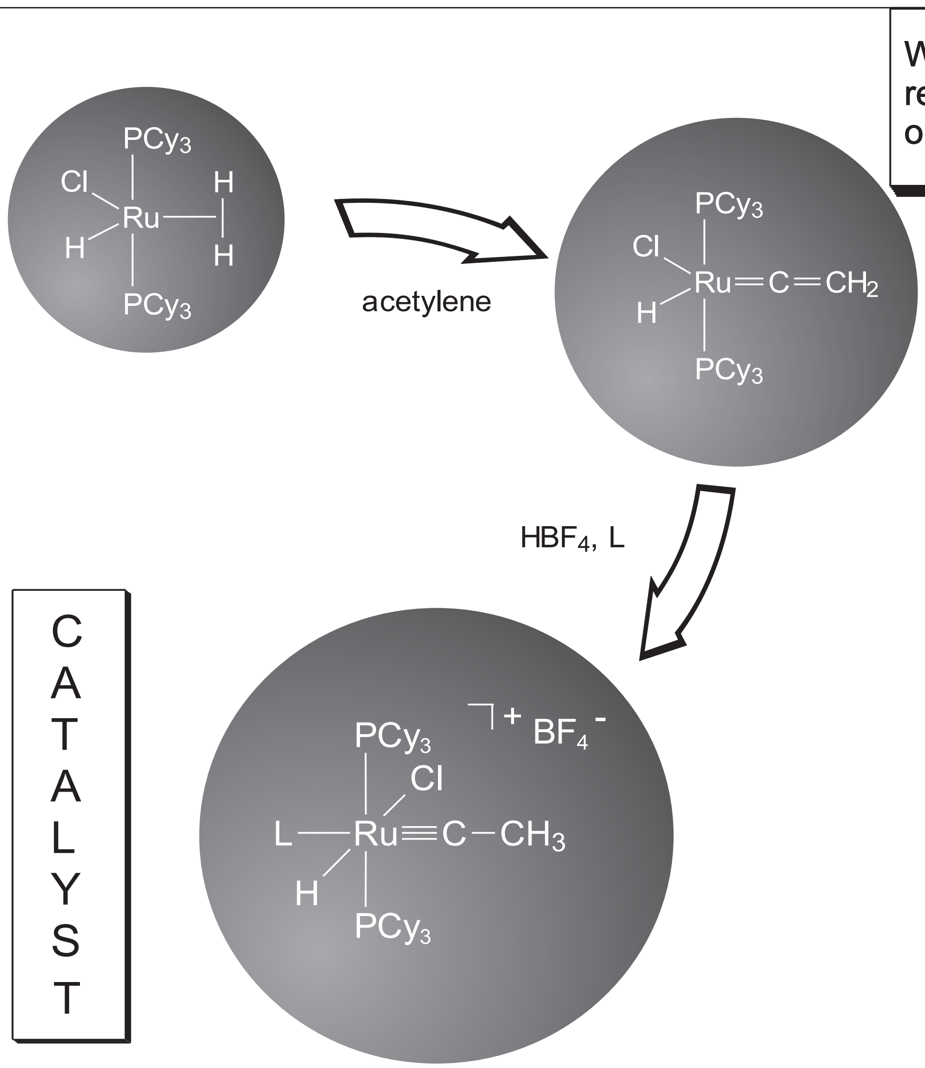
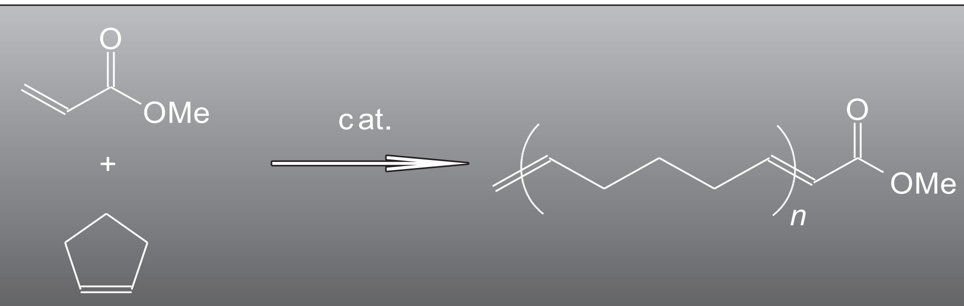
Werner and co-workers report more on this topic on pages 3421-3423.



