fashion (Cu17, Cu22, Cu23, Cu25, Cu26, Cu28, Cu36, Cu38, Cu41, Cu42, Cu45, Cu46) or in a distorted tetrahedral fashion (Cu6, Cu8, Cu11, Cu13, Cu20, Cu29, Cu31, Cu34, Cu39, Cu43).

Overall the structure of **1** can be considered as an intermediate in the formation of the Cu_3P structure. The course of events which take place during the growth of such a cluster cannot be described at present. NMR investigations show that CuCl–phosphane complexes are partly dissociated in solution and that an equilibrium exists between coordinated and free phosphane.[11] In contrast, the coordination of $P(SiMe_3)_3$ to Cu atoms is irreversible due to the elimination of $SiMe_3Cl$. The growth of the cluster is limited by the solubility of the units formed, since when cluster units become too insoluble they precipitate. The favorable position of the participating complex equilibria seems particularly important. If the stoichiometry of the reactants is changed and the PEt_3 contribution is increased, the formation of **1** can no longer be observed. From this point of view **1** is only an intermediate.

Attempts to use the reactive centers of **1** (the six μ_2-$P(SiMe_3)_2$ ligands) for the construction of larger units have been unsuccessful so far. The main reason is the poor solubility of **1**. Compound **1** decomposed on treatment with nonmetal halides such as PCl_3, $RPCl_2$, and R_2PCl; the cluster also did not react with additional CuCl.

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[3] Preparation of **1**: $P(SiMe_3)_3$ (6.7 g) was added to a suspension of CuCl (5.3 g, 53.4 mmol) and PEt_3 (3.8 g, 32.1 mmol) in THF (50 mL). The reaction mixture turned black within a few minutes. After three hours reaction time the batch was left to stand. Black crystals of **1** formed within two weeks. (Yield 15%.)
[4] X-ray structure analysis: STOE IPDS, $Mo_{K\alpha}$, data collection and refinement: lattice constants: $a = 23.863(7)$, $b = 22.127(8)$, $c = 31.518(10)$ Å, $\beta = 98.75°$, $V = 16488 \times 10^6$ pm^3, space group: $P2_1/n$ (no. 14), $Z = 2$, $\mu(Mo_{K\alpha}) = 62.5$ cm^{-1}, $2\theta_{max} = 42°$; 51478 reflections, of which 29511 are independent. 12287 with $I > 4\sigma(I)$, 1109 parameters (Cu, P, Si1–Si4 anisotropically refined), $R_1 = 0.098$, two molecules THF per asymmetric unit could be localized. Further details of the crystal structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (FRG) on quoting the depository number CSD-58058.
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An Organic Molecule with a Rigid Cavity of Nanosize Dimensions

Peter Timmerman, Willem Verboom, Frank C. J. M. van Veggel, Willem P. van Hoorn, and David N. Reinhoudt*

The rapidly expanding field of supramolecular chemistry[1–3] has revealed a number of useful building blocks for the synthesis of artificial receptor molecules, for example calixarenes,[4] cyclodextrins,[5] and resorcinol-based cavitands.[6] Nature constructs an almost infinite number of macromolecular receptors by the systematic combination of a limited number of building blocks. We are currently exploring an analogous approach for the synthesis of host molecules by the combination of simple rigid building blocks.[7] Recently, this method has resulted in the synthesis of several new receptor molecules with unique complexation properties.[8–11] These molecules possess small cavities suitable for the complexation of alkali metal cations, anions, or small organic molecules. The synthesis of receptor molecules with large cavities generally meets with the problem that such cavities collapse because of the inherent flexibility of large organic molecules. Recently, a few examples of receptor molecules with large, relatively open, rigid cavities were reported by Sanders et al.[12] in which rigid planar components (porphyrins and cholic acid derivatives) were linked by single covalent bonds.

In this paper we describe a new approach to receptor molecules with large cavities by the combination of building blocks which already possess a cavity, namely calix[4]arenes and resorcinol-based cavitands. The assembly of four such units in a cyclic array provides a convergent route for the synthesis of holand **1**,[13] an extremely rigid host molecule in which the cavities of the four components form a shielded hole of nanosize dimensions.

