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A new tetra-lactam host compound and its complexes with ethanol and dimethyl sulfoxide

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The 32-membered tetra-lactam host compound 1 is synthesized in 26% yield by the ring formation reaction of glutaryl dichloride with the diamido-diamine 2 using high dilution conditions. X-Ray crystal structure analyses of complexes of 1 with ethanol (EtOH) (1a) and dimethyl sulfoxide (DMSO) (1b) with unusual host:guest stoichiometric ratios (1:4 for 1a and 1:6 for 1b) are reported showing specific modes of host-guest hydrogen bonding.

INTRODUCTION

Recently, tetra-lactam macrorings have arisen considerable interest owing to their potential property in forming a catena structure during the cyclization.[1] Macrocyclic lactams are also known for being hosts to metal cations[2,3] and organic molecules.[4] This has stimulated the design of new macrorings that contain several lactam units, in particular tetra-lactams.[5]

In this view, we have started a systematic study involving the synthesis and complex formation of designed macrocyclic tetra-lactam hosts. Here we describe the preparation of such a host compound (**1**) and report crystal structures of two complexes of **1** with ethanol (EtOH) (**1a**) and dimethyl sulfoxide (DMSO) (**1b**). These complexes, in particular, are of interest due to their unusual host:guest stoichiometric ratios (1:4 for **1a** and 1:6 for **1b**) suggesting specific supramolecular structures in the solid state.

RESULTS AND DISCUSSION

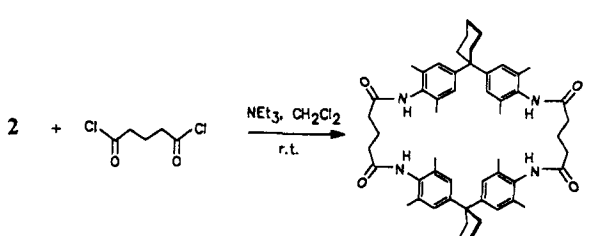
Syntheses

Host compound **1** was synthesized in 26% yield by ring formation reaction of diamido-diamine **2** with glutaryl dichloride and triethylamine in CH_2Cl_2 using the high dilution technique[6,7] (Scheme 1). Noteworthy enough, no formation of the respective catenane was observed, unlike a similar cyclization reaction where isophthaloyl dichloride was used instead of glutaryl dichloride.[1] Diamido-diamine **2** was made in 73% yield from excess **4** with glutaryl dichloride and triethylamine in CH_2Cl_2 (Scheme 2). This reaction gave also a 26% yield of tetraamido-diamine **3** which was separated from **2** by column chromatography.

The crystalline complexes **1a** and **1b** were obtained by recrystallization of **1** from CH_2Cl_2-EtOH solvent mixture and DMSO, respectively.

X-ray diffraction analyses

Crystal data of compounds **1a** and **1b** are given in Table 1. The final values of the positional parameters of **1a** and **1b** are listed in Tables 2 and 3. Selected conformational features of the host molecules are shown in Table 4. Table 5 lists the distances and angles in O-H···O and N-H···O hydrogen bonds. Perspective views of the stoichiometric units of compounds **1a** and **1b** are depicted in Fig. 1 (a, b). The molecular packings and the H-bond frameworks in crystals **1a** and **1b** are illustrated by the

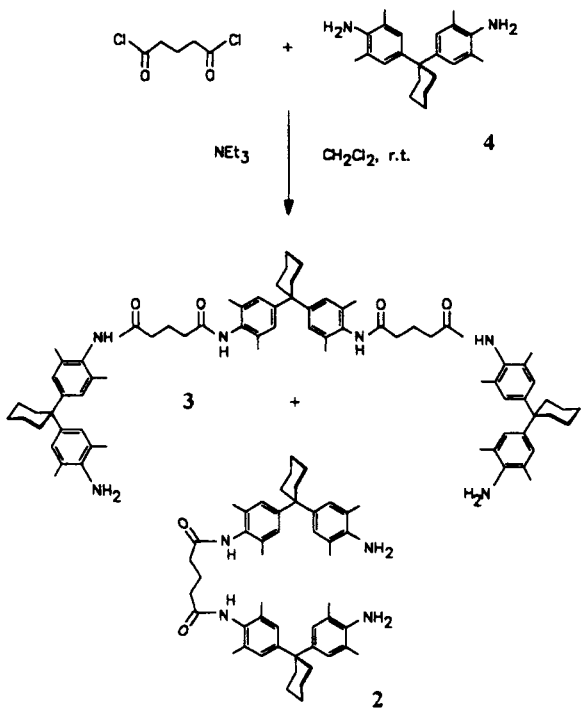


1a = **1** · EtOH (1:4)
1b = **1** · DMSO (1:6)

1

Scheme 1

*To whom correspondence should be addressed.