In contrast to neutral carbeneruthenium compounds, cationic carbynehydridoruthenium complexes catalyze the cross-olefin metathesis of cyctopentene with methyl acrylate.

REACTION



Organizational Forms of Matter: An Inorganic Super Fullerene and Keplerate Based on Molybdenum Oxide

Achim Müller,* Erich Krickemeyer, Hartmut Bögge, Marc Schmidtmann, and Frank Peters

Dedicated to Professor Heinrich Nöth
on the occasion of his 70th birthday

A knowledge of the linking rules of selected building blocks allows, in special cases, the generation of an enormous variety of structures, especially when a set of gradually differing building units is available that can be "tuned" in terms of linkage variability. Of utmost importance is that these basic units can be linked to larger fragments, which can again react in subsequent processes in different ways.[1] Of interest is certainly not only the production of mesoscopic (or nanostructured) and macroscopic objects with functional versatility, but also the aesthetic beauty of such assemblies. In this respect it should be mentioned that certain structures, such as spherical objects comprised of pentagons and hexagons, can be found repeatedly both on molecular and macroscopic levels, clearly because general organization principles are followed. Here we report on a relevant inorganic super fullerene or, in other words a giant molecular sphere with an integrated (inscribed) icosahedron, namely a Keplerate (see below) with more than 500 atoms and the highest Euclidean symmetry. The sphere consists of a total of 12 $\{Mo_{11}\}$-polyoxomolybdate fragments of fivefold symmetry. These fragments are generated from central pentagonal bipyramidal $\{MoO_7\}$ groups, which are abundant in several other giant polyoxomolybdate clusters.[2]

When an aqueous solution of ammonium molybdate is reduced to medium ratios of $[Mo^V]/[Mo^{VI}]$ at pH values at which the pentagonal bipyramidal $\{MoO_7\}$ groups are "generated" and discrete $\{Mo_2^VO_4(OOCCH_3)^+\}$, that is, $\{Mo_2^V\}$ groups, are stabilized by high concentrations of the bidentate acetate ligand, the spherical anion **1a** or the corresponding crystalline salt **1**, respectively, is formed. High $[Mo^V]/[Mo^{VI}]$ ratios inevitably result in the direct linking of the resulting $\{Mo_2^V\}$[3] groups whereas low ratios lead to the formation of molybdenum-blue species.[2, 4] (In the following text only the oxidation state of the Mo^V and not of the Mo^{VI} centers is given.)

$$(NH_4)_{42}[Mo^{VI}_{72}Mo^{V}_{60}O_{372}(CH_3COO)_{30}(H_2O)_{72}] \cdot \text{ca. } 300\,H_2O \cdot \text{ca. } 10\,CH_3COONH_4 \quad \mathbf{1}$$

The salt **1** was characterized by elemental analysis (including cerimetric titration to determine the number of Mo^V centers), thermogravimetry (for the determination of the crystal water content), bond valence sum (BVS) calculations[5] (to determine the positions of the H_2O ligands and to distinguish between Mo^{VI} and Mo^V centers), spectroscopic methods (IR, Raman, UV/Vis, and NIR spectroscopy), and single crystal X-ray structure analysis.[6]

Compound **1** crystallizes in the cubic space group $Fm\bar{3}$.[6] The $\{Mo_{132}\}$-type cluster **1a** is built up of 12 $\{Mo_{11}\}$ fragments of the type $\{(Mo)Mo_5\}\{Mo_1^V\}_5$ (Figures 1 and 2) with central pentagonal bipyramidal $\{MoO_7\}$ groups, such that the fivefold symmetry axes are retained in the resulting spherical object, which shows an overall icosahedral symmetry, which includes the respective C_2 and C_3 symmetry axes (Figures 1 and 2). The central $\{MoO_7\}$ unit is linked to five $\{MoO_6\}$ octahedra through edges, with each Mo^V center of an $\{Mo_2^V\}$ group being linked to two adjacent $\{MoO_6\}$ octahedra. The basic spherical shape of **1a** is emphasized in Figure 2a, whereby the 12 corners of an icosahedron are spanned by the Mo centers of the pentagonal $\{MoO_7\}$ bipyramids of the $\{Mo/Mo_5\}$ pentagons. In all, the 12 $\{(Mo)Mo_5\}$ groups of the $\{Mo_{132}\}$-type cluster are linked by 30 $\{Mo_2^V\}$ groups, which are "stabilized"