As part of our work on the synthesis of selectively functionalized resorcinol-based cavitands,[14] we studied the reaction between cavitand **2**[15] and upper rim 1,2-functionalized calix[4]arene **3**. Compound **3** was prepared in 72% overall yield by reduction of the corresponding 1,2-dinitro compound[16] and subsequent reaction with two equivalents of α-chloroacetyl chloride. When this reaction was performed in $CH_3CN/Cs_2CO_3/KI$ (ratio **2**/**3** 1:1), we isolated compound **4a** (20% yield), in which the calix[4]arene moiety is coupled in a 1,2 fashion, that is to two neighboring arene rings of **2** with the *endo* stereochemistry, together with 32% of the *exo* isomer **4b**.[17] In addition to these 1:1 adducts small amounts of the three possible isomeric 2:1 products **5a–c** were formed. Products in which the calix moiety is coupled in a 1,3 (distal) fashion to the cavitand could not be detected. When **2** was treated with two equivalents of **3** only the 2:1 addition products were isolated in an almost statistical ratio of *endo-endo* (**5a**, shown), *endo-exo* (**5b**), and *exo-exo* (**5c**) in a total yield of 64%.[18]

Apparently in this reaction there is a slight preference for the formation of the *exo* 1:1 product. The formation of the *endo* 1:1 product is favored by the introduction of functional groups at the calix[4]arene fragment that favorably interact with the cavitand moiety in the transition state. Reaction of a 1:1 mixture of **2** and calix[4]arene **6**,[19] in which two nitro groups have been introduced, exclusively gave the *endo* 1:1 isomer **7** together with small amounts of the 2:1 products **8a** (*endo-endo*) and **8b** (*endo-*

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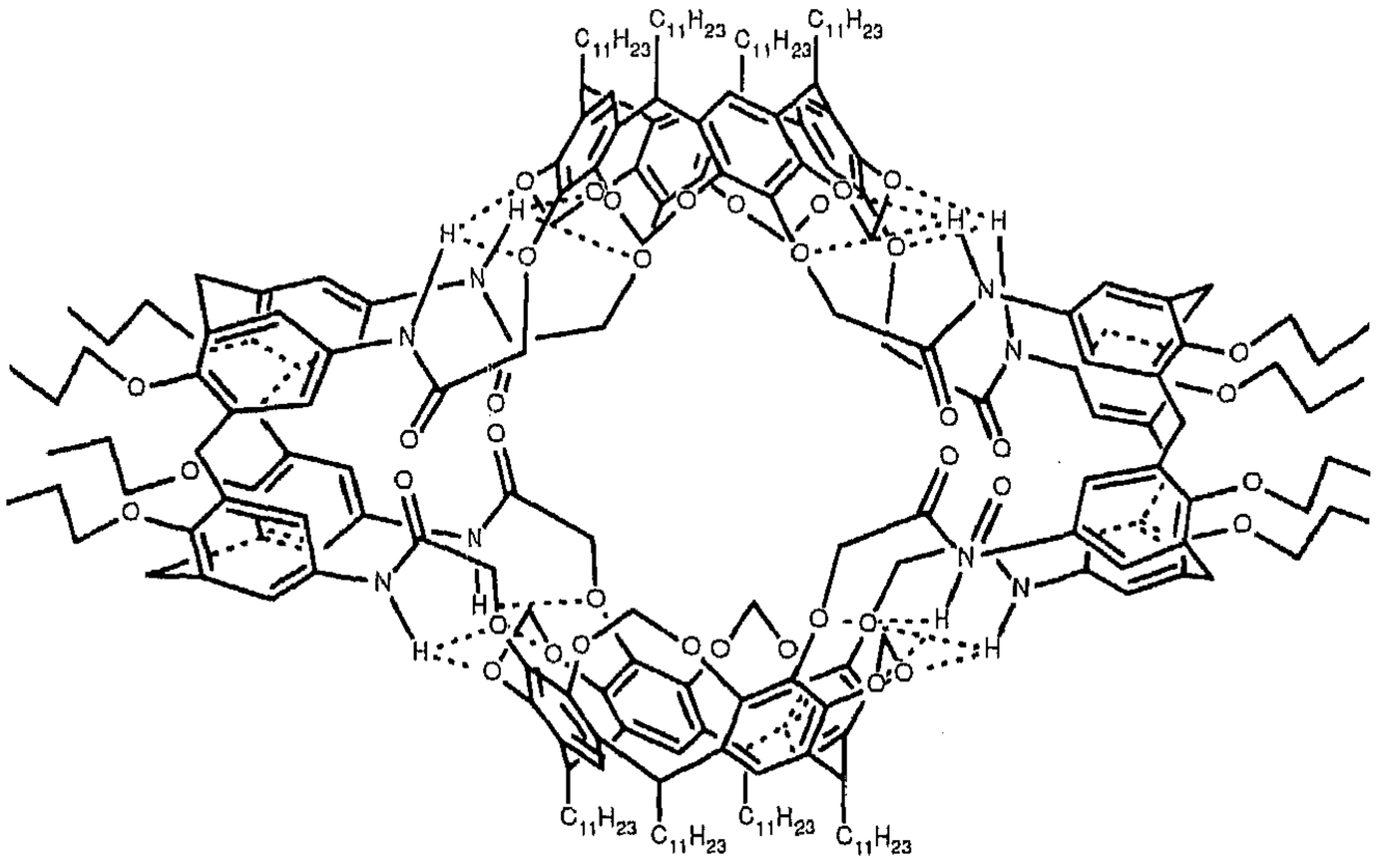
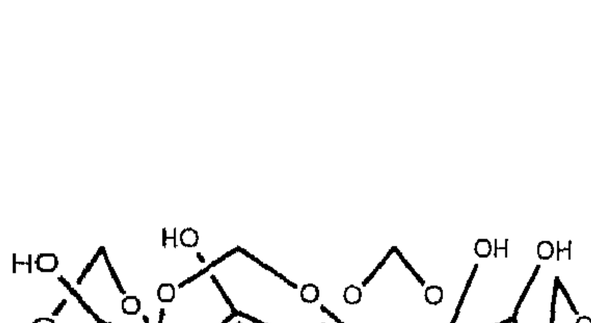


