

C14A—S1A—C15A	103.8 (1)	C14B—S1B—C15B	103.4 (1)
C14A—S3A—C18A	101.8 (1)	C14B—S3B—C18B	104.0 (1)
C7A—N1A—C1A	117.6 (2)	C7B—N1B—C1B	118.2 (2)
C15A—N2A—C16A	119.9 (3)	C15B—N2B—C16B	120.9 (3)
C15A—N2A—C17A	124.3 (2)	C15B—N2B—C17B	122.6 (3)
C16A—N2A—C17A	115.7 (3)	C16B—N2B—C17B	116.5 (3)
C18A—N3A—C19A	123.0 (3)	C18B—N3B—C19B	124.4 (2)
C18A—N3A—C20A	120.5 (4)	C18B—N3B—C20B	121.0 (2)
C19A—N3A—C20A	116.4 (3)	C19B—N3B—C20B	114.6 (2)
N1A—C1A—C2A	111.9 (2)	N1B—C1B—C2B	111.5 (2)
N1A—C1A—C6A	108.9 (2)	N1B—C1B—C6B	107.6 (2)
N1A—C7A—C8A	108.2 (2)	N1B—C7B—C8B	110.9 (2)
N1A—C7A—C12A	111.2 (2)	N1B—C7B—C12B	108.8 (2)
O1A—C13A—O2A	127.3 (3)	O1B—C13B—O2B	128.2 (3)
O1A—C13A—C14A	114.7 (2)	O1B—C13B—C14B	117.0 (3)
O2A—C13A—C14A	118.0 (2)	O2B—C13B—C14B	114.7 (3)
C13A—C14A—S1A	107.2 (2)	C13B—C14B—S1B	116.6 (2)
C13A—C14A—S3A	113.2 (2)	C13B—C14B—S3B	105.5 (2)
S3A—C14A—S1A	108.3 (1)	S3B—C14B—S1B	103.4 (1)
N2A—C15A—S1A	112.4 (2)	N2B—C15B—S1B	113.4 (2)
N2A—C15A—S2A	124.9 (2)	N2B—C15B—S2B	124.3 (3)
S2A—C15A—S1A	122.8 (2)	S2B—C15B—S1B	122.3 (2)
N3A—C18A—S3A	113.0 (3)	N3B—C18B—S3B	112.2 (2)
N3A—C18A—S4A	124.3 (3)	N3B—C18B—S4B	124.4 (2)
S4A—C18A—S3A	122.7 (2)	S4B—C18B—S3B	123.4 (2)

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

Data collection: *CAD-4 VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD-4 VAX/PC*. Data reduction: *Xtal3.0* (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994); *PLUTON* (Spek, 1994). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1009). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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7,7'-Bis[(aza-18-crown-6)carbonyl]thioindigo

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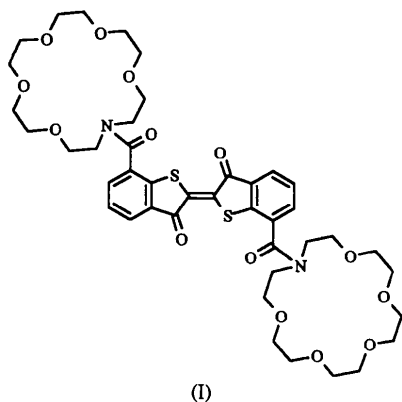
Abstract

The title compound, 7,7'-bis[(4,7,10,13,16-pentaoxa-1-azacyclooctadecane)carbonyl]thioindigo†, $C_{42}H_{54}N_2O_{14}S_2$, was found to be centrosymmetric and planar. Three members of the crown ether ring were disordered with roughly equal occupancy between two sets of positions.

Comment

Photoresponsive molecular hosts have received attention in the literature as light-controlled 'on-off switches' for supramolecular processes (Wyman & Brode, 1951; Kumar & Neckers, 1989). Azobenzene derivatives have been used to photoregulate enzyme activity (Westmark, Kelly & Smith, 1993), ion recognition and transport (Shinkai, Minami, Kusano & Manabe 1982), and membrane permeability (Aoyama, Watanabe & Inoue, 1990). Thioindigo derivatives have been studied as metal ion recognition and transport systems (Irie & Kato, 1985; Fatah-ur Rahman, Fukunishi, Kuwabara, Yamanaka & Nomura, 1993; Fatah-ur Rahman & Fukunishi, 1994), as dopants for photoresponsive media (Dinescu, Mezo, Luyt & Lemieux, 1994) and as materials for optical memory and switching (Saika, Iyoda, Honda & Shimidzu, 1992). We recently investigated the photochemistry of *N,N'*-diacylindigo dyes (Smith *et al.*, 1993, 1994). Our experience with indigo photochemistry and crowned recognition/transport systems (Bien, Shang & Smith, 1995) prompted us to prepare the title compound, (I), as a potential photoswitchable ionophore.