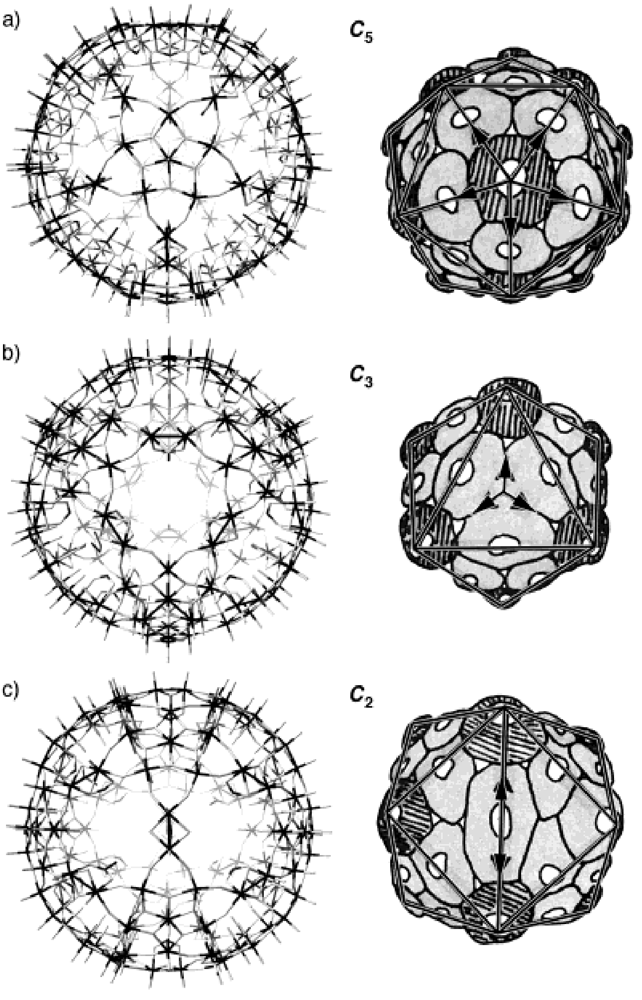


Figure 1. Illustration of the structure of **1a** with perspective views along a fivefold (a), a threefold (b), and a twofold (c) symmetry axis (because of the structural complexity of the spherical shape of **1a** in wireframe presentations). For the purpose of comparison the corresponding schematic representations of an icosahedral $T = 3$ virus capsid (see below) with 20 hexagonal and 12 pentagonal capsomers (morphology units) are presented. In both systems, the C_5 axes cross the centers of the pentagonal units (hatched) (a), the C_3 axes cross the midpoint between three units (b), and the C_2 axes cross the center of a unit (virus model figures are reproduced from reference [10c]). Whereas (a) refers to the centers of both the pentagonal $\{(Mo)Mo_5\}$ units and the pentagonal capsomers which are located at the 12 corners of an icosahedron (see Figure 2), (b) and (c) refer to the $\{Mo_2^V\}$ units of **1a** and the hexagonal capsomers, respectively.

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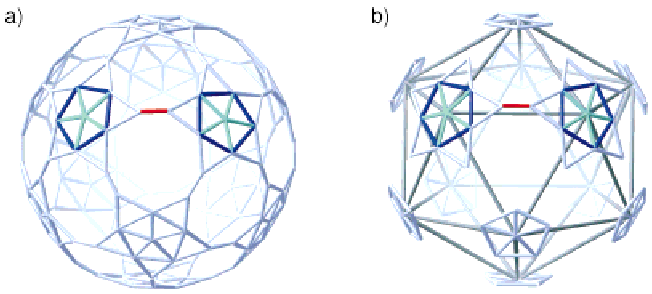

Figure 2. Schematic representation of the 132 molybdenum atom fragment of **1a** with the spherical nature highlighted (a) and the relation to the icosahedron spanned by the related $\{(\mathrm{Mo})\mathrm{Mo}_5\}$ subfragment located on the 12 corners (b). Two $\{\mathrm{Mo}_{11}\}$ units ($\{(\mathrm{Mo})\mathrm{Mo}_5\}\{\mathrm{Mo}^{\mathrm{V}}\}_5$, blue, light blue) that are linked through a $\mathrm{Mo}^{\mathrm{V}}-\mathrm{Mo}^{\mathrm{V}}$ bridge (red) are highlighted. The outer corners of an $\{\mathrm{Mo}_{11}\}$ unit are formed by the Mo centers of the $\{\mathrm{Mo}^{\mathrm{V}}-\mathrm{Mo}^{\mathrm{V}}\}$ bridges.