1

exo). Because of its instability **7** was isolated, after silylation of its free hydroxyl groups,[20] as **9** in 41% yield. The nitro groups in **9** could easily be reduced to amino groups by using Raney Ni/hydrazine.[16] The subsequent reaction with α-chloroacetyl chloride led to the bis(2-chloroacetamide) derivative **10** in quantitative yield. Compound **10** was dissolved in dimethylformamide (DMF; 5 mM) and desilylated with CsF at 80 °C; the solution was subsequently stirred for 48 hours in the presence of Cs_2CO_3 and KI to give two reaction products. The more polar product (isolated in 27% yield) is the calix[4]arene-based carcerand **11** with one molecule of DMF inside its interior. The presence of this permanent guest molecule is evident from both FAB mass spectrometry ($m/z = 2126$ (100%) [M + DMF + Na^+]) and ^{1}H NMR spectroscopy (two different singlets for the two methyl groups of DMF at $\delta = 0.66$ and -0.86.[21])

The second product (isolated in 26% yield) is the desired holand **1**. This compound could also be synthesized in 35% yield by dropwise addition of an equimolar solution of **12**[22] and **2** in DMF to a suspension of Cs_2CO_3 and KI in DMF. The FAB mass spectrum proves the formation of the 2:2 structure ($m/z = 4084$ (100%) [$M + Na^+$]) and the ^{1}H NMR spectrum (Fig. 1) reflects the high degree of symmetry expected for **1**. For its size, the molecule is extremely rigid: The calix[4]arene and cavitand moieties,



