ethanol/water (1/2) at 65°C (92%) followed by oxidation with pyridine/SO_3 complex and Et_3N in dimethyl sulfoxide at 10°C (97%).

[9] a) E. J. Corey, F. Xu, M. C. Noe, *J. Am. Chem. Soc.* **1997**, *119*, 12414–12415; b) E. J. Corey, Y. Bo, J. Busch-Petersen, *J. Am. Chem. Soc.* **1998**, *120*, 13000–13001; c) E. J. Corey, M. C. Noe, F. Xu, *Tetrahedron Lett.* **1998**, *39*, 5347–5350.

[10] Quaternary ammonium fluoride **4** was prepared from the corresponding bromide[9] by passage of a methanolic solution through a column of ion-exchange resin Amberlyst A-26 (OH^-) to afford the corresponding quaternary ammonium hydroxide followed by treatment with one equivalent of HF and removal of solvent in vacuo. The quaternary ammonium salt may be separated from the nitro alcohol **5** for reuse by selectively dissolving the latter in diethyl ether.

[11] For diastereoselective nitroaldol reactions catalyzed by rare earth-Li-BINOL complexes, see H. Sasai, W.-S. Kim, T. Suzuki, M. Shibasaki, *Tetrahedron Lett.* **1994**, *35*, 6123–6126.

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[13] The allyl ether quaternary ammonium salt **9** afforded somewhat better stereoselectivity in this reaction than did the benzyl ether analogue **4**. Bromide **9** is converted into the corresponding fluoride in situ.

[14] We are grateful to Dr. Peter Cheng of the Bristol–Myers Squibb Research Institute (Princeton, NJ) for an authentic sample of 2-*epi*-**11**[7b] and spectral data.

A Three-Dimensional Coordination Polymer with an Expanded NbO Structure**

Tianyan Niu, Xiqu Wang, and Allan J. Jacobson*

Coordination chemistry allows a systematic approach to the synthesis of extended lattices.[1] Many structures have been reported with unprecedented lattice types, while others are based on frameworks that are found in simple inorganic structures, for example, diamond,[2] PtS,[3] and quartz.[4] Several coordination polymers have been described[5] containing square-planar centers based on the NbO net ($6^4 8^2$), the tetragonal $CdSO_4$ structure ($6^5 8$, B), and the "dense" net ($7^5 9$, C). An example of an NbO net[6] (Figure 1) is the compound formed by cyanuril acid and biuret containing two interpenetrated hydrogen-bonded networks.[7,8] Examples of the other two structure types are the interpenetrated networks formed by copper(II) nitrate with 1,2-bis(4-pyridyl)ethane (B)[9] and 1,2-bis(4-pyridyl)ethyne (C).[5a] Schindler and Baur[10] have shown that several non-interpenetrated inorganic frameworks can be related to the NbO-type structure.

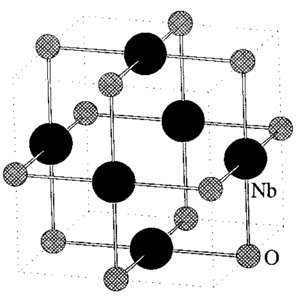

Figure 1. The framework of NbO.

For example sodalite, $[(Me_4N)_{1.3}(H_3O)_{0.7}\{Mo_4O_8(PO_4)_2\}]\cdot 2H_2O$,[11] and $[Cs_3\{V_5O_9(PO_4)_2\}]\cdot xH_2O$[12] frameworks can be constructed by connecting four rings of silicate tetrahedra $Si_4O_4O_{8/2}$, $Mo_4O_8(PO_4)_{4/2}$ cubes, and $V_5O_9(PO_4)_{4/2}$ "helmets", respectively, with 90° rotations between adjacent groups imposed by the shared tetrahedral bridging units.

We and others have investigated the syntheses and properties of coordination polymers formed by linking cyanometalate anions with trialkyl- or triaryltin cations.[13–18] The compounds in this class have the general formula $[(R_3Sn^{IV})_nM(CN)_m]$ and contain polymeric $-M-C{\equiv}N-Sn-N{\equiv}C-M-$ chains which are connected to give frameworks of various topologies. A summary of the transition metals and organic R groups that have been used in the previous studies is given elsewhere.[16] This class of compounds has the potential for use in molecular separations[19] because large cavities are formed in some examples. Intercalation of large molecules, for example, ferrocene in $[(Me_3Sn)_3Fe(CN)_6]$[20] has been reported.

In compounds formed by square-planar $[Ni(CN)_4]^{2-}$ anions and R_3Sn^+ cations in a 1:2 ratio, two possible structure types can be anticipated. The first is a two-dimensional layer structure in which all $Ni(CN)_4$ planes are parallel. The second is a hypothetical framework that can be thought of as being related to the NbO structures discussed above, but expanded by the bridging R_3Sn^+ cations. This would entail alternating parallel and perpendicular $Ni(CN)_4$ planes. Here we report the synthesis of **1**, to our knowledge the first three-dimensional cyanometalate coordination polymer with this expanded NbO-type structure. The framework of **1** is not interpenetrated, and the large central cavity in the structure is filled by inclusion of Ph_3SnOH and solvent molecules during synthesis.