by bidentate acetate ligands (Figure 2b). Therefore, **1a** can be formulated as $[\{\mathrm{Mo}^{\mathrm{V}}_2\mathrm{O}_4(\mathrm{CH}_3\mathrm{COO})\}_{30}\{(\mathrm{Mo})\mathrm{Mo}_5\mathrm{O}_{21}(\mathrm{H}_2\mathrm{O})_6\}_{12}]^{42-}$. Interestingly, the 30 classical $\{\mathrm{Mo}^{\mathrm{V}}_2\}$ dumb-bells (Mo–Mo: 2.6 Å) or 60 Mo^{V} atoms span a solid of icosahedral symmetry, which corresponds to that of the C_{60} fullerene (Figure 3),[7] in the present case a polyhedron with 12 regular pentagons and 20 trigonal hexagons. (It was possible to resolve several disordered $\mathrm{H}_2\mathrm{O}$ molecules inside the sphere, a fact that is also interesting for related aspects of supramolecular chemistry.) Remarkable is that the anions **1a**, in which all O atoms of the 132 $\mathrm{Mo-O_{term}}$ groups lie on the outer-spherical surface and correspondingly the *trans*-positioned $\mathrm{H}_2\mathrm{O}$ ligands of the 72 $\mathrm{Mo}^{\mathrm{VI}}$ centers point to the center of the sphere, form a cubic close packing in the crystal (Figure 4).

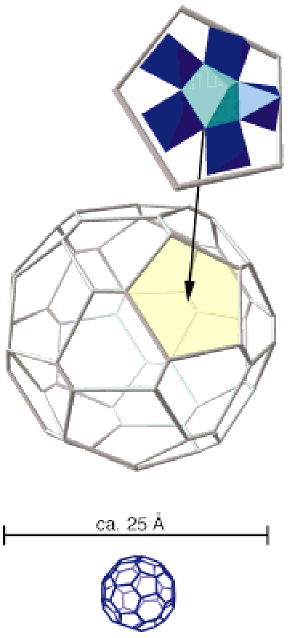

Figure 3. Structure of the icosahedral $\{\mathrm{Mo}^{\mathrm{V}}_2\}_{30}$ fragment with 12 regular pentagons and 20 trigonal hexagons as well as its coherence to the C_{60} fullerene, which is depicted on the same scale. A separated $\{(\mathrm{Mo})\mathrm{Mo}_5\}$ pentagon in a polyhedral representation is emphasized.

Furthermore, the anion **1a** belongs to the type I category within the Robin–Day classification for mixed-valence compounds,[8] which is evident from the red-brown color of the substance as well as the main absorption band at 450 nm, respectively, without any knowledge of the actual localization of the Mo^{V} and $\mathrm{Mo}^{\mathrm{VI}}$ centers.

In the current context the recognizable topological coherence to the structures of the spherical viruses, which have icosahedral capsids, should be stressed. Corresponding virus capsids—similar to a fragment of the described cluster **1a**—consist of 60 (identical) structural motifs. Interestingly, a spherical shell/capsid of icosahedral symmetry is preferred even for viruses[7c, 9, 10] with more than 60 subunits (protein monomers), though there are only 20 equilateral triangles (and for each of these only three equivalent subunits), a situation which can be understood from the rules of Caspar and Klug, who were inspired by the designs of the geodesic domes of the architect Buckminster Fuller. In the present case the centers of 12 $\{(\mathrm{Mo})\mathrm{Mo}_5\}$ pentagons with the (12 × 5) MoO_6 subunits, which are analogous to the 12 pentagonal capsomers (morphology units) of the most simple virus with the (12 × 5) protein monomers span an icosahedron. The corresponding partial structure of **1a** can thus topologically be compared to that of the most simple satellite tobacco necrosis virus (STNV) with its (only) 60 monomeric protein subunits (Figure 5).[9, 10d, e] The crucial difference between the polyoxometalate **1a** and the satellite virus is that the structure **1a** is formed by covalent bonds. Of special interest for the inorganic chemist is of course the fact that the described cluster **1a** resembles icosahedral symmetry despite its large number of atoms.[11] This represents definitively an unprecedented event in chemistry. Plato and Kepler, but probably Heisenberg too, would have been pleased.