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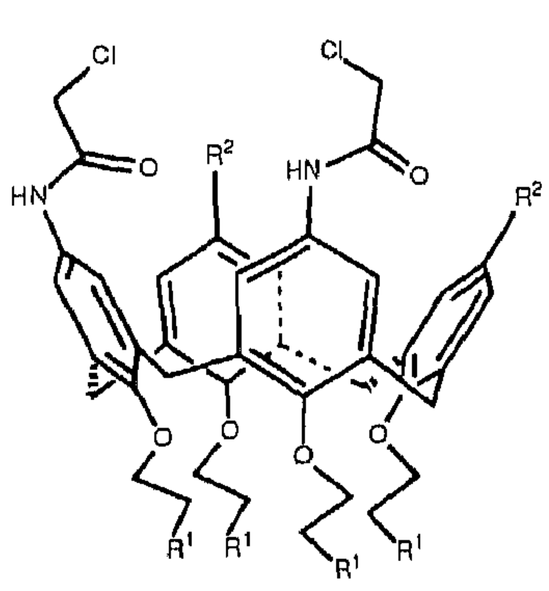
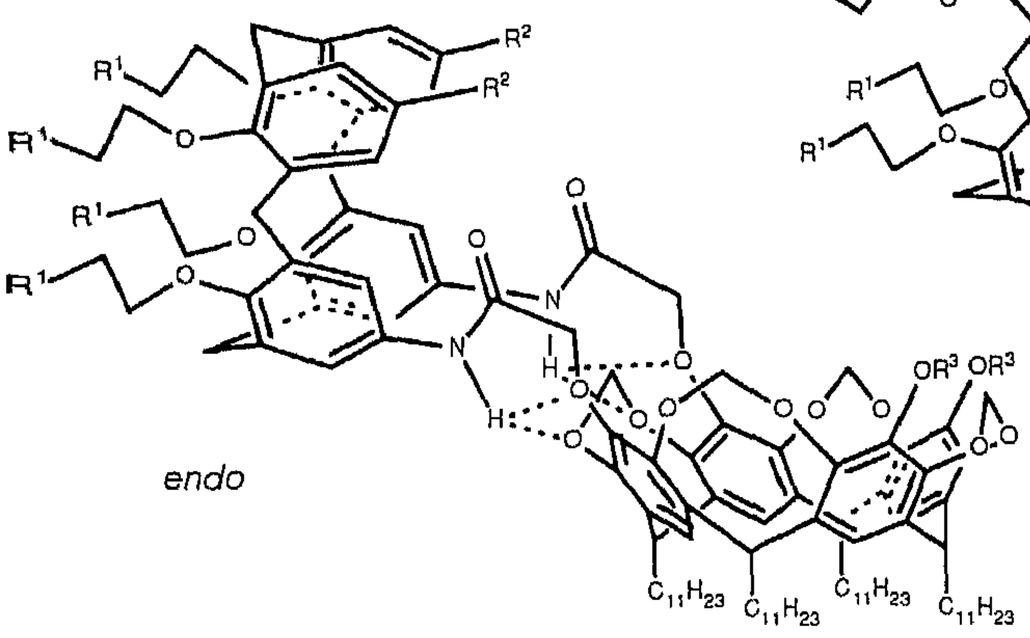
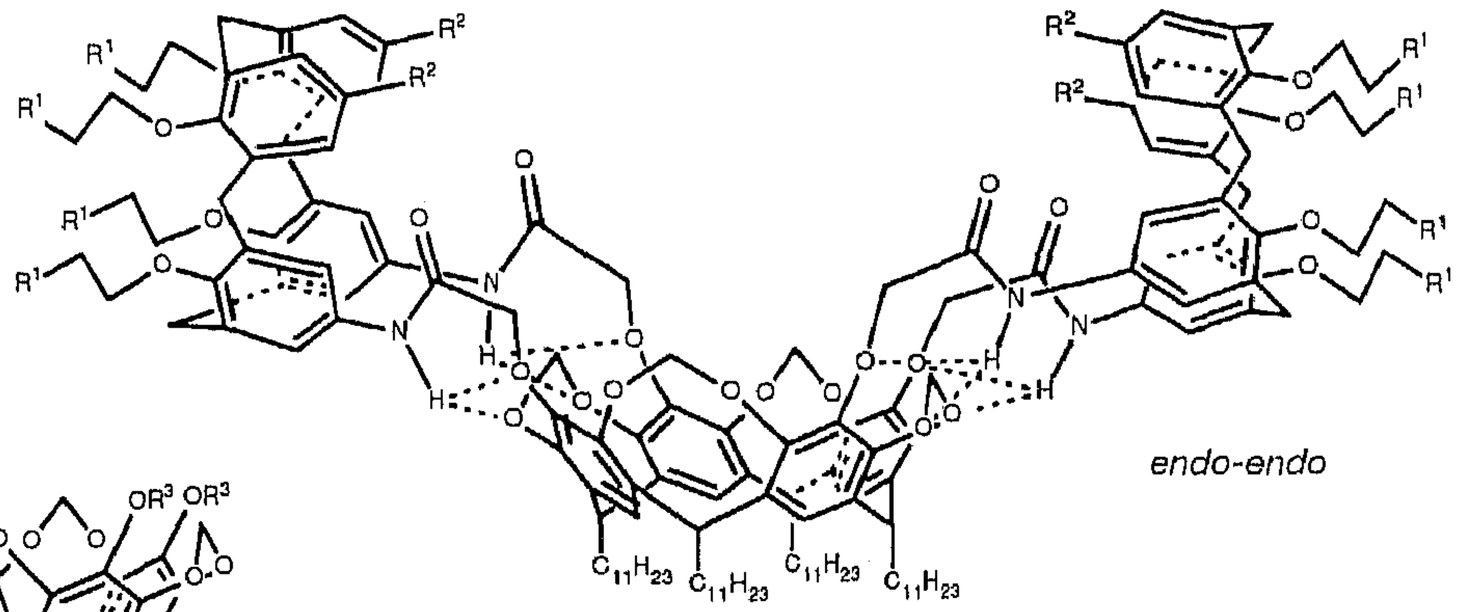