† IUPAC: 1,2-dihydro-2-[1,3-dihydro-3-oxo-7-[(4,7,10,13,16-pentaoxa-1-azacyclooctadecane)carbonyl]-1-thio-2*H*-indol-2-ylidene]-7-[(4,7,10,13,16-pentaoxa-1-azacyclooctadecane)carbonyl]-1-thio-3*H*-indol-3-one.



In its preferred *trans* configuration, the title compound absorbed at 540 nm. Irradiation of a chloroform solution with light of wavelength >530 nm produced a photo-stationary mixture of *ca* 1:1 *trans/cis*. In the dark, the *cis* configuration ($\lambda_{\max} = 485$ nm) spontaneously converted back to the *trans* with a rate constant $k = 6 \times 10^{-5} \text{ s}^{-1}$ at 323 K. In acetonitrile, this isomerization occurred about ten times faster.

The title compound is centrosymmetric and possesses inversion symmetry (Fig. 1). The thioindigo skeleton is essentially planar, deviations from the best least-squares plane being <0.07 Å. These results are in agreement with the X-ray structures of thioindigo (von Eller, 1955; Hasse-Wessel, Ohmasa & Süssle, 1977) and 7,7'-dimethylthioindigo (Süssle & Neuling, 1981). Steric effects force the 7,7'-bis(aza-18-crown-6)carbonyl groups from planarity [C(8)—C(7)—C(9)—O(2) and C(6)—C(7)—C(9)—O(1) torsion angles of $-45.0(5)$ and $127.8(4)^\circ$, respectively]. The aza-crown ether portion exhibited disorder at three positions, making structural details less certain [C(15)—C(15') 0.71(2), C(18)—C(18') 1.05(2), O(5)—O(5') 1.22(1) Å] (Fig. 2). Comparisons with reported crys-

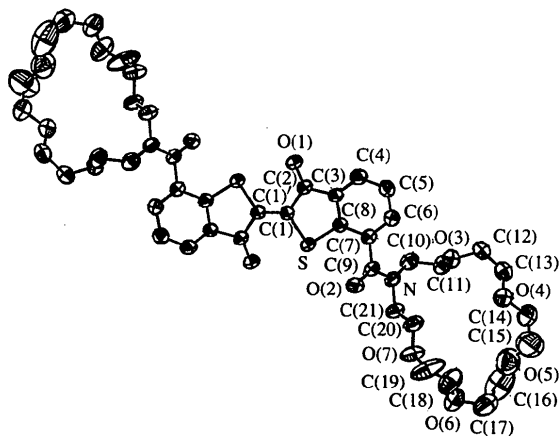


Fig. 1. Drawing of a single molecule of (I) showing 40% probability ellipsoids and the atom-labelling scheme.

tallographic studies on aza-crown derivatives revealed similar properties (Gokel & Garcia, 1977; Gokel *et al.*, 1987).

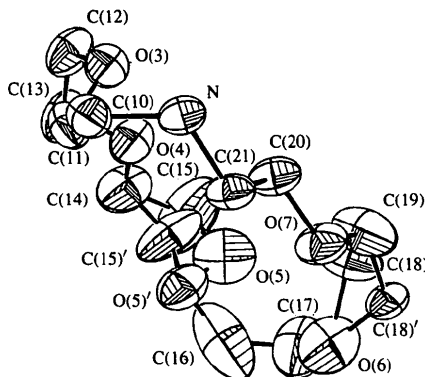


Fig. 2. Drawing of the disordered aza-18-crown group of (I) showing 40% probability ellipsoids and the atom-labelling scheme.