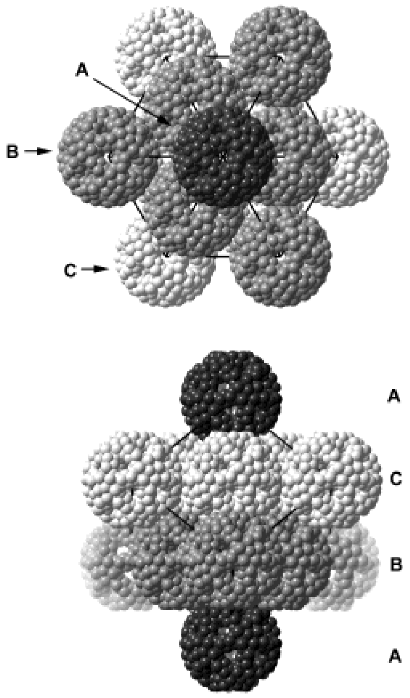

Figure 4. Representation of the packing of **1a** in the crystal lattice (space-filling model viewed along [111] (top) and perpendicular to [111] (bottom)). The spherical anions, the outer surface of which is formed by terminal oxygen atoms, are organized in a cubic–closest packing (sequence of layers ABCA…). Each edge of the additionally projected unit cell measures 46.06 Å.[15]

It can certainly be assumed that the variety of linking modes of molybdate fragments has not yet been exhausted and that it will be possible to obtain even macroscopic structures—that is, structures perceptive to our senses—in open systems. In this actual context the question arises as to whether limits exist for the molecular growth of very large assemblies formed by self-aggregation of subunits, especially in the case of objects of high, for example spherical, symmetry. In this respect, further studies not only on topological aspects

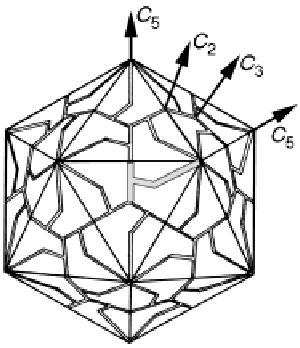

Figure 5. Representation of the packing of the 60 identical subunits of the $T = 1$ satellite tobacco necrosis virus, which are coded by only one gene.[10d] The linkage points lie on the five-, three- and twofold symmetry axes (see also Figure 1).

of carbon fullerenes[12] but also on Fuller cages in solid-state structures[13] would be stimulating. Here the comparison of **1a** with Kepler's early and famous model of the cosmos is certainly of interest. In his opus *Mysterium Cosmographicum*[14] Kepler described his vision of planetary motion, whereby distances between the orbits of the planets could be explained for him if the ratios between the successive orbits were designed to be equivalent to the spheres successively circumscribed around and inscribed within the five Platonic solids. In the case of **1a** the icosahedron inside the spherical shell of the 132 terminal oxygen atoms can be recognized. The 12 corners of the icosahedron are defined by the 12 central molybdenum atoms of the 12 $\{(Mo)Mo_5\}$ pentagons (Figure 2b). Thus we suggest that the anion **1a** should be called a Keplerate since the systematic nomenclature would be far too unwieldy.

We are convinced that **1a**, based on its high stability in solution, its cage structure, and the possibility to take-up (or exchange) certain species in its cavity, and also the possibility to modify the shells (for example to integrate hetero atoms) or link them, will lead to the development of a Keplerate-type chemistry comparable to Keggin-type chemistry. Solid-state chemistry additionally may profit from the opportunity to study nucleation and structure-forming processes on rather large molecular spherical objects, for instance to understand principles of crystal growth, and also in the search for shell aufbau principles in solid-state structures that show condensed fullerene cages.[13] Last but not least, it can be expected that the planned synthesis of highly complex molecular objects with desired functionality on the basis of metal–oxygen fragments will be possible in the future.

Experimental Section

1: $N_2H_4 \cdot H_2SO_4$ (0.8 g, 6.1 mmol) was added to a solution of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ (5.6 g, 4.5 mmol) and CH_3COONH_4 (12.5 g, 162.2 mmol) in H_2O (250 mL). The solution was then stirred for 10 min (color change to blue-green) and 50% CH_3COOH (83 mL) was subsequently added. The reaction solution, now green, was stored in an open 500-mL Erlenmeyer flask at 20 °C without further stirring (fumehood; slow color change to dark brown). After 4 d the precipitated red-brown crystals of **1** were filtered off over a glass fritt (D2), washed with 90% ethanol, ethanol, and diethyl ether, and finally dried in air. Yield: 3.3 g (52% based on molybdate).