3: R^1=OCH_2CH_3, R^2=H
6: R^1=CH_3, R^2=NO_2



endo



endo-endo

4: R^1 = OCH_2CH_3, R^2 = R^3 = H; **a**: *endo*, **b**: *exo*
7: R^1 = CH_3, R^2 = NO_2, R^3 = H; *endo*
9: R^1 = CH_3, R^2 = NO_2, R^3 = $Si(CH_3)_2C(CH_3)_3$; *endo*
10: R^1 = CH_3, R^2 = $NHC(O)CH_2Cl$, R^3 = $Si(CH_3)_2C(CH_3)_3$; *endo*

5: R^1 = OCH_2CH_3, R^2 = H
a: *endo-endo*, **b**: *endo-exo*, **c**: *exo-exo*
8: R^1 = CH_3, R^2 = NO_2
a: *endo-endo*, **b**: *endo-exo*
12: R^1 = CH_3, R^2 = $NHC(O)CH_2Cl$, *endo-endo*

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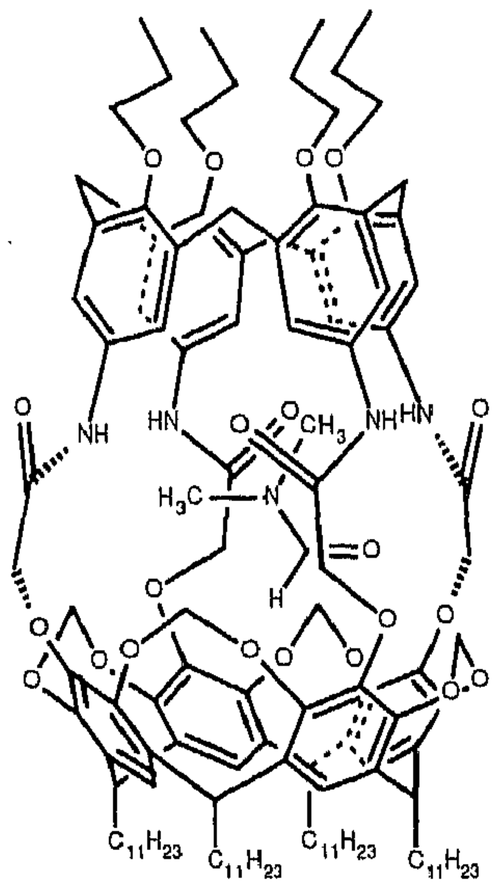

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which are intrinsically rigid, are connected by two highly organized spacers. This becomes evident when the ^{1}H NMR spectra of compounds **3** and **5a** are compared with that of **1**. The signals of the aromatic protons *ortho* to the amide in **3** differ only by 0.08 ppm in chemical shift as a result of almost free rotation around the C(arene)–N bond. However, in **5a** the $\Delta\delta$ value is much greater because two of these calixarene moieties are coupled with the cavitand. The 0.85 ppm chemical shift difference is illustrative for the rigidity in the amide spacers of **5a**. In holand **1** the mobility of the calixarene fragments is further reduced to give a $\Delta\delta$ value of 1.0. The rigidity of the structure of **1** is even better exemplified when the chemical shift difference between the two methylene protons in the spacer in **3** and **5a** is compared with that in **1**. Whereas these protons in **3** give a singlet, this splits in **5a** to show an AB system with $\Delta\delta = 0.4$.

