were restrained to be approximately equal. Additionally, the displacement parameters for all disordered atoms were restrained to be similar to their nearest neighbor, or any atom within 0.7 Å. A 1.1 mm collimator was used. All H atoms were placed in calculated positions, refined using a riding model, and given an isotropic displacement parameter equal to 1.2 times the equivalent isotropic displacement parameter of the atom to which they were attached. The C—H distances used depended on the type of C atom: $C_{\rm methylene}$—H = 0.98 Å and $C_{\rm arene}$—H = 0.94 Å.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993); cell refinement: *CAD*-4-*PC*; data reduction: *XCAD*4 (Harms, 1995); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2001).

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References

Bryan, J. C., Sachleben, R. A., Gakh, A. A. & Bunick, G. J. (1999). *J. Chem. Crystallogr.* **29**, 513–521.

Bryan, J. C., Lavis, J. M., Hay, B. P. & Sachleben, R. A. (2000). *Acta Cryst.* C**56**, 391–392.

Enraf–Nonius (1993). *CAD*-4-*PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Gakh, A. A., Sachleben, R. A. & Bryan, J. C. (1997). *CHEMTECH*, **27**(11), 26–33.

Gakh, A. A., Sachleben, R. A., Bryan, J. C. & Moyer, B. A. (1995). *Tetrahedron Lett.* **45**, 8163–8166.

Harms, K. (1995). *XCAD*4. Universität Marburg, Germany.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2001). *PLATON*. University of Utrecht, The Netherlands.