

Tabelle 1. Atomkoordinaten und isotrope äquivalente Verschiebungsparameter (Å<sup>2</sup>)
$$U_{\text{äq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{äq}}$
C1	0,6300 (5)	0,6057 (2)	0,3459 (3)	0,0407 (11)
C2	0,6229 (5)	0,5299 (2)	0,3345 (3)	0,0374 (11)
C3	0,4704 (4)	0,5007 (2)	0,2935 (2)	0,0293 (9)
C4	0,3886 (5)	0,4491 (2)	0,3588 (3)	0,0377 (11)
C5	0,2258 (5)	0,4363 (2)	0,3236 (3)	0,0409 (11)
C6	0,2299 (6)	0,4094 (2)	0,2147 (3)	0,0483 (13)
C7	0,3164 (5)	0,4553 (2)	0,1448 (3)	0,0384 (11)
C8	0,4824 (5)	0,4717 (2)	0,1829 (3)	0,0305 (9)
C9	0,5576 (4)	0,5186 (2)	0,1028 (3)	0,0300 (9)
C10	0,4909 (5)	0,5885 (2)	0,0917 (3)	0,0318 (9)
C11	0,4965 (5)	0,6297 (2)	0,1870 (3)	0,0309 (9)
C12	0,3673 (5)	0,6451 (2)	0,2353 (3)	0,0303 (10)
C13	0,3764 (5)	0,6700 (2)	0,3425 (3)	0,0372 (11)
C14	0,4935 (5)	0,6328 (2)	0,4075 (3)	0,0448 (11)
C15	0,6479 (5)	0,6425 (2)	0,2418 (3)	0,0377 (11)
C16	0,7982 (5)	0,6211 (2)	0,1896 (3)	0,0511 (13)
C17	0,6700 (5)	0,7179 (2)	0,2577 (3)	0,0535 (13)
C18	0,2078 (5)	0,6361 (2)	0,1920 (3)	0,0379 (11)
C19	0,5850 (6)	0,4103 (2)	0,1840 (3)	0,0428 (12)
C20	0,4583 (6)	0,3956 (2)	0,4191 (3)	0,0500 (13)
O1	0,6667 (3)	0,50387 (15)	0,4326 (2)	0,0521 (8)
O2	0,3979 (4)	0,45467 (14)	0,4693 (2)	0,0497 (9)
O3	0,1374 (3)	0,49486 (15)	0,3257 (2)	0,0469 (8)
O4	0,5422 (3)	0,48775 (12)	0,0045 (2)	0,0346 (7)
O5	0,5872 (3)	0,61810 (12)	0,0115 (2)	0,0383 (7)
C21	0,5290 (6)	0,6710 (2)	-0,0363 (3)	0,0437 (12)
O6	0,4054 (4)	0,69413 (14)	-0,0174 (2)	0,0548 (9)
C22	0,6392 (6)	0,6953 (2)	-0,1144 (3)	0,065 (2)
O7	0,2304 (3)	0,66016 (13)	0,3956 (2)	0,0426 (8)
C23	0,1407 (5)	0,7133 (2)	0,4094 (3)	0,0428 (11)
O8	0,1747 (4)	0,76708 (15)	0,3792 (3)	0,0731 (12)
C24	0,0017 (6)	0,6953 (2)	0,4667 (3)	0,0570 (14)

Tabelle 2. Torsionswinkel (°)