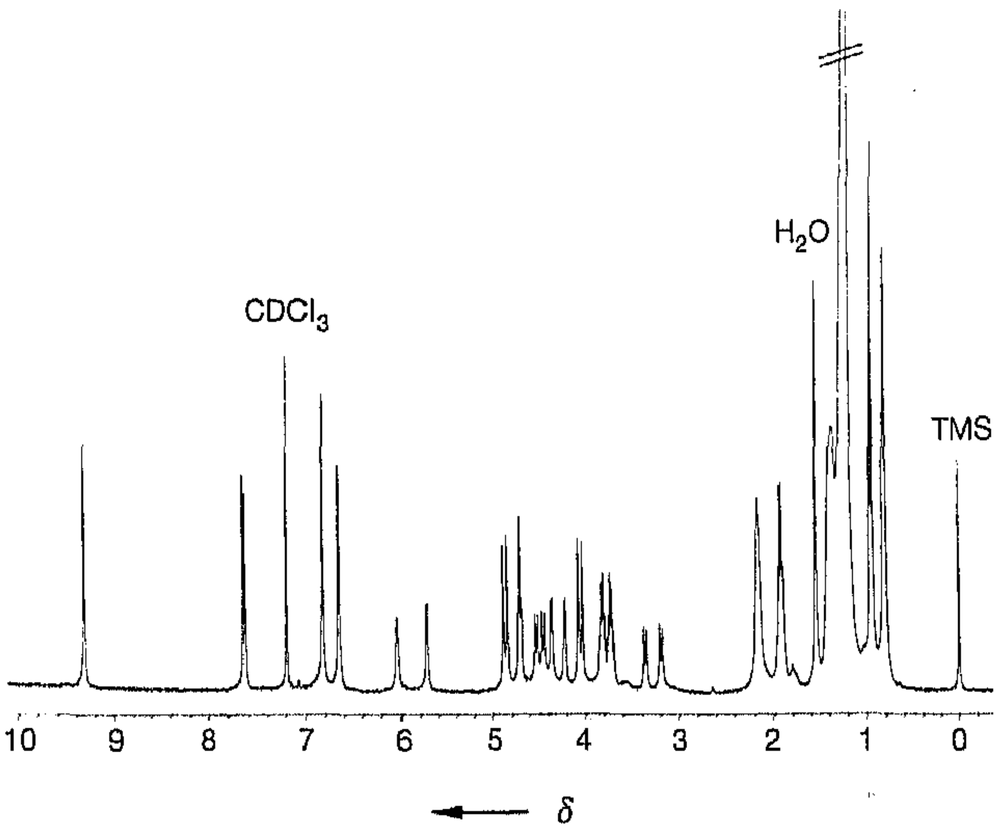

Fig. 1. ^{1}H NMR spectrum (400 MHz) of **1** in $CDCl_3$ at room temperature.

The motion of the calixarene fragments still present in **5a** mainly takes place by rotation around the O–CH_2 bond in the spacer. When this motion is finally frozen completely in **1** the $\Delta\delta$ value between the two AB doublets is 0.85. The above mentioned rigidity of **1** is partly a result of the three-center hydrogen bonds between the amide hydrogen atoms, the oxygen atoms in the spacer itself, and the oxygen atoms in the methylenedioxy bridges. These bonds are indicated in **1** with dashed lines.[23]

When compound **10** was used at a higher concentration (11 mM), the yields decreased from 27 (**11**) and 26% (**1**) to 12 (**11**) and 12% (**1**), respectively. Apparently, polymerization predominates at this concentration.

Holand **1** contains a cavity of nanosize dimensions. According to CPK models, the axes are about 1.5 and 2.0 nm long. The calculated internal volume is approximately 1.0 nm^3 (1000 $Å^3$). Figure 2 represents a picture of the energy-minimized structure

