Monoclinic
$P2_1/n$
$a = 9.7987\,(8)$ Å
$b = 19.1424\,(14)$ Å
$c = 13.3366\,(11)$ Å
$\beta = 98.816\,(7)^\circ$
$V = 2472.0\,(3)$ Å^3
$Z = 4$
$D_x = 1.250$ Mg m^{-3}
D_m not measured

Cell parameters from 74
  reflections
$\theta = 30.1-39.7^\circ$
$\mu = 3.578$ mm^{-1}
$T = 160\,(2)$ K
Prism
$0.50 \times 0.29 \times 0.15$ mm
Colourless

Data collection

Stoe Stadi-4 four-circle
  diffractometer
ω/θ scans
Absorption correction:
  ψ scans (North *et al.*,
  1968)
  $T_{min} = 0.338$, $T_{max} = 0.585$
8127 measured reflections
4091 independent reflections

3693 reflections with
  $I > 2\sigma(I)$
$R_{int} = 0.0229$
$\theta_{max} = 64.39^\circ$
$h = -11 \rightarrow 11$
$k = -22 \rightarrow 22$
$l = -14 \rightarrow 15$
3 standard reflections
  frequency: 60 min
  intensity decay: 3.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.090$
$S = 1.054$
4091 reflections
315 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 1.2956P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.032$
$\Delta\rho_{max} = 0.513$ e Å^{-3}
$\Delta\rho_{min} = -0.377$ e Å^{-3}
Extinction correction: none
Scattering factors from
  International Tables for
  Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

B1—B2	1.757 (3)	B4—B9	1.758 (3)
B1—B4	1.760 (3)	B4—B8	1.761 (3)
B1—B5	1.775 (3)	B4—B10	1.764 (3)
B1—B10	1.785 (3)	B4—Cl4	1.827 (2)
B1—B3	1.838 (3)	B5—B6	1.878 (3)
B2—B6	1.742 (3)	B5—B10	1.879 (3)
B2—B3	1.758 (3)	B6—B7	1.846 (3)
B2—B5	1.764 (3)	B6—P1	1.922 (2)
B2—B7	1.767 (3)	B7—B8	1.886 (3)
B2—Cl2	1.823 (2)	B8—B9	1.887 (3)
B3—B4	1.750 (3)	B9—B10	1.888 (3)
B3—B7	1.779 (3)	B9—P2	1.924 (2)
B3—B8	1.785 (3)		
B6—B2—B1	116.24 (15)	B10—B4—Cl4	119.17 (14)
B6—B2—B3	115.40 (16)	B6—B5—B10	112.66 (14)
B6—B2—Cl2	115.57 (13)	B7—B6—B5	104.85 (14)
B1—B2—Cl2	118.38 (14)	B2—B6—P1	117.38 (14)
B3—B2—Cl2	118.05 (14)	B7—B6—P1	122.38 (13)
B5—B2—Cl2	119.64 (14)	B5—B6—P1	120.33 (13)
B7—B2—Cl2	118.87 (14)	B6—B7—B8	113.42 (14)
B3—B4—B9	118.21 (16)	B7—B8—B9	115.68 (14)
B9—B4—B1	118.20 (16)	B8—B9—B10	102.33 (14)
B3—B4—Cl4	118.63 (14)	B4—B9—P2	157.66 (15)
B9—B4—Cl4	112.19 (13)	B8—B9—P2	111.47 (13)
B1—B4—Cl4	118.45 (14)	B10—B9—P2	111.90 (13)
B8—B4—Cl4	118.98 (14)	B5—B10—B9	116.25 (14)

B—B—B acute angles are in the range 57.05 (12)–64.85 (13)°.

Phosphine H atoms were constrained to idealized positions with a riding model including free rotation of the methyl groups and with PPh units restrained to have C_{2v} symmetry.

Borane H atoms were located in difference Fourier maps and all associated parameters were refined freely.

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(1R,7S,8aS)-1,7-Dihydroxyindolizidine Hydrate

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Abstract

The title compound [(1R,7S,8aS)-1,7-dihydroxyperhydroindolizine hydrate, $C_8H_{15}NO_2.H_2O$] crystallizes with one water solvate molecule, which is involved in strong hydrogen bonds with the hydroxyl O atoms. Bond lengths and angles in the molecule are in agreement with literature values.