Characteristic IR bands of the basic cluster vibrations assignable to the irreducible representation F_{1u} (KBr pellet): $\tilde{\nu}$ [cm^{-1}]: 1626 (m, $\delta(H_2O)$), 1546 (m, $\tilde{\nu}_{as}(COO)$), 1440 (sh) and 1407 (m) ($\delta(CH_3)$, $\tilde{\nu}_s(COO)$, ($\delta_{as}(NH_4^+)$), 969 (m) and 936 (w–m) ($\tilde{\nu}$ (Mo=O)), 853 (m), 792 (s), 723 (s), 628 (w), 567 (s); characteristic Raman bands assignable to the irreducible representations A_{1g} (those with highest intensity) and H_g (KBr pellet; $\lambda_e = 1064$ nm; 1000–200 cm^{-1}): $\tilde{\nu}$ [cm^{-1}]: 953 (w), 935 (w) and 875 (s) ($\tilde{\nu}$(Mo=O)), ca. 845 (sh), 374 (m–s), 314 (m), 212 (w); Vis/NIR spectra (in H_2O/CH_3COOH; λ [nm]): 450, 260 (sh), 215 (KBr pellets gave basically the same spectrum).

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[1] a) To our knowledge the following conditions and circumstances favor the generation of a variety of complex molecular structures in the present case (over several reaction steps) under one-pot conditions: 1) The presence of linkable groups, that is building units, with the general possibility to generate intermediates with high free enthalpy, which lead to high negative reaction enthalpies (for example, by formation of H_2O molecules); 2) the *possibility* of an easy structural change of the building units, of the uptake of hetero atoms, of the formation of larger and variably linkable fragments (especially with energetically low-lying unoccupied molecular orbitals), of a template-driven structure formation, of the generation of defects in reaction intermediates (with the consequence of different types of linking), of the localization and delocalization of valence electrons in different ways and the variation and control of the charge (for example by protonation, electron transfer reactions, and substitution of certain building units). b) In this context a novel *(polyoxometalate) nucleation growth* process has even been discovered inside a ring-shaped cluster compartment (A. Müller, S. Q. N. Shah, H. Bögge, M. Schmidtmann, *Nature*, in press). Aspects of related limited growth have been discussed: P. Ball, *Nature* **1998**, *395*, 745–748.

[2] See A. Müller, F. Peters, M. T. Pope, D. Gatteschi, *Chem. Rev.* **1998**, *98*, 239–271.

[3] A. Müller, J. Meyer, E. Krickemeyer, C. Beugholt, H. Bögge, F. Peters, M. Schmidtmann, P. Kögerler, M. J. Koop, *Chem. Eur. J.* **1998**, *4*, 1000–1006; A. Müller, S. Dillinger, E. Krickemeyer, H. Bögge, W. Plass, A. Stammler, R. C. Haushalter, *Z. Naturforsch. B* **1997**, *52*, 1301–1306; M. I. Khan, J. Zubieta, *Progr. Inorg. Chem.* **1995**, *43*, 1–149; H. K. Chae, W. G. Klemperer, T. A. Marquart, *Coord. Chem. Rev.* **1993**, *128*, 209–224.

[4] A. Müller, M. Koop, H. Bögge, M. Schmidtmann, C. Beugholt, *Chem. Commun.* **1998**, 1501–1502, and references therein.

[5] I. D. Brown in *Structure and Bonding in Crystals*, *Vol. II* (Eds.: M. O'Keeffe, A. Navrotsky), Academic Press, New York, **1981**, pp. 1–30.