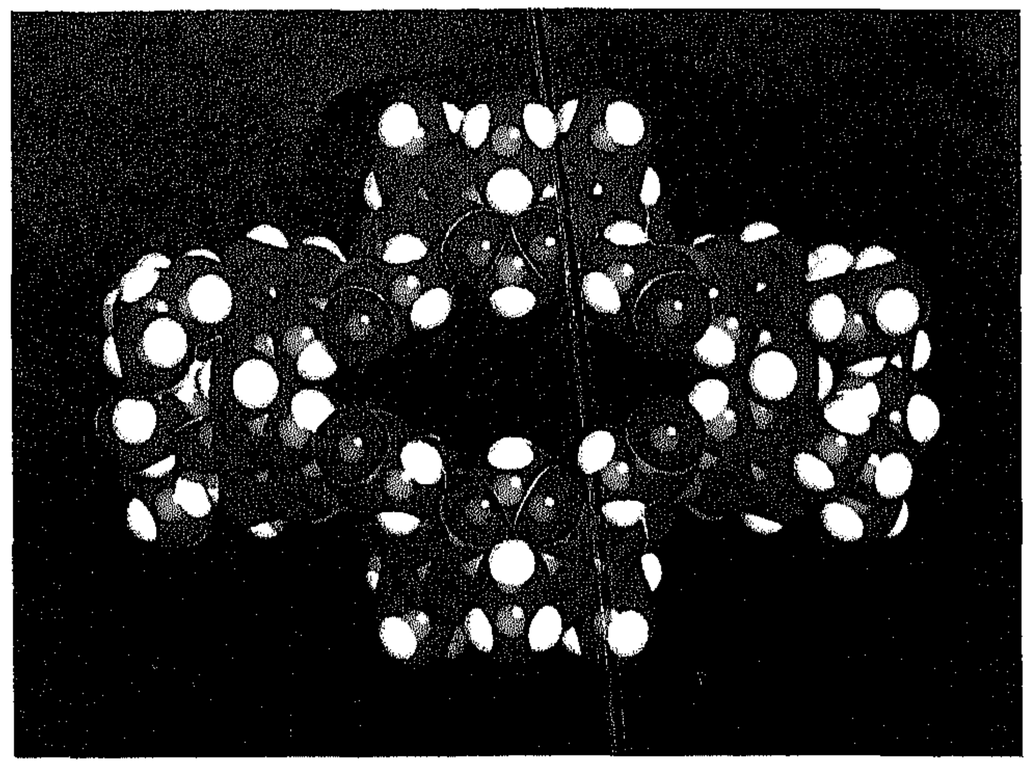

Fig. 2. Energy-minimized structure of holand **1** (undecyl chains have been replaced by methyl chains for simplicity).

of **1**. This minimized structure was subjected to a 50 ps dynamics simulation at 300 K in a $CHCl_3$ box with 46 Å edges. During this simulation four chloroform molecules entered the cavity and stayed there for the rest of the simulation without changing the shape of the cavity. This supports likewise the rigid structure of **1**. The organization of the amide spacers was clearly observed in this simulation. Although the hydrogen bonds were sometimes broken, they reformed after a short period of time. Rotation around the C(arene)–N bond was not observed.

Holand **1** is expected to have unique complexation properties. The size of the cavity permits complexation of host molecules which themselves are good complexing agents. Such complexation studies are currently under investigation.

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[17] The configuration was determined by using NOE spectroscopy. In a compound very similar to **5c** in which the undecyl chains have been replaced by methyl chains, a short-distance contact was observed between the protons of the unsubstituted aromatic rings and the protons of the methyl groups, indicating that this compound adopts the *exo* configuration. The configurations of all the other compounds could be easily determined by comparing their ^{1}H NMR spectra with that of **5c**.
[18] Compounds **5a–c** have large hydrophobic surfaces and show selective complexation of several corticosteroids in $CDCl_3$ solutions. These results will be described elsewhere.
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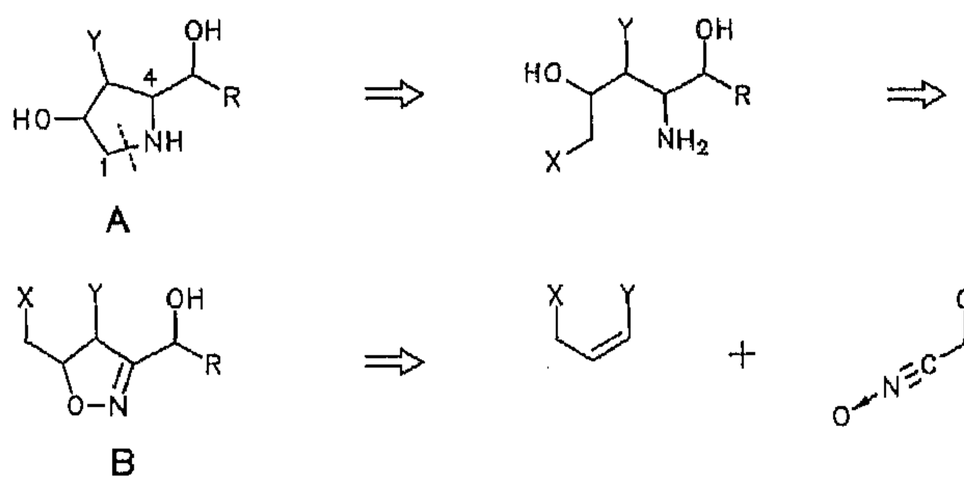
[20] The crude reaction mixture was first subjected to a silylation reaction with 20 equiv of *tert*-butyldimethylsilyl chloride, 20 equiv of NEt_3, and a catalytic amount of dimethylaminopyridine in CH_2Cl_2 for 24 h at room temperature, because of the instability of **7** on silica gel.
[21] This incarceration of solvent molecules has been frequently observed by Cram, see ref. [15] and also: a) D. J. Cram, S. Karbach, Y. H. Kim, L. Baczynskyi, K. Marti, R. M. Sampson, G. W. Kalleymeyn, *J. Am. Chem. Soc.* **1988**, *110*, 2554–2560; b) J. C. Sherman, D. J. Cram, *ibid.* **1989**, *111*, 4527–4528; c) J. A. Bryant, M. T. Blanda, M. Vincenti, D. J. Cram, *J. Chem. Soc. Chem. Commun.* **1990**, 1403–1405. The stability of **7** was determined by an exchange experiment in [D_7]DMF at 100 °C for 1 h. No exchange of DMF was observed at this temperature. Further work on these carcerands will be described elsewhere.
[22] Compound **12** was obtained from **8a** in quantitative yield in a similar way as **10** was prepared starting from **9**.
[23] Evidence for these hydrogen bonds was obtained from a quantitative NOE analysis performed with **5a** in which the distance between the aromatic protons *ortho* to the amide and the outer methylene proton was determined to be 2.5 Å and the distance between the amide protons and the outer methylene proton was determined to be 2.8 Å.