Experimental

1-Aza-18-crown-6 was acylated with 7,7'-bis(chlorocarbonyl)-thioindigo (Irie & Kato, 1985) in refluxing pyridine. Chromatography (SiO₂, 6:1 ethyl acetate:ethanol) afforded the title compound. Crystals were obtained by slow evaporation of a dilute benzene solution. Yield 50%, m.p. 406–408 K; ¹H NMR (300 MHz, CDCl₃/TMS): δ (p.p.m.) 7.93 (2H, *d*, $J = 7.5$ Hz), 7.68 (2H, *d*, $J = 7.5$ Hz), 7.38 (2H, *t*, $J = 7.5$ Hz), 3.70 (*bs*, 48H); ¹³C NMR (75 MHz, CDCl₃/TMS): δ (p.p.m.) 189.4, 168.2, 146.3, 133.9, 133.2, 132.9, 129.3, 127.0, 126.1, 70.6, 49.9, 45.9; IR (KBr): 1660, 1630, 1270, 1110 cm⁻¹; UV (acetone) $\lambda_{\max} = 540$ nm; MS (FAB) calculated for C₄₂H₅₅N₂O₁₄S₂ [MH⁺]: 876; found: 876.

Crystal data

C₄₂H₅₄N₂O₁₄S₂
 $M_r = 875.03$
 Triclinic
 $P\bar{1}$
 $a = 9.267(2)$ Å
 $b = 10.344(3)$ Å
 $c = 12.149(2)$ Å
 $\alpha = 97.29(2)^\circ$
 $\beta = 104.51(2)^\circ$
 $\gamma = 105.49(2)^\circ$
 $V = 1062.7(5)$ Å³
 $Z = 1$
 $D_x = 1.367$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 12\text{--}14^\circ$
 $\mu = 0.185$ mm⁻¹
 $T = 293$ K
 Plate
 $0.38 \times 0.17 \times 0.07$ mm
 Pink

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical *via* ψ scan (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.9671$, $T_{\max} = 0.9961$

$\theta_{\max} = 26^\circ$
 $h = -10 \rightarrow 0$
 $k = -11 \rightarrow 11$
 $l = -13 \rightarrow 13$
 3 standard reflections monitored every 200 reflections
 frequency: 120 min
 intensity decay: 2.9%

2955 measured reflections	1994 observed reflections
2955 independent reflections	$[I > 3\sigma(I)]$
<i>Refinement</i>	
Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.05085$	$(\Delta/\sigma)_{\max} = 0.03$
$wR = 0.06747$	$\Delta\rho_{\max} = 0.37(4) \text{ e } \text{\AA}^{-3}$
$S = 2.256$	$\Delta\rho_{\min} = -0.24(4) \text{ e } \text{\AA}^{-3}$
1994 reflections	Extinction correction: none
298 parameters	Atomic scattering factors
H-atom parameters not refined but located from difference Fourier maps	from Cromer & Waber (1974)

C(2)—C(3)—C(4)	127.2 (3)	C(7)—C(9)—O(2)	118.4 (3)
C(2)—C(3)—C(8)	111.9 (3)	C(7)—C(9)—N	120.6 (3)
C(4)—C(3)—C(8)	120.9 (3)	O(2)—C(9)—N	121.0 (4)
C(3)—C(4)—C(5)	119.9 (4)	C(9)—N—C(10)	125.1 (3)
C(4)—C(5)—C(6)	119.6 (4)	C(9)—N—C(21)	116.9 (3)
C(5)—C(6)—C(7)	121.4 (4)	C(10)—N—C(21)	117.7 (3)
C(6)—C(7)—C(8)	118.2 (3)		