[6] Space group $Fm\bar{3}$; $a = 46.0576(14)$ Å, $V = 97702(5)$ Å^3; $Z = 4$; the structure was solved with direct methods, $R = 0.0883$ for 3445 independent reflections ($F_0 > 4\sigma(F_0)$); Siemens-axs-SMART diffractometer (94920 reflections obtained at 158 K from 1271 frames each covering 0.3° in ω, $2\theta_{max} = 45°$). The crystals were taken directly from the reaction solution and measured immediately to prevent loss of crystal water. The measurement of several individual crystals led to the same results. An untypical coordination of an additional acetate group instead of H_2O ligands at the $\{(Mo)Mo_5\}$ pentagons, observed in a similar compound, cannot be completely excluded because of the disorder present; but in any event this coordination is definitely not necessary for the structure formation. (There is no coordination of this type in the corresponding $\{Mo_{132}\}$-type cluster that contains formate instead of acetate ligands.) The structures were solved with the program SHELXS-96 and refined with the program SHELXL-93 (G. M. Sheldrick, University of Göttingen, **1996** and **1993**, respectively). Further details on the crystal structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany (fax: (+49)7247-808-666; e-mail: crysdata@fiz-karlsruhe.de), on quoting the depository number CSD-410097.

[7] a) H. W. Kroto, J. R. Heath, S. C. O'Brien, R. F. Curl, R. E. Smalley, *Nature* **1985,** *318*, 162–163; b) *Buckminsterfullerenes* (Eds.: W. E.

Billups, M. A. Ciufolini), VCH, Weinheim, **1993**; c) *Von Fuller bis zu Fullerenen, Beispiele einer interdisziplinären Forschung* (Eds.: W. Krätschmer, H. Schuster), Vieweg, Braunschweig, **1996**.

[8] M. B. Robin, P. Day, *Adv. Inorg. Chem. Radiochem.* **1967**, *10*, 247–422.

[9] Spherical viruses display a 60-fold identically packed fundamental structural motif that can formally be assigned to pentagonal and also hexagonal capsomers (morphology units or chemically definable oligomers of one or more viral proteins or subunits). The most simple example is presented by the satellite tobacco necrosis virus (Figure 5), in which only 12 pentagonal capsomers with a total of 60 identical viral proteins (subunits) coded by one gene only are placed at the 12 corners of an icosahedron. In the case of the larger spherical viruses—for example, those with $3 \times 60 = 180$ subunits—the motif that occurs 60 times shows (different) subunits organized in capsomers of five identically connected protein monomers (pentagons) centered at the corners and 20 additional capsomers (hexagons) built up by six protein monomers (see Figure 1). In accordance with Caspar and Klug[10a, h] the triangular faces of an icosahedron can be subdivided into an integral number of equilateral triangles. This procedure demonstrates that the "normal" spherical viruses, such as that of the TBSV (tomato bushy stunt virus) can have more than 60 subunits (which is also the case for **1a**). The resulting solid is an icosadeltahedron (a capped icosahedron is such a solid) with local symmetry elements that relate to its subunits in addition to the exact symmetry elements of the icosahedron. (Subunits that are distributed about each exact triangular vertex of a $T = 3$ icosadeltahedron are related by a local C_6 symmetry axis; corresponding adjacent subunits are only quasi-equivalent.) The number of subunits in an icosadeltahedron equals $60\,T$ (where $T = h^2 + hk + k^2$ denotes the triangulation number, which is important for the architecture of viruses; h and k are integers[10a, d, h]). A virus structure with $T = 3$ (Figure 1) resembles 180 ($= 3 \times 60$) subunits (for $h = k = 1$) whereas the triangulation number $T = 1$ (with $h = 1$, $k = 0$) stands for the only 10 nm large satellite tobacco necrosis virus, and also for the fragment of **1a** with its 60 $\{MoO_6\}$ octahedral subunits of the 12 $\{(Mo)Mo_5\}$ pentagons. For a known, topologically comparable geodesic dome of Fuller $T = 36$[10a] (for extensive literature on this subject see reference [10]).

[10] a) See D. Voet, J. G.Voet, *Biochemie*, VCH, Weinheim, **1992**; see also A. Müller, C. Beugholt, *Nature* **1996**, *383*, 296–297; b) B. Alberts, D. Bray, J. Lewis, M. Raff, K. Roberts, J. D. Watson, *Molekularbiologie der Zelle*, VCH, Weinheim, **1990**; c) B. D. Davis, R. Dulbecco, H. N. Eisen, H. S. Ginsberg, *Microbiology*, Harper, Philadelphia, **1980**, p. 869; d) "The Structure of Satellite Tobacco Necrosis Virus": L. Liljas, B. Strandberg in *Biological Macromolecules and Assemblies, Vol. 1, Virus Structures* (Eds.: F. A. Jurnak, A. McPherson), Wiley, New York, **1984**, pp. 97–119; e) L. Liljas, T. Unge, T. A. Jones, K. Fridborg, S. Lövgren, U. Skoglund, B. Strandberg, *J. Mol. Biol.* **1982**, *159*, 93–108; f) S. C. Harrison, *Trends Biochem. Sci.* **1984**, 345–351; g) H. G. Schlegel, *Allgemeine Mikrobiologie*, Thieme, Stuttgart, **1992**; h) D. L. D. Caspar, A. Klug, *Cold Spring Harbor Symp. Quant. Biol.* **1962**, *27*, 1–24; i) M. Kemp, *Nature*, 1998, *396*, 123.