C1—C2—C3—C4	-123,5	C10—C11—C12—C13	163,9
C1—C2—C3—C8	109,9	C10—C11—C12—C18	-12,7
C2—C3—C4—C5	166,7	C10—C9—C8—C19	-173,6
C2—C3—C4—C20	-38,9	C10—C11—C15—C1	-113,4
C2—C3—C4—O2	34,8	C10—C11—C15—C16	8,8
C2—C3—C8—C7	-170,5	C10—C11—C15—C17	127,2
C2—C3—C8—C9	-50,2	C11—C12—C13—C14	-41,3
C2—C3—C8—C19	67,5	C11—C12—C13—O7	-158,1
C3—C4—C5—C6	61,8	C11—C10—C9—O4	-179,8
C3—C4—C5—O3	58,6	C11—C10—O5—C21	-76,3
C3—C4—C20—O2	104,5	C11—C15—C1—C2	67,2
C3—C4—O2—C20	-119,4	C11—C15—C1—C14	-60,1
C3—C8—C7—C6	-56,0	C12—C13—C14—C1	30,1
C3—C8—C9—C10	-53,3	C12—C13—O7—C23	-104,2
C3—C8—C9—O4	-173,1	C12—C11—C15—C16	176,4
C4—C5—C6—C7	-55,7	C12—C11—C15—C17	-65,2
C4—C3—C2—O1	-0,7	C12—C11—C15—C1	54,2
C4—C3—C8—C7	57,4	C12—C11—C10—O5	132,9
C4—C3—C8—C9	177,8	C13—C14—C1—C2	-107,9
C4—C3—C8—C19	-64,6	C13—C14—C1—C15	20,2
C5—C6—C7—C8	54,7	C13—C12—C11—C15	-3,8
C5—C4—C3—C8	-63,5	C14—C1—C2—C3	51,8
C5—C4—C20—O2	-101,6	C14—C1—C2—O1	-75,0
C5—C4—O2—C20	107,3	C14—C1—C15—C16	171,9
C6—C7—C8—C9	179,4	C14—C1—C15—C17	58,1
C6—C7—C8—C19	64,7	C14—C13—C12—C18	135,6
C6—C5—C4—C20	-96,0	C14—C13—O7—C23	133,8
C6—C5—C4—O2	-162,0	C15—C1—C2—C3	-75,2
C7—C8—C9—C10	67,3	C15—C1—C2—O1	158,1
C7—C8—C9—O4	-52,5	C15—C11—C12—C18	179,6
C7—C6—C5—O3	66,4	C15—C11—C10—O5	-59,7
C8—C9—C10—C11	59,9	C16—C15—C1—C2	-60,8
C8—C9—C10—O5	-180,0	C17—C15—C1—C2	-174,6
C8—C3—C2—O1	-127,2	C18—C12—C13—O7	18,8
C8—C3—C4—C20	91,0	C19—C8—C9—O4	66,6
C8—C3—C4—O2	163,7	C20—C4—C5—O3	143,5

C9—C10—C11—C12	-111,0	O3—C5—C4—O2	77,5
C9—C10—C11—C15	56,3	O4—C9—C10—O5	-59,7
C9—C10—O5—C21	160,5		

Die Listen der Strukturparameter, anisotropen Verschiebungsparameter, H-Atom Koordinaten, und vollständigen geometrischen Daten sind bei der IUCr (Aktenzeichen: SE1076) hinterlegt. Kopien sind erhältlich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 1,4,7,10,13-Pentaoxa-16,19-dithiacyclohenicosane Sesquihydrate at 150 K

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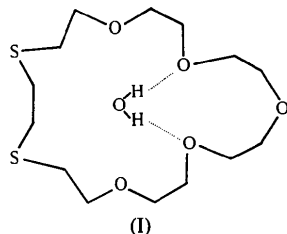
## Abstract

Molecules of 1,4,7,10,13-pentaoxa-16,19-dithiacyclohenicosane, [21]aneS<sub>2</sub>O<sub>5</sub>, are linked in the sesquihydrate, C<sub>14</sub>H<sub>28</sub>O<sub>5</sub>S<sub>2</sub>·3/2H<sub>2</sub>O, via intermolecular O···H—O hydrogen-bonding interactions with the water of crystallization. The conformation of the macrocycle is discussed in terms of its potential as a multifunctional ligand.

## Comment

The crystal structure of the title compound consists of two crystallographically independent [21]aneS<sub>2</sub>O<sub>5</sub> macrocycles (Fig. 1) linked via hydrogen-bonding interactions to three crystallographically independent water molecules. Each molecule of [21]aneS<sub>2</sub>O<sub>5</sub> is associated with one molecule of water, bridging O10 and O16 or O10' and O16' [see (I) below]. One hydrated macrocycle, [21]aneS<sub>2</sub>O<sub>5</sub>·H<sub>2</sub>O (labelled with primes in Figs. 1 and 2 and Tables 1 and 2), is fur-

ther linked to another through interactions of O19' and O2W with a pair of water molecules (O3W) (Fig. 2) to form a centrosymmetric hydrogen-bonded dimer. Hydrogen-bonded O...O distances lie between 2.813 (7) and 3.215 (7) Å, and therefore fall in the range anticipated for unsymmetrical interactions of this type (Wells, 1985).



