were observed; $^{31}P\{^1H\}$ NMR (C_6D_6, 85% H_3PO_4 external): $\delta = -319.4$ (K_2PR^*). The reaction of KR* and PCl_3 in the molar ratio 2:1 gave quantitative amounts of R*ClP–PClR* ($\delta(^{31}P) = 188$).[2]

3d: A solution of **1b** (0.199 g, 0.500 mmol) in THF (2 mL) was added to CuI (0.160 g, 0.840 mmol), and after 1 h the insoluble components were removed by centrifugation. Hexameric dicopper supersilylphosphanediide (**3d**) crystallized in the form of yellow-green prisms (yield ca. 50%) from the remaining solution in the course of five days. The compound **3d**, which was insoluble in organic media, was characterized by X-ray structure analysis (Figure 5).

Received: December 27, 1999 [Z 14465]

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[15] Crystal structure analyses: **3a**: orthorhombic, space group $P4(2)/n$, $a = 17.9170(2)$, $c = 13.0721(2)$ Å, $Z = 9$, $V = 4196.39(9)$ Å^3, 2439 independent reflections ($I > 2\sigma(\mathrm{I})$). All non-hydrogen atoms were refined anisotropically and the H atoms were included in calculated positions. $R1 = 0.0904$ (observed reflections), $wR2 = 0.1984$ (all data). **3c**: monoclinic, space group $P2(1)/m$, $a = 16.320(2)$, $b = 22.210(4)$, $c = 18.410(4)$ Å, $\beta = 108.46(2)°$, $Z = 4$, $V = 6329.5(1)$ Å^3, 10419 independent reflections ($I > 2\sigma(I)$). All non-hydrogen atoms were refined anisotropically and the H atoms were included in calculated positions. $R1 = 0.099$ (observed reflections), $wR2 = 0.265$ (all data). The cell contained twelve solvent molecules (C_6H_6). The $R1$ value could not be improved further as a result of the poor quality of the crystal and a disorder of the *t*Bu groups bound to Si as well as the cocrystallizing benzene molecules. **3d**: trigonal, space group $R\bar{3}$ (no. 148), $a = 14.742(3)$, $c = 39.586(7)$ Å, $Z = 3$, $V = 7451(2)$ Å^3, 2488 independent reflections ($I > 2\sigma(I)$). All non-hydrogen atoms were refined anisotropically and the H atoms were included in calculated positions. $R1 = 0.0541$ (observed reflections), $wR2 = 0.1334$ (all data). The intensities were measured with a Siemens-P4 apparatus with a CCD area detector (**3a**), a Stoe imaging plate (**3c**), and a Nonius Mach-3 appratus (**3d**) ($Mo_{K\alpha}$ radiation, $\lambda = 0.71073$ Å, respectively, ω scan, **3c**: $\phi = 0-120°$; $\Delta\phi = 0.3°$; 10 min irradiation time), $T = 173(2)$, 193(2), and 293(2) K), respectively. The structures were solved by direct methods (SHELXS-97, SHELXL-93) and refined with full matrix against F^2. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-138960 (**3a**), CCDC-139064 (**3c**), and CCDC-138956 (**3d**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Confinement of $[(H_2O)_2 \cap ([18]\mathrm{Crown}\text{-}6)]$ in a Disc-Shaped Cavity Lined with Six Nickel(II) Macrocycle Dimers Acting as Divergent Receptor Molecules[**]

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Krisztian Baranyai

The nickel(II) complex [Ni(tmtaa)] (Equation (1)) and its analogue with two methyl groups on each of the phenyl rings of the ligand, [Ni(omtaa)], are versatile receptors for a range of globular molecules.[1–4] A 1:1 complex between [Ni(tmtaa)] and fullerene is known, in which the macrocycle acts as a divergent receptor for C_{60}. A fullerene perches in each macrocycle cavity in an infinite zigzag array.[1] A similar structure is found for C_{70} with [Ni(omtaa)].[3] For smaller globular molecules, including 1,2-dicarba-*closo*-dodecaborane(12), tetraphosphorus trisulfide, tetraphosphorus triselenide, and *cyclo*-octasulfur, the [Ni(tmtaa)] complex self associates through aromatic faces into dimers and these dimers then act as divergent homotopic receptors with a 1:2 ratio of guest:host