Scheme 2

stereo plots of Fig. 2 (a, b). A simplified scheme of the H-bond network provided by **1a** is shown in Fig. 3.

Molecular structures

No anomalous values have been observed for the covalent bond distances and bond angles in the host (**1**) and guest molecules (EtOH, DMSO). In both structures, **1a** and **1b** (Figs. 1a and 1b), the macroring has C_i-symmetry with crystallographic inversion centres at (0, 1/2, 1/2) (**1a**) and (1/2, 1/2, 1/2) (**1b** (Fig. 2). Host macrocycles adopt approximately rectangular overall shape with dihedral angles between phenylene moieties *I* and *II* (respectively *Ia* and *IIa*) near 90° (Table 4). This feature corresponds to other macrorings comprising diphenylmethane building blocks[8] unlike a similar benzophenone-containing macrocycle[9] with the aromatic components partly filling the lumen of the ring. All conformations involving O-C-N-H are in the usual *anti*.[10] Two of the four N-H bonds of each macroring in **1a** and **1b** are oriented *endo*, the others are *exo*. The same holds true for the carbonyl groups, thus making possible specific intra- and extracavity H-bonding to the guests.

Table 1 Crystal data and selected details of the data reduction and structure refinement calculations

Compound	*1a*	*1b*
Formula	$C_{54}H_{68}N_4O_4$•4 EtOH	$C_{54}H_{68}N_4O_4$•6 DMSO
Formula weight	1021.4	1305.9
Space group	$P\bar{1}$ (No.2)	$P2_1/n$ (No. 14)
Temp. °C	20	-80
Cell[a] dimensions		
a, Å	9.492(1)	14.081(2)
b, Å	13.286(1)	13.159(3)
c, Å	13.366(1)	19.185(3)
α, deg	99.06(1)	
β, deg	109.00(1)	93.70(1)
γ, deg	98.84(1)	
V_c, Å^3	1535.5(2)	3547(1)
Z	1	2
Radiation/ λ, Å	Cu*K*α/1.54178	Cu*K*α/1.54178
D_c, g cm^{-3}	1.11	1.22
μ, mm^{-1}	0.57	2.23
R	0.065	0.071
R_w	0.078	0.080

a) Least-squares refinement of 25 reflections Θ>20° (**1a**), 40° (**1b**).

Table 2 Fractional atomic coordinates (× 10^4) and equivalent isotropic displacement coefficients (Å^2 × 10^3) for non-hydrogen atoms of **1a**