Comment

Several polyhydroxylated indolizidines show interesting biological activity as inhibitors of glycosidases (Elbein & Molineux, 1987), key enzymes involved in the cell–cell recognition process. Numerous investigations have been reported in the attempt to correlate their structure with their biological activity (Hempel *et al.*, 1993; Brandi *et al.*, 1995). In the course of our investigations, we have synthesized the title compound, (I) (Cicchi *et al.*, 1995), and determined its crystal structure by X-ray diffraction.

(I)
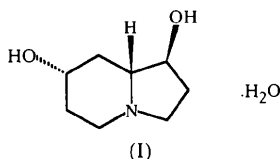
The compound crystallizes with one water solvate molecule, which plays an important role in the packing of the molecules by making strong hydrogen bonds with the hydroxyl O atoms [O···O1^i 2.774 (3), O···O7^{ii} 2.789 (3) and O7···O^{iii} 2.682 (3) Å; symmetry codes: (i) $-x$, $y - \frac{1}{2}$, $\frac{3}{2} - z$; (ii) $x - \frac{1}{2}$, $\frac{1}{2} - y$, $1 - z$; (iii) $1 + x$, y, z]. An additional O1···N^{iv} hydrogen bond of 2.794 (3) Å completes the three-dimensional hydrogen-bond network [symmetry code: (iv) $-x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$]. Bond lengths and angles in the molecule are in agreement with values from the literature (Hempel *et al.*, 1993). The six-membered ring adopts the inverted chair conformation [$\theta = 175.6$ (2)°; Cremer & Pople, 1975].
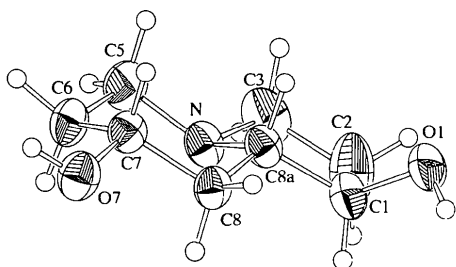

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of $C_8H_{15}NO_2$. Displacement ellipsoids are shown at the 50% probability level for non-H atoms.

Experimental

The title compound was prepared according to Cicchi *et al.* (1995) and was recrystallized from tetrahydrofuran.

Crystal data

$C_8H_{15}NO_2.H_2O$
$M_r = 175.23$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Orthorhombic
$P2_12_12_1$
$a = 7.796$ (3) Å
$b = 9.627$ (4) Å
$c = 12.792$ (4) Å
$V = 960.0$ (4) Å^3
$Z = 4$
$D_x = 1.212$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 17$–20°
$\mu = 0.092$ mm^{-1}
$T = 293$ (2) K
Prism
$0.5 \times 0.4 \times 0.4$ mm
Transparent, colourless

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1616 measured reflections
1616 independent reflections
1333 reflections with
$I > 2\sigma(I)$

$\theta_{max} = 29.97$°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 17$
3 standard reflections
every 500 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.131$
$S = 1.062$
1616 reflections
162 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.0582P]$
where $P = F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.011$
$\Delta\rho_{max} = 0.225$ e Å^{-3}
$\Delta\rho_{min} = -0.185$ e Å^{-3}
Extinction correction:
SHELXL93
Extinction coefficient:
0.016 (7)
Scattering factors from
International Tables for
Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C5—N	1.459 (3)	C8a—N	1.469 (2)
C5—C6	1.515 (3)	C8a—C1	1.521 (3)
C6—C7	1.527 (3)	C1—O1	1.417 (2)
C7—O7	1.422 (2)	C1—C2	1.538 (4)
C7—C8	1.520 (3)	C2—C3	1.508 (5)
C8—C8a	1.517 (3)	C3—N	1.469 (3)
N—C5—C6	109.5 (2)	O1—C1—C8a	111.5 (2)
C5—C6—C7	111.0 (2)	O1—C1—C2	112.2 (2)
O7—C7—C8	107.7 (2)	C8a—C1—C2	103.2 (2)
O7—C7—C6	111.9 (2)	C3—C2—C1	106.0 (2)
C8—C7—C6	110.6 (2)	N—C3—C2	103.7 (2)
C8a—C8—C7	109.6 (2)	C5—N—C8a	110.9 (2)
N—C8a—C8	109.71 (14)	C5—N—C3	116.2 (2)
N—C8a—C1	103.4 (2)	C8a—N—C3	103.3 (2)
C8—C8a—C1	117.6 (2)		