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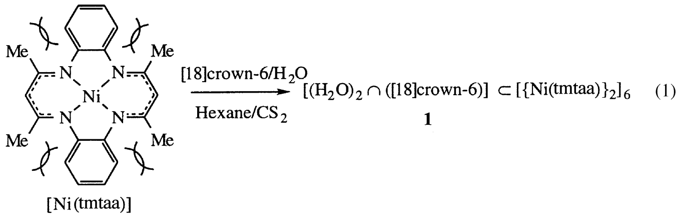
[**] This work is supported by the Australian Research Council.

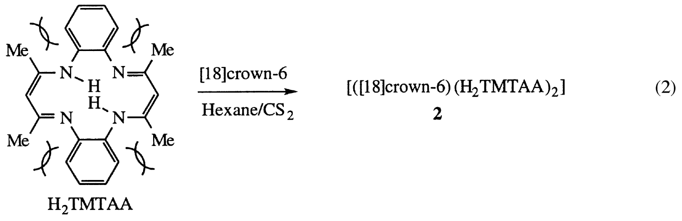
species.[1, 2] Inclusion complexes with [Ni(tmtaa)] form with the solvent toluene; the unsolvated form has been found to have at least three different structures.[2, 5, 6]

Complementary sizes and curvatures of the interacting components seems to be a prime determinant of the supramolecular outcomes. The molecule Ni(tmtaa) is rigid and distorts into a saddle shape of C_{2v} symmetry as a consequence of the methyl groups otherwise clashing with the H-atoms on the *ortho*-carbon atoms of the phenyl rings. In some of these structures the metal macrocycle self associates through the methyl-lined faces into dimers.[2]

While the complexation chemistry of the $TMTAA^{2-}$ ligand to a range of metals, metalloids, and nonmetals is well developed,[7] its supramolecular chemistry is less known,[1, 2] and the control of the assembly process to build engineered solid-state architectures is problematic. We now report remarkable findings on the host–guest chemistry of [Ni(tmtaa)]: The extended self association of the divergent receptors around a large disc-shaped cavity which contains an [18]crown-6 unit linked via two hydrogen-bonded water-molecule bridges. Furthermore, the potential for the metal-free molecule H_2TMTAA, which also has a saddle shape, towards forming self assembled complex with [18]crown-6 is established. This complex has a different stoichiometry and supramolecular synthon interplay relative to the [Ni(tmtaa)]–crown ether complex.

The new compounds **1** and **2** were prepared by the slow evaporation of solutions of the macrocyclic complex with [18]crown-6 in CS_2/hexane with a trace of water [Eq. (1) and (2)]. Compound **1** was isolated from reactions involving 1:1 and 1:12 ratios of crown ether to macrocycle. These mole ratio experiments demonstrate the propensity of **1** to crystallise from solution despite the weak intermolecular interactions. In contrast, **2** only formed with excess [18]crown-6. UV/Vis studies of mixtures of [Ni(tmtaa)] and [18]crown-6 in CS_2 and toluene indicate strong interaction in solution (absorption changes at submillimolar concentrations) but it was not possible to determine the nature of the complexes formed in solution or whether supermolecules of **1** are present.


$$[(H_2O)_2 \cap ([18]\text{crown-6})] \subset [\{\text{Ni(tmtaa)}\}_2]_6 \quad (1)$$


$$[([18]\text{crown-6})(H_2TMTAA)_2] \quad (2)$$

Compound **1** crystallises[8] in the space group $R\bar{3}(h)$; the supermolecules reside on the $\bar{3}$ symmetry sites with the asymmetric unit comprised of two molecules of [Ni(tmtaa)], one sixth of a molecule of [18]crown-6, and a water molecule lying on a $\bar{3}$ symmetry axis (Figure 1). Dimers of $([Ni(tmtaa)])_2$ are locked through the pendant aromatic rings of the saddle-shaped monomers, an outcome of the complementarity of curvature between the two components as well as Ni-π_{arene} interactions. The [18]crown-6 is cradled within the methyl-lined faces of six of these dimers. The divergent receptor prowess of the dimers[1, 2] is borne out by one methyl-lined face directed towards the crown ether and the other towards a neighboring supermolecule dimer: A pair of methyl groups of one dimer lies in the concave cavity of another dimer and vice versa. Thus, each unit of **1** is linked to six other units, as depicted in cartoon form for the central supermolecule in Figure 2. The two interaction styles described for the [Ni(tmtaa)] complex ("locking" of pendant aromatic rings and of methyl-lined faces) have been described previously.[2]