Atom	*x*	*y*	*z*	*U(eq)*
O(1)	1402(3)	4212(2)	9405(2)	86(1)
O(2)	-1525(2)	1851(2)	1649(2)	72(1)
N(1)	1733(3)	4726(2)	7961(2)	62(1)
N(2)	766(2)	1892(2)	1484(2)	51(1)
C(1)	1910(4)	6729(2)	9837(3)	67(1)
C(2)	708(3)	5809(2)	9038(2)	55(1)
C(3)	1319(3)	4843(2)	8832(2)	56(1)
C(4)	2194(3)	3828(2)	7541(2)	49(1)
C(5)	1087(3)	2918(2)	6947(2)	53(1)
C(6)	1543(3)	2102(2)	6447(2)	50(1)
C(7)	3044(3)	2147(2)	6511(2)	44(1)
C(8)	4113(3)	3059(2)	7135(2)	48(1)
C(9)	3717(3)	3895(2)	7659(2)	51(1)
C(10)	3424(3)	1263(2)	5823(2)	44(1)
C(11)	5151(3)	1339(2)	6115(2)	50(1)
C(12)	5965(3)	1142(2)	7234(2)	64(1)
C(13)	5216(4)	109(3)	7369(3)	75(2)
C(14)	3526(4)	35(2)	7119(2)	64(1)
C(15)	2748(3)	190(2)	5980(2)	52(1)
C(16)	2738(3)	1376(2)	4639(2)	45(1)
C(17)	1714(3)	581(2)	3795(2)	46(1)
C(18)	1081(3)	722(2)	2741(2)	47(1)
C(19)	1477(3)	1693(2)	2541(2)	48(1)
C(20)	2530(3)	2507(2)	3356(2)	55(1)
C(21)	3146(3)	2327(2)	4383(2)	56(1)
C(22)	-695(3)	1990(2)	1128(2)	54(1)
C(23)	-1260(3)	2291(2)	53(2)	60(1)
C(24)	-556(4)	2825(3)	6842(3)	77(2)
C(25)	4918(4)	4864(2)	8323(3)	73(1)
C(26)	-13(4)	-171(2)	1858(2)	64(1)
C(27)	2959(5)	3571(3)	3138(3)	92(2)
O(1E)	2519(3)	2613(2)	10189(2)	88(1)
C(1E)	3637(4)	2292(3)	9853(3)	75(2)
C(2E)	4156(5)	1447(3)	10381(4)	108(2)
O(2E)	-1980(7)	3465(3)	2956(4)	196(4)
C(3E)	-2036(15)	3286(6)	3899(6)	260(9)
C(4E)	-2405(14)	4151(11)	4461(8)	305(11)

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Supramolecular structures: Packing relations and host-guest interactions

The stoichiometric unit of **1a** contains one host macroring and four EtOH guest molecules being involved in a complex network of host-guest hydrogen bonds (Table

Table 3 Fractional atomic coordinates (× 10^4) and equivalent displacement coefficients ($Å^2$ × 10^3) for non-hydrogen atoms of **1b**

Atom	*x*	*y*	*z*	*U(eq)*	
O(1)	7509(2)	4431(2)	2485(2)	49(1)	
O(2)	1674(2)	5552(2)	4820(2)	45(1)	
N(1)	6407(2)	4511(2)	3298(2)	32(1)	
N(2)	1125(2)	7087(2)	4454(2)	29(1)	
C(1)	8781(3)	4050(3)	3795(2)	32(1)	
C(2)	7833(3)	3550(3)	3572(2)	32(1)	
C(3)	7235(3)	4197(3)	3063(2)	32(1)	
C(4)	5699(3)	5111(3)	2928(2)	29(1)	
C(5)	4826(3)	4641(3)	2727(2)	27(1)	
C(6)	4095(3)	5244(3)	2430(2)	29(1)	
C(7)	4193(3)	6278(3)	2318(2)	27(1)	
C(8)	5082(3)	6707(3)	2502(2)	33(1)	
C(9)	5834(3)	6143(3)	2805(2)	34(1)	
C(10)	3339(3)	6904(3)	2020(2)	27(1)	
C(11)	2864(3)	6335(3)	1382(2)	35(1)	
C(12)	2048(3)	6906(3)	1005(2)	41(1)	
C(13)	2359(4)	7947(3)	774(2)	51(2)	
C(14)	2818(3)	8536(3)	1381(2)	41(1)	
C(15)	3638(3)	7955(3)	1748(2)	33(1)	
C(16)	2671(2)	7018(3)	2625(2)	24(1)	
C(17)	2835(3)	7772(3)	3133(2)	27(1)	
C(18)	2310(3)	7826(3)	3727(2)	27(1)	
C(19)	1600(3)	7098(3)	3816(2)	26(1)	
C(20)	1382(3)	6380(3)	3297(2)	27(1)	
C(21)	1924(2)	6337(3)	2721(2)	26(1)	
C(22)	1212(3)	6328(3)	4921(2)	33(1)	
C(23)	699(3)	6467(3)	5587(2)	35(1)	
C(24)	4684(3)	3527(3)	2846(2)	37(1)	
C(25)	6761(3)	6645(3)	3029(3)	50(2)	
C(26)	2504(3)	8654(3)	4262(2)	37(1)	
C(27)	559(3)	5661(3)	3355(2)	38(1)	
S(1D)	5975(1)	3379(1)	5169(1)	48(1)	
O(1D)	5599(2)	3790(3)	4481(2)	58(1)	
C(1D)	6208(3)	4396(3)	5743(3)	48(1)	
C(2D)	4944(6)	2900(7)	5544(3)	110(3)	
S(2D)	-1027(1)	8981(1)	4471(1)	51(1)	s.o.f.=0.85(1)
S(2D')	-572(8)	8986(7)	3988(5)	69(4)	s.o.f.=0.15(1)
O(2D)	-107(3)	8694(3)	4810(2)	86(2)	
C(3D)	-869(5)	10051(4)	3940(3)	75(2)	
C(4D)	-1236(6)	8055(5)	3793(4)	93(3)	
S(3D)	9720(2)	5209(3)	1127(2)	90(1)	s.o.f.=0.70(1)
S(3D')	9358(7)	4730(6)	1462(6)	126(4)	s.o.f.=0.30(1)
O(3D)	10297(5)	4510(6)	1589(5)	169(4)	
C(5D)	8596(7)	4557(7)	881(6)	139(5)	
C(6D)	9150(8)	6059(6)	1658(6)	129(4)	