While the lengths of the C—S and C—O bonds [1.783 (7)–1.810 (7) and 1.405 (7)–1.437 (7) Å, respectively] have typical values (1.819 and 1.426 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1992), the C—C bonds [1.484 (9)–1.510 (9) Å] are somewhat shorter than is usual for  $C_{sp^3}$ — $C_{sp^3}$  linkages (1.54 Å), although this is frequently observed in crystallographic studies of related crown ethers (Dunitz, Dobler, Seiler & Phizackerley, 1974). Chemically equivalent bond lengths and angles in the ordered region of the structure are not significantly different. Each macrocycle adopts a conformation in which the O atoms O10 and O16 occupy *endo*-dentate positions, while the S atoms are *exo*-dentate. The conformations about the C—C bonds are controlled by 1,4-interactions between heteroatoms. The O—C—O fragments all adopt approximately *gauche* conformations (62.0–69.7°), while the S—C—C—S and S—C—C—O torsion angles are 172.8 and 175.8–179.2°, respectively (in the ordered regions of the structure).

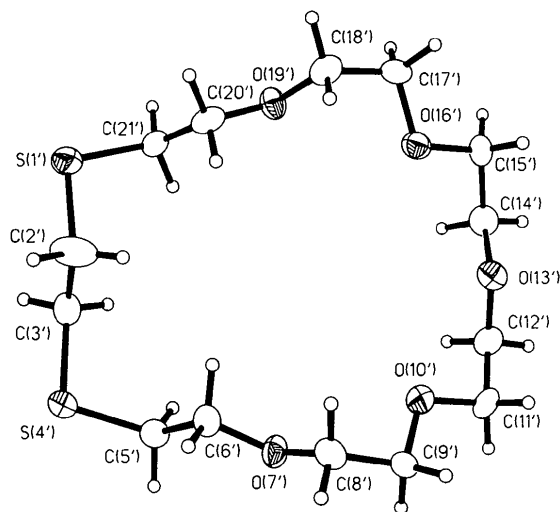


Fig. 1. The structure of the [21]aneS<sub>2</sub>O<sub>5</sub> macrocycle. There are two such molecules in the asymmetric unit, the other exhibits disorder of C2. Displacement ellipsoids enclose 50% probability surfaces.

The C—C—O—C torsion angles in both macrocycles correspond to approximately *anti* conformations (163.6–177.4°) such that unfavourable H...H interactions between 1,4-methylene groups are avoided. The longer C—S bonds remove this constraint in the C—C—S—C linkages, which therefore display smaller torsion angles in order to accommodate the cyclic nature of the [21]-aneS<sub>2</sub>O<sub>5</sub> molecule (73.7–97.4° in the ordered regions of the structure). There are no significant differences between chemically equivalent torsion angles in the ordered regions of the structure and the overall conformation of the macrocycle is thus [334344] (Dale, 1973).

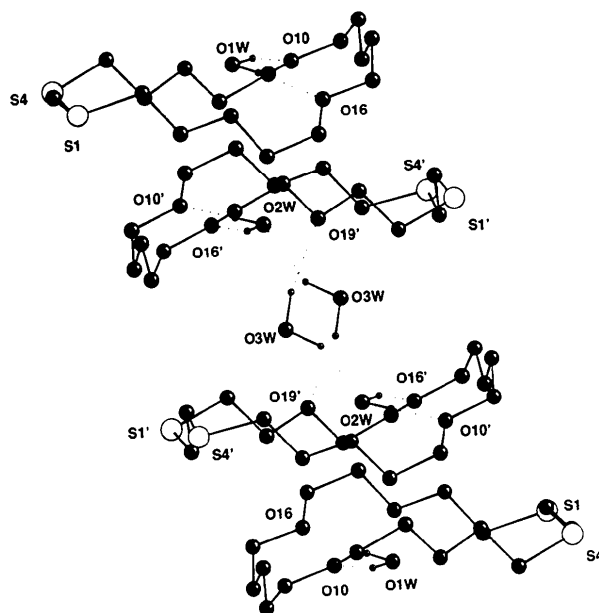


Fig. 2. Hydrogen-bond formation in the crystal structure of [21]-aneS<sub>2</sub>O<sub>5</sub>·3/2H<sub>2</sub>O.

Although the conformation adopted by the [21]-aneS<sub>2</sub>O<sub>5</sub> macrocycle is consistent with structural trends established previously for this class of compound, the configuration described here is likely to be related to the presence of water of crystallization. Mean-plane analysis shows that the seven heteroatoms in [21]aneS<sub>2</sub>O<sub>5</sub> are essentially coplanar, so that O7, O10, O16 and O19 all have lone pairs directed towards a common point such that they may act simultaneously as donor sites. Since the S atoms are also potential complexation sites, this macrocycle is of interest as a multifunctional ligand, and we are currently engaged in studies of its ligation behaviour in the presence of transition metal ions.