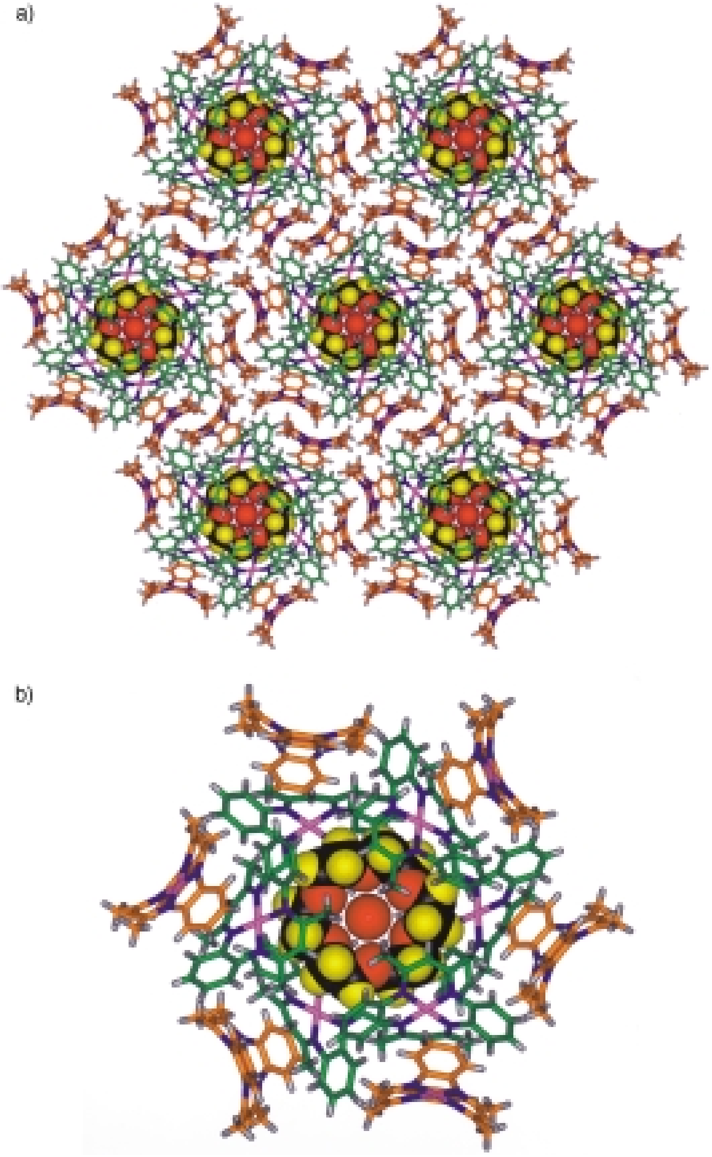


Figure 1. Crystalline structure of **1**: a) the extended structure (see Figure 2), and b) a supermolecule.

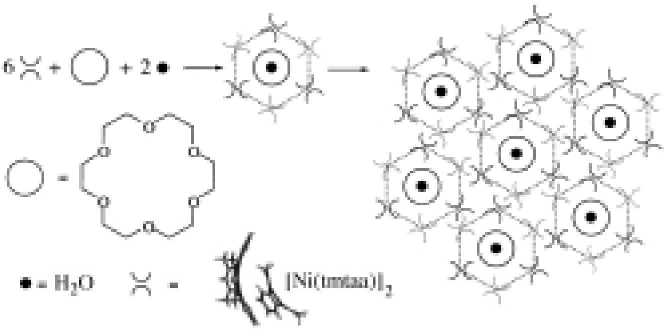


Figure 2. Simplified representation of the crystalline structure of **1**.