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

5), where all of the hydrogen donor and acceptor sites of the functional groups are used. Two of the EtOH guest molecules are located inside the host macroring. They

Table 4 Selected conformational features of the host molecules in **1a** and **1b**

Comp	*1a*	*1b*
Z = i at	0, 1/2, 1/2	1/2, 1/2, 1/2
Z-B	4.54 Å	4.64 Å
Z-C	5.60 Å	5.81 Å
Z-Cl	6.06 Å	6.07 Å
Z-C10	6.39 Å	6.53 Å
B-D	9.09 Å	9.31 Å
C-E	11.21 Å	11.62 Å
Cl-C1a	12.13 Å	12.14 Å
C10-C10a	12.77 Å	13.07 Å
B-C	4.57 Å	4.60 Å
C-D	9.12 Å	9.47 Å
$\sphericalangle\mu_B/\mu_C$	95°	85°

I = $Z_{C4\cdots C9}$
II = $Z_{C16\cdots C21}$
Ia = $Z_{C4a\cdots C9a}$
IIa = $Z_{C16a\cdots C21a}$
μ_B=C4...C9-plane
μ_C=C16...C21-plane

maintain strong hydrogen bonds to N(1) and O(2) (Fig. 1a). Moreover, each host macroring is hydrogen-bonded at its outside to four molecules of EtOH, two of them being proton donors to carbonyl groups of the ring and two being proton acceptors with reference to NH. Each of the laterally bound molecules of EtOH is then connected via an additional H-bond to a neighbouring macroring, thus forming a supramolecular framework of infinite chains of interpenetrating H-bonded rings and host macrocycles, as shown schematically in Fig. 3. A complete view of the packing is given in Fig. 2a, showing that H-bond interactions determine the structure of **1a**.

By way of contrast, there is no such scheme of interwoven rings in the case of **1b**, owing to the lack of pronounced H-donorship of the DMSO guest (Fig. 1b). Instead, each of the four NH-groups of the macroring is hydrogen-bonded to the oxygen of an individual DMSO guest (Table 5). As a result of the orientation of the NH (see above), two of the DMSO guests are located inside and two are located outside the macroring. There are two more DMSO guests being part of the stoichiometric unit. They are not involved in any H-bond interaction but fill up free space between the H-bonded units (Fig. 2b). The voids located around the nonhydrogen-bonded molecules of DMSO are large enough to allow disorder for these loosely bound guest molecules.

Table 5 Bond distances (Å) and angles (°) of hydrogen bonds in **1a** and **1b**

Donor···Acceptor	Donor-H	H···Acceptor	Donor-H···Acceptor
Cmp **1a**			
N(1)···O(2e) 2.88	N(1)-H(1) 0.90	H(1)···O(2e) 1.99	N(1)-H(1)···O(2e) 171
N(2)···O(1e) 2.93	N(2)-H(2) 0.90	H(2)···O(1e) 2.07	N(2)-H(2)···O(1e) 160
O(1)···O(1e) 2.72	O(1e)-H(1e) 0.85	H(1e)···O(1) 1.88	O(1e)-H(1e)···O(1) 170
O(2)···O(2e) 2.73	O(2e)-H(2e) 0.85	H(2e)···O(2) 1.88	O(2e)-H(2e)···O(2) 171
Cmp **1b**			
N(1)···O(1d) 2.77	N(1)-H(1) 0.90	H(1···O(1d) 1.91	N(1)-H(1)···O(1d) 159
N(2)···O(2d) 2.85	N(2)-H(2) 0.90	H(2)···O(2d) 1.95	N(2)-H(2)···O(2d) 175