## Experimental

The title compound was prepared according to the literature procedure (Bradshaw, Reeder, Thompson, Flanders, Carruth, Izatt & Christensen, 1976). The crude product was purified

by washing a dichloromethane solution with water. Following removal of the dichloromethane, the residue was redissolved in ethyl acetate and the solution carefully layered with *n*-hexane. The sample vial was sealed and cooled to 243 K, causing large crystals to grow within 2–4 days. The selected crystal was cooled using an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986).

### Crystal data

C<sub>14</sub>H<sub>28</sub>O<sub>5</sub>S<sub>2</sub>·3/2H<sub>2</sub>O

*M<sub>r</sub>* = 367.5

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 17.550 (12) Å

*b* = 11.750 (8) Å

*c* = 18.531 (9) Å

β = 91.52 (4)°

*V* = 3820 (4) Å<sup>3</sup>

*Z* = 8

*D<sub>x</sub>* = 1.278 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 30

reflections

θ = 13–14°

μ = 0.295 mm<sup>-1</sup>

*T* = 150.0 (2) K

Column

0.40 × 0.40 × 0.20 mm

Colourless

### Data collection

Stoe Stadi-4 four-circle diffractometer

ω–2θ scans [ω half-width (1.5 + 0.35tanθ)°]

Absorption correction:

ψ scans (North, Phillips & Mathews, 1968)

*T<sub>min</sub>* = 0.716, *T<sub>max</sub>* = 0.873

5424 measured reflections

4976 independent reflections

3064 observed reflections [*I* > 2σ(*I*)]

*R<sub>int</sub>* = 0.1406

θ<sub>max</sub> = 22.51°

*h* = –18 → 18

*k* = –1 → 12

*l* = 0 → 19

3 standard reflections

frequency: 60 min

intensity decay: 2%

### Refinement

Refinement on *F*<sup>2</sup>

*R*(*F*) = 0.0714

*wR*(*F*<sup>2</sup>) = 0.2805

*S* = 1.097

4861 reflections

423 parameters

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0831*P*)<sup>2</sup> + 14.20*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.345

Δρ<sub>max</sub> = 0.52 e Å<sup>-3</sup>

Δρ<sub>min</sub> = –0.41 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

*U*<sub>iso</sub> for C2a and C2b; *U*<sub>eq</sub> = (1/3)Σ<sub>i</sub>Σ<sub>j</sub>U<sub>ij</sub>*a<sub>i</sub>*\**a<sub>j</sub>* for others.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> / <i>U</i> <sub>iso</sub>
S1	0.38865 (12)	0.0792 (2)	0.21888 (10)	0.0448 (5)
C2a†	0.3937 (9)	0.1011 (13)	0.1243 (4)	0.044 (4)
C2b†	0.4124 (7)	0.0512 (17)	0.1249 (5)	0.051 (5)
C3	0.3458 (5)	0.0305 (10)	0.0773 (5)	0.080 (3)
S4	0.36427 (14)	0.0550 (2)	–0.01810 (11)	0.0553 (6)
C5	0.3958 (4)	–0.0817 (6)	–0.0485 (4)	0.040 (2)
C6	0.3317 (4)	–0.1658 (6)	–0.0559 (4)	0.038 (2)
O7	0.3618 (2)	–0.2699 (4)	–0.0814 (2)	0.0351 (12)
C8	0.3031 (4)	–0.3500 (6)	–0.0974 (4)	0.033 (2)
C9	0.3352 (4)	–0.4665 (6)	–0.1031 (3)	0.034 (2)
O10	0.3694 (2)	–0.4972 (4)	–0.0356 (2)	0.0311 (11)
C11	0.3923 (4)	–0.6121 (5)	–0.0335 (4)	0.032 (2)
C12	0.4406 (4)	–0.6336 (6)	0.0336 (4)	0.035 (2)