The closest approach of the crown ether to the [Ni(tmtaa)] molecules are the O-C$_{methyl}$, O-C$_{phenyl}$, and O-N$_{imine}$ distances of 3.99(1), 3.96(1), and 3.92(1) Å, respectively and C-C$_{methyl}$, C-C$_{phenyl}$, and C-N$_{imine}$ distances of 4.08(1), 3.83(1), and 3.75(1) Å, respectively. Two water molecules perch on either side of the crown ether and the oxygen atoms of the water molecules are 3.44(2) Å apart. The short 3.19(1) Å distances beween each O$_{H_2O}$ atom and three crown-ether O$_{crown}$ atoms are consistent with hydrogen bonding. The three longer contacts for the alternate crown-ether oxygen atoms is 3.36(1) Å. The closest approach of the O$_{H_2O}$ atom to [Ni(tmtaa)] are to methyl groups at 3.29(1) Å (Figure 1 (b)).

The supermolecule **1** (Figure 1 b), comprised of 15 interacting components, is held together by hydrogen bonding, π-interactions, and van der Waals interactions. The shrouding of $[(H_2O)_2 \cap ([18]\text{crown-6})]$ by six dimers is arguably a shape-induced process; however, in the absence of the crown ether, crystals have been obtained with the same unit cell parameters and with a similar arrangement of [Ni(tmtaa)] dimers around the same shape and size cavity, in this case occupied by a highly disordered CS$_2$ molecule $((CS_2) \subset [\{Ni(tmtaa)\}_2]_6)$.[9]

Compound **2** crystallizes[8] in the space group $P\bar{1}$ with a molecule of H$_2$TMTAA and one half of an [18]crown-6 molecules in the asymmetric unit. Layers of (H$_2$TMTAA)$_2$ link through a mismatched overlap of methyl-lined faces, as described previously[2] (distances: C$_{methyl}$-C$_{methyl}$, C$_{methyl}$-C$_{imine}$, C$_{methyl}$-C$_{methine}$ are 3.904(3), 3.381(3), and 3.466(3) Å, respectively). These layers are separated by layers of discrete [18]crown-6 molecules (Figure 3). The H$_2$TMTAA layers consist of dimers (H$_2$TMTAA)$_2$ aligned with coplanar orientations of the pentanediimine fragments (about 4.0 Å apart) and result in the phenyl-lined surfaces presenting themselves to the crown ether layers on the top and bottom. The closest approaches of the crown ether to the H$_2$TMTAA molecule are [Å]: O-C$_{phenyl}$ 3.360(3), O-C$_{methyl}$ 3.509(3), C$_{[18]crown-6}$-C$_{phenyl}$ 3.554(4), C$_{[18]crown-6}$-C$_{methine}$ 3.683(3), C$_{[18]crown-6}$-C$_{methyl}$ 4.092(3), and C$_{[18]crown-6}$-N 3.739(3).

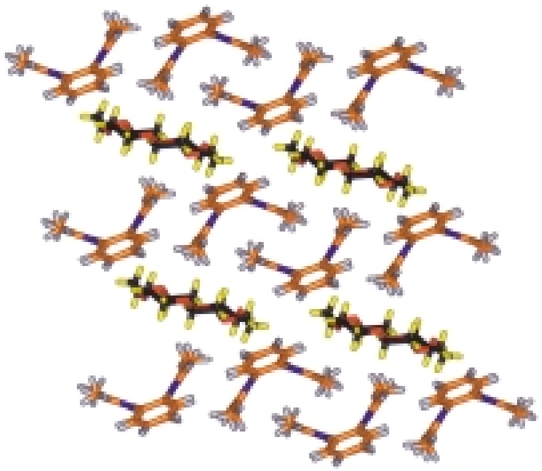


Figure 3. Packing of [18]crown-6 in **2**.

The ability of [Ni(tmtaa)] and H$_2$TMTAA to form novel inclusion complexes suggests that other binary/ternary systems are likely. We note that in **1** the macrocycle dimers are disposed to shroud the crown ether and associated water molecules. Larger crown-ether systems may be accommodated by rotation of the macrocycle dimers relative to each other when orientated closer to the equatorial plane of the supermolecule. A similar potential exists for [Ni(omtaa)] towards constructing new architectures, perhaps even with some predictability, upon replacing the metal centers by paramagnetic ones leading to materials with novel function.