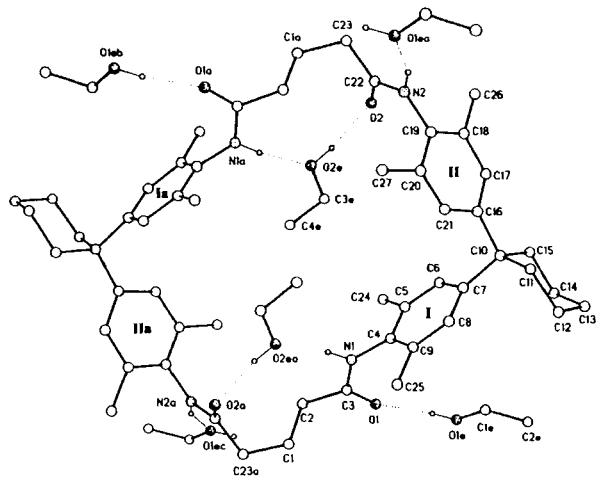


Figure 1a

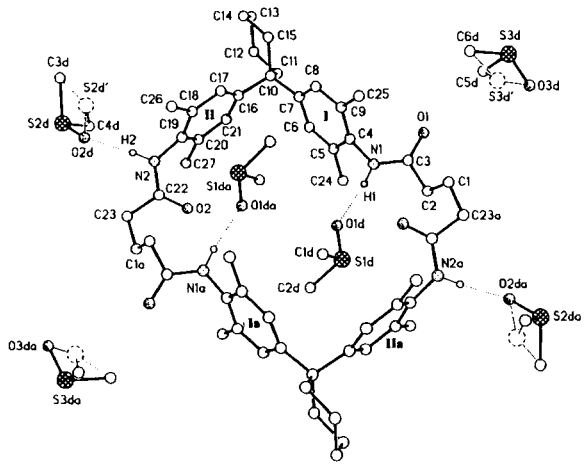


Figure 1b

Figure 1 Perspective views (including atom numbering) of the stoichiometric units showing the conformation and the relative positioning of the supramolecular constituents: (a) for **1a**, (b) for **1b**. Solid and dotted lines represent covalent and hydrogen bonds, respectively. Heteroatoms are shaded. H atoms are omitted for clarity, except those involved in H bonds.

CONCLUSIONS

The present investigation clearly demonstrates the usefulness of the tetra-lactam macrocycle **1** as a host for organic guest molecules, in two ways: (1) The host macroring offers a preorganized cavity as deducible from the two inclusion structures. A least-squares fit shows that the host conformations in **1a** and **1b** are nearly identical. (2) The macroring **1**, under prime guest conditions (e.g. EtOH), renders possible no less than eight H-bonding contacts (four H-donor and four H-acceptor sites), thus making feasible high host-guest stoichiometric ratios and extensive H-bonded networks. These are promising features for future host developments[11] including precursor behavior for catena structures.[12]

EXPERIMENTAL

Synthesis

General: Melting points (uncorrected) were determined with a Reichert hot-stage apparatus. IR spectra were recorded on a Perkin-Elmer 1600 FT-IR spectrometer; spectral bonds are reported in cm^{-1}. Proton and ^{13}C-NMR spectra were measured with a Bruker WM-250 spectrometer; chemical shifts in δ down-field from internal Me_4Si. Mass spectra were obtained using a Kratos Concept ^1H instrument. Microanalyses were carried out by the Microanalytical Laboratory of the Institut für Organische Chemie und Biochemie, Universität Bonn. For column chromatography SiO_2 (40–60 μm, Merck) was used. Solvents were purified and dried in the usual manner before use.

Diamine **4** was prepared as described in the literature;[1a] glutaryl dichloride was purchased from Janssen.