All non-H atoms were refined anisotropically. H atoms were located in the difference Fourier map and were refined isotropically, with a common displacement parameter.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1992). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Polysulfonylamines. CVI.† 1,4,7,10,13,16-Hexaoxacyclooctadecane–Dimethyl Sulfoxide–Bis(4-fluorophenylsulfonyl)amine (1/2/2)

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Abstract

In the centrosymmetric formula unit of the title complex, $C_{12}H_{24}O_6.2C_2H_6OS.2C_{12}H_9F_2NO_4S_2$, each dimethyl sulfoxide molecule is bonded to 18-crown-6 *via* a set of C—H···O interactions involving two H atoms of one methyl group. The sulfoxide O atoms accept a nearly linear N—H···O hydrogen bond from the disulfonylamine NH functions [N···O 2.651 (2) Å]. The macrocycle has approximately D_{3d} symmetry, and the disulfonylamine molecules feature a folded pseudo-C_s symmetric conformation.

Comment

In earlier work we have described the crystal structures of two binary complexes between monofunc-

† Part CV: Näveke *et al.* (1998).

tional di(organosulfonyl)amines, $(RSO_2)_2NH$ (R = Me or 2-naphthyl), and the potentially hexafunctional acceptor species 1,4,7,10,13,16-hexaoxacyclooctadecane (18-crown-6). These complexes involved N—H···O hydrogen bonds (Blaschette *et al.*, 1994; Henschel, Näveke *et al.*, 1995). In a number of similar cases, however, the isolation of binary adducts was found to be inherently impeded by the donor–acceptor disparity of the components; co-crystallizations led preferentially to polynary complexes, in which polar solvent molecules such as water or lower aliphatic alcohols were inserted as ambifunctional hydrogen-bonding links (complementarity mediators) between the NH donor and the hexaether ring (Henschel *et al.*, 1996, and references therein). The title compound, (I), was prepared intentionally. It is the first example of a ternary complex in which dimethyl sulfoxide acts as a link between a disulfonylamine and 18-crown-6, and in which the mediator is bonded to the polyether ring exclusively *via* C—H···O interactions. A search of the Cambridge Structural Database (Allen & Kennard, 1993) located no structural information for any other 18-crown-6 complex containing an uncharged sulfoxide molecule as the guest species. The X-ray structure of the related binary complex 2[(4-F-$C_6H_4SO_2$)$_2$NH].OSMe$_2$, (II), has been determined in our laboratory (Blaschette *et al.*, 1993).

(I)
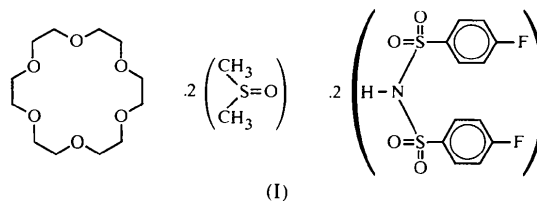
The centrosymmetric formula unit of the title complex is shown in Fig. 1. A traditional N—H···O hydrogen bond links the NH acids to the dimethyl sulfoxide molecules. The latter are situated one above and one below the plane of the ether O atoms; one methyl group of each sulfoxide species is bonded to the crown ether through a set of C—H···O interactions (Fig. 2). Selected intramolecular geometric data and hydrogen bond geometries are listed in Tables 1 and 2, respectively.

In the present structure, the disulfonylamine molecules adopt a folded conformation approximating to C_s symmetry (*vide* torsion angles in Table 1). The essentially planar phenyl rings are stacked, forming an interplanar angle of 12.86 (11)°. A similar conformation was observed for the analogous ternary complex 18-crown-6.2H$_2$O.2[(4-F-$C_6H_4SO_2$)$_2$NH] (Henschel, Blaschette & Jones, 1995), in contrast to complex (II) and to the pure disulfonylamine component (*loc. cit.*), where the molecule features an extended geometry with pseudo-C_2 symmetry. The internal bond lengths and angles of the amine component of (I) are similar to those in the refer-