O13	0.3968 (2)	–0.6155 (4)	0.0955 (2)	0.0284 (11)
C14	0.4432 (4)	–0.6197 (6)	0.1597 (3)	0.036 (2)
C15	0.3974 (4)	–0.5940 (6)	0.2238 (4)	0.038 (2)
O16	0.3764 (3)	–0.4785 (4)	0.2228 (2)	0.0350 (12)
C17	0.3419 (4)	–0.4429 (6)	0.2864 (3)	0.033 (2)
C18	0.3146 (4)	–0.3253 (6)	0.2782 (4)	0.032 (2)
O19	0.3781 (2)	–0.2507 (4)	0.2721 (2)	0.0347 (12)
C20	0.3524 (4)	–0.1418 (6)	0.2479 (4)	0.039 (2)
C21	0.4197 (4)	–0.0600 (6)	0.2481 (4)	0.042 (2)
S1'	0.15677 (12)	0.1705 (2)	0.02431 (9)	0.0422 (5)
C2'	0.1685 (4)	0.1804 (8)	–0.0715 (4)	0.050 (2)
C3'	0.0973 (4)	0.1601 (6)	–0.1150 (4)	0.037 (2)
S4'	0.11010 (10)	0.15244 (15)	–0.21150 (9)	0.0331 (5)
C5'	0.0859 (4)	0.2934 (5)	–0.2416 (4)	0.030 (2)
C6'	0.1510 (4)	0.3765 (5)	–0.2383 (4)	0.030 (2)
O7'	0.1247 (2)	0.4799 (4)	–0.2683 (2)	0.0308 (11)
C8'	0.1835 (4)	0.5590 (6)	–0.2783 (4)	0.036 (2)
C9'	0.1511 (4)	0.6747 (6)	–0.2893 (3)	0.037 (2)
O10'	0.1205 (3)	0.7127 (4)	–0.2237 (2)	0.0372 (12)
C11'	0.0912 (4)	0.8250 (6)	–0.2270 (4)	0.039 (2)
C12'	0.0516 (4)	0.8479 (6)	–0.1583 (3)	0.036 (2)
O13'	0.1048 (2)	0.8481 (4)	–0.0989 (2)	0.0316 (11)
C14'	0.0670 (4)	0.8526 (6)	–0.0323 (3)	0.035 (2)
C15'	0.1237 (4)	0.8406 (5)	0.0293 (4)	0.034 (2)
O16'	0.1535 (2)	0.7282 (4)	0.0287 (2)	0.0312 (11)
C17'	0.1888 (4)	0.6961 (6)	0.0965 (3)	0.035 (2)
C18'	0.2211 (4)	0.5799 (6)	0.0897 (4)	0.034 (2)
O19'	0.1608 (2)	0.5004 (4)	0.0762 (2)	0.0294 (11)
C20'	0.1892 (3)	0.3952 (6)	0.0522 (4)	0.031 (2)
C21'	0.1252 (4)	0.3094 (5)	0.0481 (4)	0.030 (2)
O1W	0.4067 (3)	–0.3207 (5)	0.0931 (3)	0.0548 (15)
O2W	0.1144 (3)	0.5684 (4)	–0.0944 (2)	0.0403 (12)
O3W	0.0033 (3)	0.5933 (4)	0.0859 (3)	0.0447 (13)

† Occupancy = 0.5.

Table 2. Selected geometric parameters (Å, °)

S1'–C21'	1.783 (7)	C11'–C12'	1.490 (10)
S1'–C2'	1.796 (7)	C12'–O13'	1.423 (8)
C2'–C3'	1.489 (10)	O13'–C14'	1.419 (8)
C3'–S4'	1.810 (7)	C14'–C15'	1.502 (9)
S4'–C5'	1.795 (7)	C15'–O16'	1.420 (8)
C5'–C6'	1.503 (9)	O16'–C17'	1.437 (7)
C6'–O7'	1.409 (7)	C17'–C18'	1.484 (9)
O7'–C8'	1.405 (7)	C18'–O19'	1.429 (8)
C8'–C9'	1.485 (10)	O19'–C20'	1.409 (8)
C9'–O10'	1.415 (8)	C20'–C21'	1.510 (9)
O10'–C11'	1.416 (8)		
C21'–S1'–C2'	103.2 (4)	O13'–C12'–C11'	110.4 (6)
C3'–C2'–S1'	114.1 (5)	C14'–O13'–C12'	111.1 (5)
C2'–C3'–S4'	114.8 (5)	O13'–C14'–C15'	110.0 (5)
C5'–S4'–C3'	103.1 (3)	O16'–C15'–C14'	108.5 (5)
C6'–C5'–S4'	114.4 (5)	C15'–O16'–C17'	112.8 (5)
O7'–C6'–C5'	107.7 (5)	O16'–C17'–C18'	108.9 (5)
C8'–O7'–C6'	112.9 (5)	O19'–C18'–C17'	109.5 (5)
O7'–C8'–C9'	110.1 (6)	C20'–O19'–C18'	111.2 (5)
O10'–C9'–C8'	108.9 (5)	O19'–C20'–C21'	109.4 (5)
C9'–O10'–C11'	113.7 (5)	C20'–C21'–S1'	112.9 (5)
O10'–C11'–C12'	108.0 (5)		
C21'–S1'–C2'–C3'	–85.2 (7)		
S1'–C2'–C3'–S4'	–172.8 (4)		
C2'–C3'–S4'–C5'	–97.4 (6)		
C3'–S4'–C5'–C6'	86.7 (5)		
S4'–C5'–C6'–O7'	175.8 (4)		
C5'–C6'–O7'–C8'	–170.9 (5)		
C6'–O7'–C8'–C9'	–163.6 (5)		
O7'–C8'–C9'–O10'	69.4 (7)		
C8'–C9'–O10'–C11'	177.1 (6)		
C9'–O10'–C11'–C12'	172.3 (6)		
O10'–C11'–C12'–O13'	65.5 (7)		
C11'–C12'–O13'–C14'	–171.2 (6)		
C12'–O13'–C14'–C15'	174.2 (5)		
O13'–C14'–C15'–O16'	–68.2 (7)		
C14'–C15'–O16'–C17'	–161.4 (5)		
C15'–O16'–C17'–C18'	–177.4 (5)		
O16'–C17'–C18'–O19'	–64.1 (7)		