Diamines 2 and 3

To a stirred solution of compound **4** (10 g, 31 mmol) and triethylamine (1.4 mL) in dry CH_2Cl_2 (50 mL) was added dropwise during 4 h at room temperature and under argon glutaryl dichloride (0.83 g, 4.9 mmol) dissolved in dry CH_2Cl_2 (100 mL). The mixture was stirred over night and the solvent evaporated under reduced pressure. Purification by column chromatography [SiO_2, eluent CH_2Cl_2-EtOH (15:1)] yielded **2** (R_f = 0.31) and **3** (R_f = 0.14) in two fractions. Specific details for **2** and **3** are given below.

2: colorless solid, 84%, m.p. 113°C. IR (KBr): 3320, 3254, 2927, 2855, 2363, 2333, 1651, 1558, 1491, 1455, 1240, 1084, 860, 752. ^1H-NMR (250 MHz, $CDCl_3$): 1.4–1.6, br, 12 H (Cyclohex-CH_2); 2.05–2.30, br, 8H (Cyclohex-CH_2); 2.10, s, 2H (Glutaryl-CH_2); 2.12, s, 12H (Me); 2.14, s, 12H (Me). 2.17, s, 4H (Glutaryl-CH_2); 3.44, br, 4H (NH_2). 6.82, s, 4H (Ar); 6.94, s, 4H (Ar). ^{13}C-NMR (62.9 MHz, $CDCl_3$): 18.31, 18.80, 22.52, 22.89, 26.38, 35.20, 37.11, 44.80, 121.45, 126.74, 126.97, 130.90, 134.49, 137.58, 139.96, 148.30. FAB-MS (*m/z*): 740.4 (M^+, calcd. for $C_{49}H_{64}N_4O_2$: 740).

3: colorless solid, 26%, m.p. 167–168°C. ^1H-NMR (250 MHz, $CDCl_3$): 1.40–2.60, br, 36H (Cyclohex-CH_2); 2.08, s, 12H (Me); 2.15, s, 12H (Me); 2.19, s, 12H (Me); 3.42, br, 4H (NH_2); 6.70–7.10, m, 12H (Ar). FAB-MS (*m/z*): 1159.6 [$(M+H)^+$; calcd for $C_{76}H_{98}N_6O_4$: 1158].

Host compound 1

Solutions of **2** (956 mg, 1.29 mmol) with triethylamine (0.4 ml) and of glutaryl dichloride (218 mg, 1.29 mmol) in separate 250 ml portions of dry CH_2Cl_2 were simultaneously added over a period of 8 h and under argon to vigorously stirred dry CH_2Cl_2 (1.2 L) at room temperature. Stirring was continued for 2 h at the same tempera-