[11] This leads to the presence of the fivefold degenerate irreducible representation H_g (F. A. Cotton, *Chemical Applications of Group Theory*, 3rd ed., Wiley, New York, **1990**), for example with the consequence of only a few bands in the IR and Raman spectra.

[12] G. Brinkmann, A. W. M. Dress, *J. Algorithms* **1997**, *23*, 345–358.

[13] R. Nesper, *Angew. Chem.* **1994**, *106*, 891–894; *Angew. Chem. Int. Ed. Engl.* **1994**, *33*, 843–846.

[14] J. Kepler, *Mysterium Cosmographicum* **1596**; see also M. Kemp, *Nature* **1998**, *393*, 123.

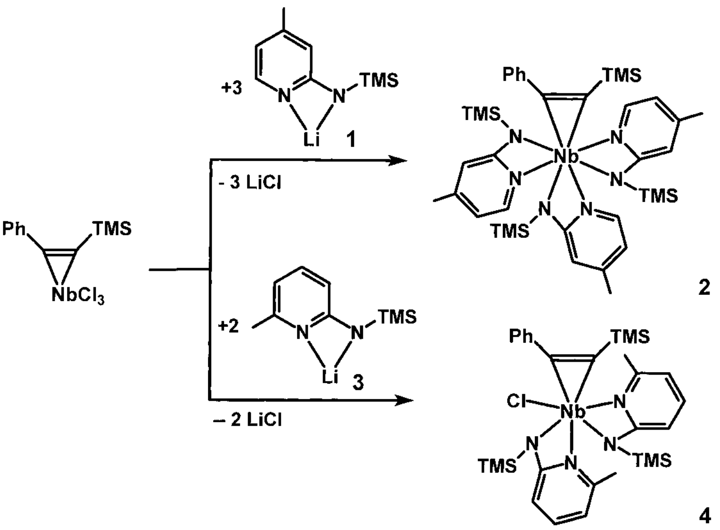
[15] There is also an additional, but marginal relationship to Kepler's conjectures of 1611 that no packing of spheres can be denser than in the face-centered-cubic lattice, observed for instance in crystals of **1**, a conjecture that could only now be confirmed (see N. J. A. Sloane, *Nature* **1998**, *395*, 435–436).

Novel Amidoniobium Complexes with a Functional Relationship to the $[Cp_2ZrR]^+$ Ion*

Anke Spannenberg, Hans Fuhrmann, Perdita Arndt, Wolfgang Baumann, and Rhett Kempe*

Niobium and tantalum compounds live in the shadow of metal complexes of Group 4. Possible reasons for this are the extremely interesting applications of the latter compounds in olefin polymerization[1] or in selective couplings of α-olefins for organic synthesis.[2] Hopes of catalysts with more versatility with regard to the functional groups of the olefins which are to be coupled have recently stimulated increased activity in the search for catalytically active species in Group 5;[3] for analogues of the known $[Cp_2MR]^+$ ions (M = Ti, Zr, Hf). Amido ligands might be useful in this respect.[4] Herein we report the first cationic amidoniobium compound, which can be used for selective coupling of α-olefins.

Treatment of $[NbCl_3(dme)(PhC{\equiv}CTMS)]$[5] (dme = dimethoxyethane, TMS = trimethylsilyl) with **1** (3 equiv) gave the orange-red alkyneniobium complex **2** (Scheme 1). The IR spectrum and the ^{13}C NMR data of **2** indicate that the alkyne ligand functions as a 4e donor.[6] The niobacyclopropene ring in **2** is extremely stable and does not react with ketones, styrene oxide, alkynes, and olefins, as known for the metallocene alkyne complexes of the titanium triad.[7] Complexes with only two amido ligands are accessible by using the sterically more demanding aminopyridinato (Ap) ligands.



Scheme 1. Synthesis of **2** and **4**.

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