C17'—C18'—O19'—C20'	165.7 (5)
C18'—O19'—C20'—C21'	172.6 (5)
O19'—C20'—C21'—S1'	-175.8 (4)
C2'—S1'—C21'—C20'	-77.9 (5)

All non-H atoms in the two crystallographically independent macrocycles were located by direct methods (SHELXS86; Sheldrick, 1990). A difference synthesis calculated on the basis of this phasing model exhibited three peaks of  $ca\ 7\ e\ \text{\AA}^{-3}$ , which were assigned to O atoms of water of crystallization, the H atoms of which were located in a difference synthesis performed after several cycles of least-squares refinement weighted towards the high-angle data. They were refined with O—H and H...H restrained to chemically reasonable values of 1.000 (5) and 1.59 (1) Å, respectively. Other H atoms were placed in calculated positions (C—H = 0.99 Å) and allowed to ride on their neighbouring C atoms with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Full-matrix isotropic refinement caused atom C2 to develop a displacement parameter approximately twice as large as those of the other C atoms in the macrocycle. On anisotropic refinement, C2 developed an elongated displacement ellipsoid which was interpreted as the result of C2 being disordered over two positions, C2a and C2b, with equal occupancies. Chemically reasonable restraints were placed on the geometry of the S1—C2a,b—C3—S4 units [C—C = 1.48 (1), C—S = 1.80 (1) Å]. However, even under these conditions the disordered C atoms were still highly anisotropic; it seems reasonable to conclude that the disorder is more extensive than modelled here, and C2a and C2b were therefore refined with isotropic displacement parameters.

Data collection: DIF4 (Stoe & Cie, 1990a). Cell refinement: DIF4. Data reduction: REDU4 (Stoe & Cie, 1990b). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1992), CAMERON (Pearce & Watkin, 1993). Software used to prepare material for publication: CALC (Gould & Taylor, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1168). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 2-(10-Diethylaminomethyl-2,3,6,7-tetra-methoxy-9-phenanthrylmethyl)-1-pyrroli-dinecarbonitrile

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## Abstract

The title molecule,  $\text{C}_{29}\text{H}_{37}\text{N}_3\text{O}_4$ , consists of a pyrrolidine and a phenanthrene ring. The mean plane through the pyrrolidine ring atoms is almost perpendicular to that through the phenanthrene ring atoms. The pyrrolidine ring adopts a half-chair conformation, while the fused rings of the phenanthrene system are almost coplanar. In the crystal, molecules are stabilized by van der Waals interactions.

## Comment

The alkaloid tylophorine, isolated from *tylophora asthmatica*, contains the interesting phenanthraindolizidine system. There are conflicting reports in the literature on the absolute configuration of tylophorine (Ferguson & Robertson, 1963; Barth & Lawton, 1971), which has a chiral centre, as derived from degradation studies and synthesis. Unfortunately, tylophorine crystals were not suitable for X-ray investigation. However, a degradation product obtained from tylophorine by treatment with cyanogen bromide followed by diethylamine yielded good single crystals. It was our aim to determine the absolute configuration of the title compound, (I), which