Experimental Section

[Ni(tmtaa)] and H$_2$TMTAA were prepared according to published procedures.[10] Other reagents and solvents were commercial products.

1: A CS$_2$ solution of [18]crown-6 (5 mg, 20 mL) was mixed with a CS$_2$ solution of [Ni(tmtaa)] (102 mg, 20 mL). The mixture was layered with hexane (equal volume) and allowed to evaporate slowly. The complex was collected and washed with a small amount of hexane (m.p. 242–248 °C). Elemental analysis [%]: calcd: C 64.82, H 5.68, N 13.11; found: C 64.55, H 5.76, N 13.15.

2: A toluene solution of [18]crown-6 (13 mg, 20 mL) was mixed with a toluene solution of H$_2$TMTAA (34 mg, 20 mL). The mixture was layered hexane (equal volume) and allowed to evaporate slowly. H$_2$TMTAA crystallised from solution and the collected crystals were washed with hexane. From the filtrate and hexane washings, a small amount of **2** crystallised but was difficult to purify.

Received: November 12, 1999 [Z14263]

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[8] Crystal structure analysis for **1** and **2**: Enraf-Nonius Kappa CCD diffractometer, $T = 123$ K, crystal mounted in oil. **1**: $Ni_{12}C_{276}H_{292}N_{48}O_8$, $M = 5114.06$, trigonal, space group $R\bar{3}(h)$ (No. 148), $a = 41.573(6)$, $c = 11.646(2)$ Å, $V = 17432.8(1)$ Å^3, $\rho_{calc} = 1.461$ g cm^{-3}, $\mu = 10.22$ cm^{-1} (no correction), $Z = 3$, Mo$_{K\alpha}$ radiation, 11302 unique reflections ($R_{int} = 0.098$), $2\theta_{max} = 60°$ (5051 observed, $I > 3\sigma(I)$, $R = 0.064$, $Rw = 0.060$). Hydrogen atoms on water molecules showing disorder over three sites were not included in the calculations. **2**: $C_{56}H_{72}N_8O_6$, $M = 953.2$, triclinic, space group $P\bar{1}$ (No. 2), $a = 11.2248(4)$, $b = 11.3120(4)$, $c = 11.9329(3)$ Å, $\alpha = 63.367(2)°$, $\beta =$

$71.013(2)^\circ$, $\gamma = 72.862(2)^\circ$, $V = 1260.7(5)$ Å^3, $\rho_{calc} = 1.255$ g cm^{-3}, $\mu = 0.82$ cm^{-1} (no correction), $Z = 1$, Mo$_{K\alpha}$ radiation, 7007 unique reflections($R_{int} = 0.043$), $2\theta_{max} = 60^\circ$ (4661 observed, $I > 3\sigma(I)$, $R = 0.063$, $Rw = 0.067$). Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-136348 and -136349. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

[9] Synthesis of $(CS_2) \subset [\{Ni(tmtaa)\}_2]_6$. A CS_2 solution of [Ni(tmtaa)] (50 mg, 20 mL) was mixed with a CS_2 solution of anthracene (18 mg, 20 mL). The mixure was layered with hexane (equal volume) and allowed to evaporate slowly (m.p. 241 – 245 °C). Elemental analysis [%]: calcd: C 65.09, H 5.44, N 13.75; found: C 64.98, H 5.47, N 13.68. Crystal structure analysis: Enraf-Nonius Kappa CCD diffractometer, $T = 123$ K, crystal mounted in oil. $Ni_{12}C_{265}H_{264}N_{48}S_2$, $M = 4889.84$, $R\bar{3}(h)$ (No. 148), $a = 41.5300(17)$, $c = 11.6099(17)$ Å, $V = 17341.3(0.8)$ Å^3.