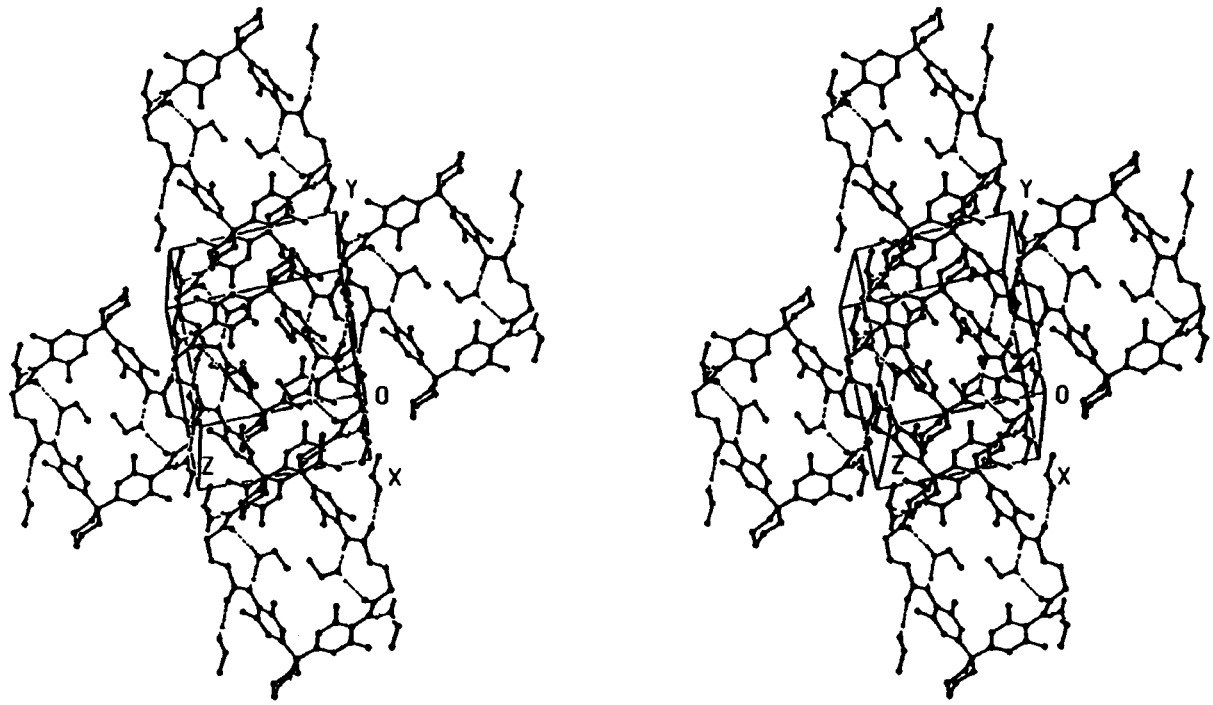


Figure 2a

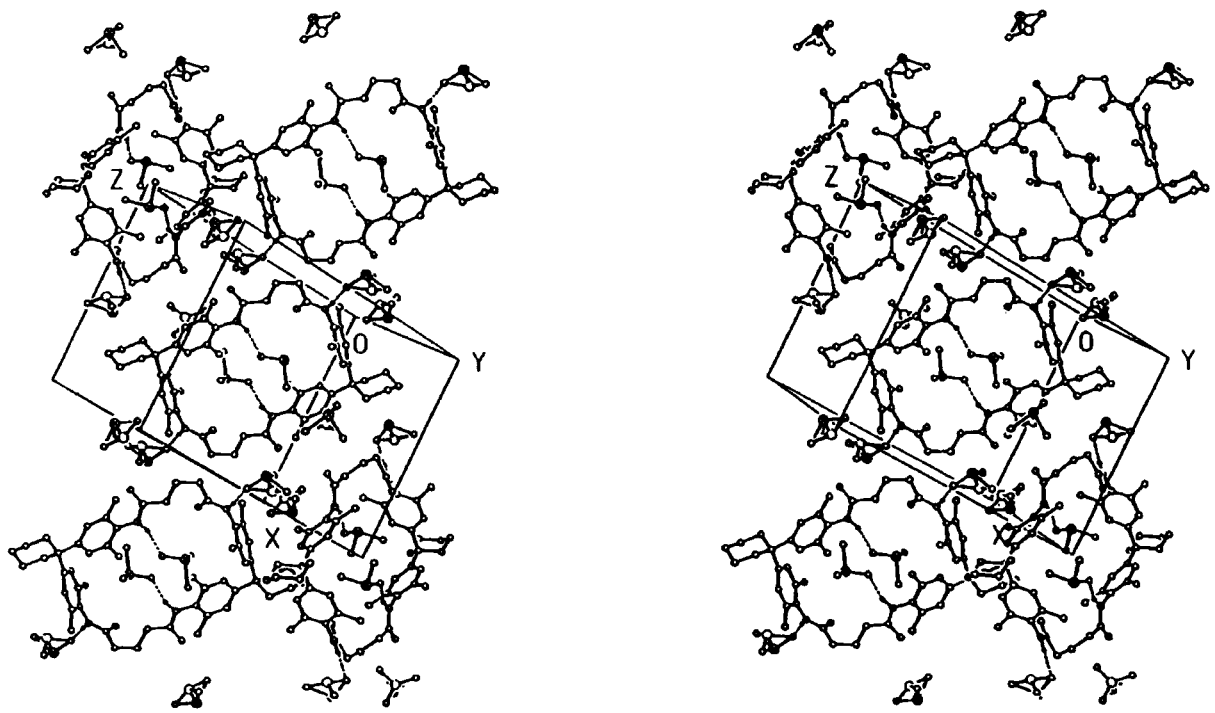


Figure 2b

Figure 2 Stereoscopic packing illustrations of (a) **1a** and (b) **1b**. H atoms are omitted for clarity, except those involved in H bonds. H bonds are indicated as dotted lines.