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

H atoms attached to disordered C atoms were not located. Refinement on different space groups ($P\bar{1}$ versus $P1$) did not resolve the disorder. Examination and data collection were performed on an Enraf-Nonius CAD-4 computer-controlled κ -axis diffractometer equipped with a graphite crystal incident-beam monochromator. All calculations were performed on a VAX station 3200 computer using SDP/VAX (Frenz, 1978) software.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{eq}
C(1)	0.5600 (4)	0.5127 (3)	0.4767 (3)	3.59 (8)
C(2)	0.6444 (4)	0.4122 (3)	0.4563 (3)	3.76 (9)
O(1)	0.6132 (3)	0.2996 (2)	0.4819 (2)	5.21 (7)
C(3)	0.7664 (4)	0.4692 (3)	0.4031 (3)	3.50 (8)
C(4)	0.8652 (4)	0.4058 (4)	0.3687 (3)	4.32 (9)
C(5)	0.9732 (4)	0.4739 (4)	0.3187 (3)	4.5 (1)
C(6)	0.9834 (4)	0.6083 (4)	0.3058 (3)	4.35 (9)
C(7)	0.8854 (4)	0.6742 (3)	0.3403 (3)	3.81 (9)
C(8)	0.7715 (4)	0.6013 (3)	0.3864 (3)	3.49 (8)
S	0.6305 (1)	0.66343 (9)	0.43002 (9)	4.01 (2)
C(9)	0.8806 (4)	0.8133 (4)	0.3192 (3)	4.26 (9)
O(2)	0.7535 (3)	0.8264 (3)	0.2724 (3)	6.78 (8)
N	1.0129 (3)	0.9212 (3)	0.3529 (3)	4.22 (7)
C(10)	1.1636 (5)	0.9223 (4)	0.4265 (3)	4.8 (1)
C(11)	1.2960 (5)	0.9604 (4)	0.3737 (4)	5.5 (1)
O(3)	1.2560 (3)	0.8719 (3)	0.2652 (2)	5.28 (7)
C(12)	1.3898 (5)	0.8611 (5)	0.2301 (4)	6.8 (1)
C(13)	1.4975 (5)	0.9928 (5)	0.2233 (5)	7.5 (1)
O(4)	1.4198 (3)	1.0505 (3)	0.1385 (3)	6.71 (8)
C(14)	1.5291 (6)	1.1636 (6)	0.1180 (6)	9.3 (2)
C(15)	1.463 (2)	1.234 (2)	0.029 (1)	16.5 (5)
C(15)'	1.479 (1)	1.261 (1)	0.089 (1)	10.7 (3)
O(5)	1.370 (1)	1.2961 (9)	0.0568 (7)	11.2 (3)
O(5)'	1.4148 (8)	1.3324 (6)	0.1621 (6)	8.3 (2)
C(16)	1.388 (1)	1.444 (1)	0.1319 (7)	15.0 (3)
C(17)	1.2465 (7)	1.4557 (6)	0.0572 (6)	10.9 (2)
O(6)	1.1196 (4)	1.4112 (4)	0.1008 (3)	9.3 (1)
C(18)	1.041 (1)	1.258 (1)	0.027 (1)	8.9 (4)
C(18)'	0.976 (1)	1.3245 (8)	0.0390 (8)	5.7 (2)
C(19)	0.9517 (9)	1.1859 (5)	0.0588 (5)	10.5 (2)
O(7)	0.9664 (4)	1.1759 (3)	0.1758 (3)	6.32 (8)
C(20)	0.9969 (5)	1.0557 (4)	0.1996 (4)	5.5 (1)
C(21)	0.9983 (5)	1.0518 (4)	0.3232 (4)	4.5 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

C(1)—C(1')	1.350 (5)	C(6)—C(7)	1.384 (6)
C(1)—C(2)	1.489 (5)	C(7)—C(8)	1.404 (5)
C(1)—S	1.744 (4)	C(7)—C(9)	1.502 (5)
C(2)—O(1)	1.221 (4)	C(8)—S	1.764 (4)
C(2)—C(3)	1.466 (5)	C(9)—O(2)	1.223 (5)
C(3)—C(4)	1.373 (6)	C(9)—N	1.348 (4)
C(3)—C(8)	1.397 (5)	N—C(10)	1.454 (5)
C(4)—C(5)	1.379 (6)	N—C(21)	1.473 (5)
C(5)—C(6)	1.399 (6)		
C(1')—C(1)—C(2)	123.3 (3)	C(6)—C(7)—C(9)	124.0 (4)
C(1')—C(1)—S	124.3 (3)	C(8)—C(7)—C(9)	117.5 (3)
C(2)—C(1)—S	112.4 (3)	C(3)—C(8)—C(7)	119.9 (4)
C(1)—C(2)—O(1)	123.2 (4)	C(3)—C(8)—S	114.7 (3)
C(1)—C(2)—C(3)	110.1 (3)	C(7)—C(8)—S	125.5 (3)
O(1)—C(2)—C(3)	126.7 (4)	C(1)—S—C(8)	91.0 (2)

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: KA1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1 β -(4-Methoxybenzyl)-8 α -vinyl-7-oxa-bicyclo[4.3.0]nonan-6 β -ol