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Globular Amphiphiles: Membrane-Forming Hexaadducts of C_{60}[**]

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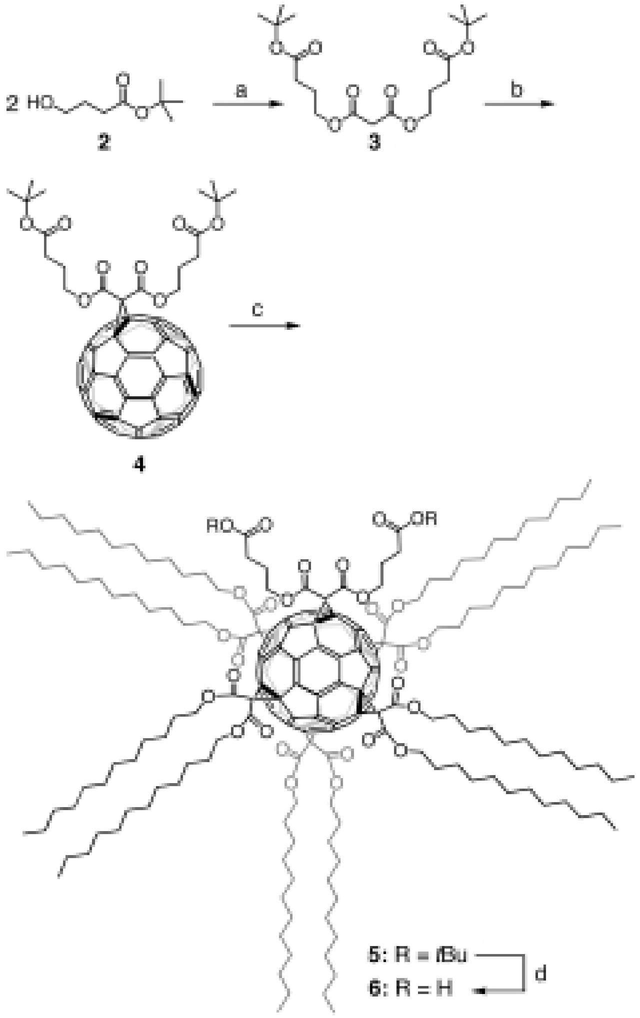
and Andreas Hirsch*

Most of the biological membrane-forming lipids are double-chain phospho- or glycolipids with 16 – 22 carbon atoms per chain and which may be mono- or polyunsaturated. They perform many functions in biological systems. At the interface between biology and materials sciences, the development of artifical nanostructures as mimics of natural systems or as prototypes of novel functional aggregates is playing an important role. This article describes the synthesis and the membrane and vesicle formation of a new class of artificial lipids. They have a globular structure with the spherical C_{60} being used to define the molecular geometry. Five pairs of dodecyl chains and one pair of polyamide dendrons are bound to C_{60} in an octahedral [1:5]-addition pattern[1] by methylene bridges.[2]

For a synthesis of the globular amphiphile **1** (see Scheme 2), the alcohol[3] **2** and malonyl dichloride were allowed to react to yield malonate **3**. A nucleophilic cyclopropanation reaction[4, 5] of C_{60} with **3** leads to the monoadduct **4** (Scheme 1).



Scheme 1. Syntheses of dicarboxylic acid **6**. a) Malonyldichloride, pyridine, CH_2Cl_2; b) C_{60}, DBU, CBr_4, toluene; c) 1) DMA, 2) CBr_4, DBU, didodecyl malonate, toluene; d) TFA, toluene. DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene, DMA = dimethylanthracene, TFA = trifluoroacetic acid.

The five pairs of lipophilic C_{12} chains, which are in octahedral positions relative to the first addend (Figure 1), were inserted in one step by cyclopropanation of **4** with didodecyl malonate,

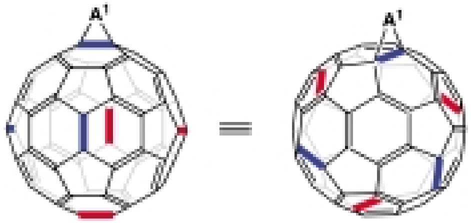


Figure 1. Octahedral positions relative to the first addend A^1 in a C_{2v} symmetrical hexaadduct of C_{60}.

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[**] This work was been supported by the Deutsche Forschungsgemeinschaft and by the Fonds der Chemischen Industrie. We thank Dr. K. Fischer and Prof. Dr. K. Schmidt from the Institute of Physical Chemistry at the University of Mainz for performing the light scattering measurements.

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