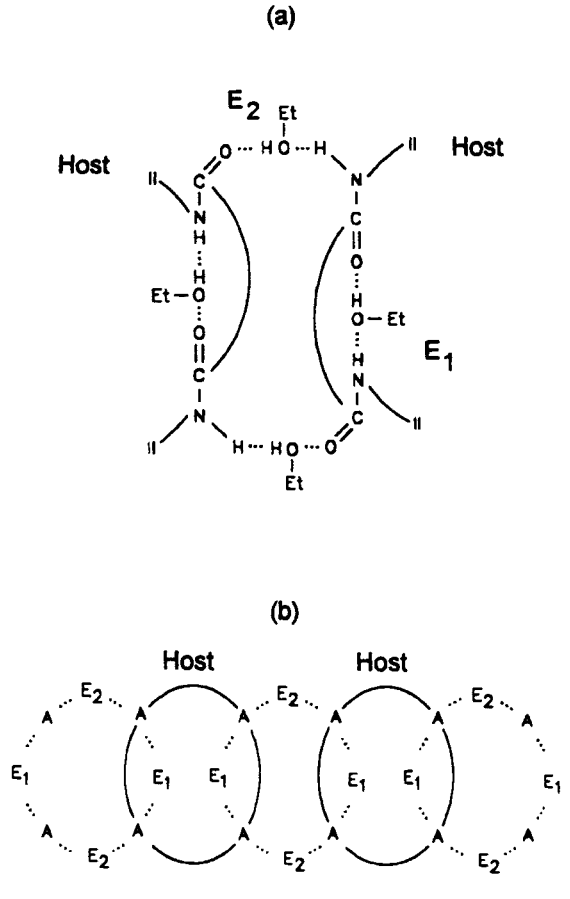


Figure 3 Simplified diagram of the system of H bonds involved in **1a**: (a) individual unit of the H-bonded ring constitution formed of two host and four EtOH molecules; (b) supramolecular framework showing an infinite chain of interpenetrating H-bonded rings [cf. (a)] and host macrocycles. Dotted lines represent H bonds, solid lines are segments of the host; A stands for the carbonamide unit; E_1 are the intra-cavity, E_2 are the extracavity molecules of EtOH, relative to the host macroring.

ture. The solvent was removed under reduced pressure and the residue purified by column chromatography [SiO_2, eluent CH_2Cl_2-EtOH (15:1), R_f = 0.41] to yield colorless solid, 26%, m.p. > 300 °C. IR (KBr): 3256, 2934, 2854, 2656, 1596, 1525, 1443, 1266, 1170, 860, 726. ^{1}H-NMR (250 MHz, DMSO-d_6): 1.30–1.60, br, 12H (Cyclohex-CH_2); 1.70–2.40, m, 20H (Cyclohex-CH_2, Glutaryl-CH_2); 1.81, s, 6H (Me); 2.04, s, 12H (Me); 2.13, s, 6H (Me); 6.92, s, 4H (Ar); 7.10, s, 2H (Ar); 7.12, s, 2H (Ar); 8.52, s, 1H (NH); 8.85, s, 1H (NH); 9.04, s, 2H (NH). ^{13}C-NMR (62.9 MHz, DMSO-d_6): 18.40, 18.54, 18.64, 18.67, 22.49, 22.61, 35.42, 35.73, 41.37, 43.87, 44.55, 125.54, 126.21, 132.53, 132.60, 134.45, 134.72, 135.65, 145.84, 146.07, 170.30, 170.35. FAB-MS (*m/z*): 837.5 [$[M+H)^+$, calcd. 836]. Anal. Calcd. for $C_{54}H_{68}N_4O_4$: C, 75.84; H, 8.25; N, 6.55%. Found: C, 76.10; H, 8.24; N, 6.65%.

X-ray data collection, structure determination, and refinement

Single crystals of **1a** and **1b** were prepared by slow evaporation of solvent from CH_2Cl_2-EtOH and DMSO solutions of **1**, respectively.

A summary of data collection is given in Table 1. All data were collected on an Enraf-Nonius CAD4 diffractometer ($2\Theta_{max}$ = 120°). An empirical absorption correction was applied for **1a**. In two DMSO molecules of **1b** the S atoms are disordered [S(2d) and S(3d) in Fig. 1b].

Lists of final atomic coordinates for the hydrogen atoms, bond distances and angles, and anisotropic thermal parameters of the non-hydrogen atoms are available from the authors (MN).

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