A New Synthesis of 1,3,4-Trideoxy-1,4-iminoglycitols of Varying Chain Length by (C_3 + C_n)-Coupling of Allyl Halides with Glyconitrile Oxides**

Rudolf Müller, Thomas Leibold, Michael Pätzel, and Volker Jäger*

Dedicated to Professor Heinz Günther Viehe on the occasion of his 65th birthday

Iminoglycitols such as **A** have attracted attention both as target compounds and as intermediates for polyhydroxylated N-bicycles. Some of the well-known representatives of this series that often act as powerful glycosidase inhibitors are pyrrolidine derivatives such as 1,4-dideoxy-1,4-imino-L-arabinitol (LAB)[2] and -D-lyxitol,[3–5] piperidines such as deoxynojirimycin (DNJ) and DNJ derivatives or analogues,[6] as well as indolizidine derivatives of the swainsonine[7] or castanospermine type.[8] The increasing interest in simple syntheses of specific compounds and derivatives with related unnatural structures stems from their potential for pharmaceutical application: inhibition of the corresponding enzymes is often accompanied by diverse physiological effects (anti-retroviral, -bacterial, -tumoral effects; reduction of the blood-sugar level). Topographical equivalence with the corresponding glycoside[3, 6, 9] or pyranosyl cation[4, 6] generally serves as a guideline to find active products of this type. The aim of our investigations is to provide access to unnatural analogues in the pyrrolidine series—by omission of OH groups, by placing them in different positions, or by inclusion of additional ones. Recently, two routes to *cis*-dihydroxypyrrolidines, based on a nitroaldol reaction and aminopentenediol cyclizations, have been presented.[5] Here we describe a third approach to 4-hydroxypyrrolidines (1,3,4-trideoxy-1,4-iminoglycitols) for which the highly selective, catalytic hydrogenation of 5-halomethyl-4,5-dihydro-1,2-oxazoles (**B**) has been developed as a new key step (Scheme 1). This route is exemplified in detail by the sequence leading to L-*ribo*-hexitol **5** and to the diastereomers **6–8** (Scheme 2).



Scheme 1. Retrosynthesis of 1,4-iminoglycitols (hydroxypyrrolidines) **A** by the isoxazoline route. Y = H, R = $(CHOH)_xH$: this work; Y = OH, R = $(CHOH)_xH$: Refs. [10,19]. The hydroxypyrrolidines **A** are named as 1,4-iminoglycitols to facilitate the comparison with the structures and configurations of natural glycosidase substrates.

The glyceraldehyde **1**, available from diethyl L-(+)-tartrate,[13] was converted into the hydroxamic acid chloride **2** by oximation/chlorination.[12a,b] Cycloaddition of the nitrile oxide with allyl chloride according to Huisgen's in situ method[12b] gave, as expected,[14] the 5-chloromethyl-dihydro-1,2-oxazoles **3/4** as an approximately 1:1 mixture of diastereomers, each of which was isolated in 46% yield (in gram quantities) after chromatographic separation. Thus, an entry to two series of stereoisomers is provided (actually to four if the D-tartrate is also taken into account).

The catalytic hydrogenation of **3** and **4** in the presence of platinum on charcoal gave, in each case, the corresponding *trans*-hydroxypyrrolidine as major product. Thus, the 2-*O*-benzyl derivatives (L-*ribo*/L-*xylo*) were formed from **3** with a diastereomer ratio (*dr*) of 93:7, and the L-*lyxo*/L-*arabino*-isomers from **4** with *dr* = 80:20. The free iminoglycitols **5/6** and **7/8**, respectively, were obtained after removal of the O-protecting benzyl group with H_2/palladium on charcoal. The major isomer **5** was obtained pure in 73% yield from the mixture of **5/6** by crystallization (*dr* > 95:5 according to ^{1}H and ^{13}C NMR). The bromo compounds (from **2**/Et_3N and allyl bromide) corre-

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