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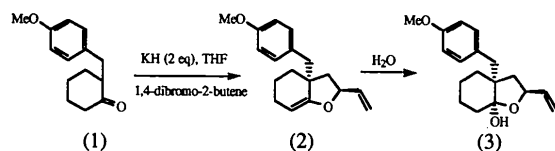
(Received 28 October 1994; accepted 20 June 1995)

Abstract

The crystal structure analysis of 1 β -(4-methoxybenzyl)-8 α -vinyl-7-oxabicyclo[4.3.0]nonan-6 β -ol, C₁₈H₂₄O₃, reveals that the unit cell consists of two dimers, each formed by the linkage of double head-to-head hydrogen bonds between hemiacetal groups.

Comment

In our search for novel methods of stereoselective formation of remote stereocenters, we developed a route for the synthesis of 4-hydroxyketone derivatives *via* temporarily formed bicyclic systems such as (2). Compound (2) was prepared from potassium hydride treatment of the 2-substituted cycloketone (1) and 1,4-dibromo-2-butene in tetrahydrofuran (Wang, Chen & Zhao, 1994), but the determination of the relative stereochemistry of (2) by ¹H and ¹³C NMR spectra was difficult. Recrystallization of the corresponding hydrate product, (3), from a mixture of cyclohexane and ethyl acetate gave colorless needles. A single-crystal X-ray analysis indicates that the vinyl group is located on the concave face of structure (3).



Structural features of the bicyclic rings are shown in Fig. 1. The cyclopentane and cyclohexane rings are *cis* fused. The dihedral angle between the cyclopentane ring and atoms C12, C13, C15 and C16 of the cyclohexane ring is 96.4°. The cyclohexane ring is in a distorted chair conformation with atoms C2 and O3 at equatorial positions, and atoms C17 and O1 at axial positions. The torsion angle C12—C13—C14—C15 on one side of the distorted ring is -59.9°, which exceeds the mean value of 52.9° by 7.0°. There is a compensation of 8.3° on the other side (C12—C11—C16—C15) from a flattening effect. The cyclopentane ring adopts a half-chair conformation [torsion angles ω_1 (C11—O1—C1—C2), ω_2 (O1—C1—C2—C16), ω_3 (C1—C2—C16—C11), ω_4 (C2—C16—C11—O1) and ω_5 (C16—C11—O1—C1) of 16.5, 10.3, -30.4, 40.8 and -36.6°, respectively], which is also distorted from the ideal half-chair conformation of cyclopentane [torsion angles $\omega_1 = 14.4$, $\omega_2 = 14.4$, $\omega_3 = -37.8$, $\omega_4 = 46.7$ and $\omega_5 = -37.8^\circ$] (Geise, Altona & Romers, 1967; Altona, Geise & Romers, 1968). The C3=C4 double bond is approximately parallel to the O1—C1 bond, with a torsion angle O1—C1—C3=C4 of 3.2°.

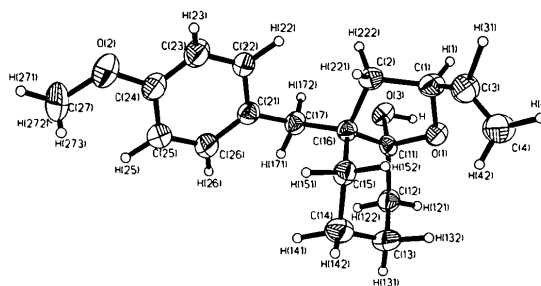


Fig. 1. The molecular structure and atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

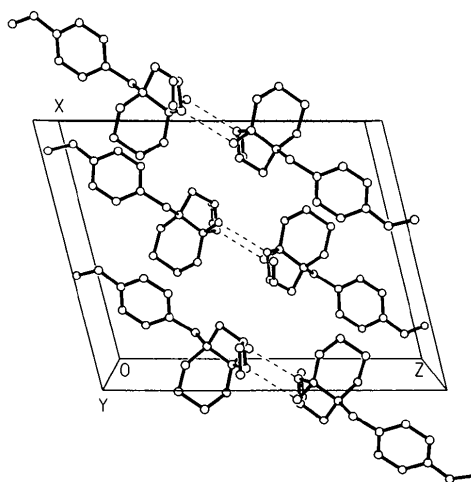


Fig. 2. The packing arrangement as viewed along *b*. Hydrogen bonds are shown by dashed lines.