$$[(Ph_3Sn)_2Ni(CN)_4\cdot Ph_3SnOH\cdot{\approx}0.8\,MeCN\cdot{\approx}0.2\,H_2O] \qquad \mathbf{1}$$

Compound **1** was prepared in single-crystal form by slow interdiffusion of solutions of Ph_3SnCl in acetonitrile and $K_2[Ni(CN)_4]$ in water. The structure of **1** was determined by single-crystal X-ray diffraction,[21] and the composition was determined by elemental analysis. The local coordination environments of Ni and Sn atoms are shown in Figure 2. The

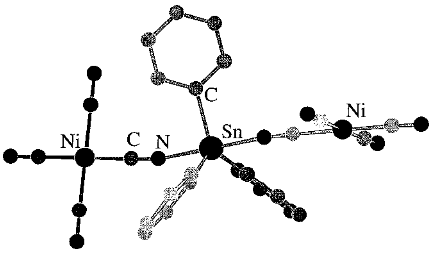

Figure 2. The local coordination environments of the nickel and tin atoms in **1**.

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[*] Prof. A. J. Jacobson, T. Niu, Dr. X. Wang
Department of Chemistry
University of Houston
Houston, TX 77204-5641 (USA)
Fax: (+1)713-743-2787
E-mail: ajjacob@uh.edu

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$Ni(CN)_4$ unit remains square planar with the expected bond lengths for Ni–C and C≡N. Coordination of nitrogen atoms from cyanide groups in adjacent $Ni(CN)_4$ units gives a trigonal-bipyramidal coordination environment for the Sn atoms. The N–Sn distance (2.25 Å) is somewhat shorter than distances observed in similar compounds, for example, 2.37 Å (av) in $[(n\mathrm{Bu}_3\mathrm{Sn})_3\mathrm{M(CN)}_6]$ (M = Fe, Co),[16] 2.34 Å in $[(Me_3Sn)_4Mo(CN)_8]$,[17] and 2.33 Å in $[(Ph_3Sn)_3Fe(CN)_6 \cdot H_2O \cdot 2\,MeCN]$.[18]

The building unit shown in Figure 2 is connected to similar units to form a low-density open framework structure with cubic symmetry $Fd\bar{3}c$ (Figure 3). Each $Ni(CN)_4$ unit is connected through Ph_3Sn bridges to similar units that are rotated by 90° to form an NbO-like arrangement that is expanded by the bridging units (C-N-Sn 169.6°). The phenyl groups in the framework are disordered about a twofold axis. The unit shown in Figure 3 has dimensions of $a/2$ Å on each side. The doubling is a consequence of the relative rotations of adjacent $Ni(CN)_4$ units that are in the same orientation.

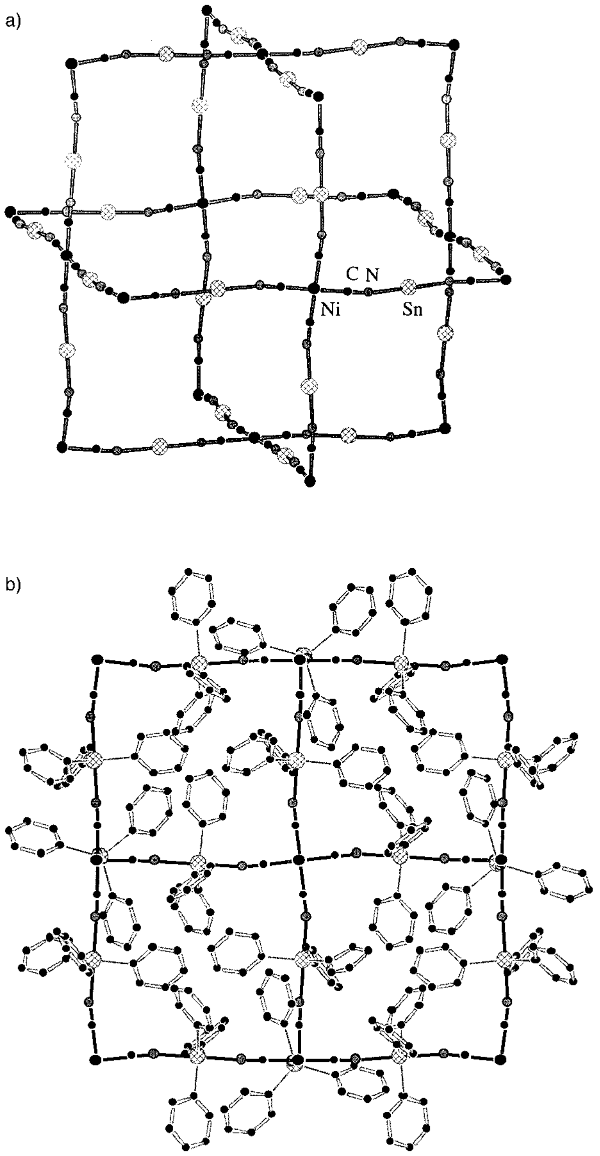

Figure 3. Views of the $(Ph_3Sn)_2Ni(CN)_4$ framework in **1**. a) Phenyl groups and solvent molecules are omitted. b) Solvent molecules are omitted, and only one orientation of each disordered phenyl ring is shown.

A very large cavity is present in the cubic array (see Figure 3). The free diameter of the cavity, allowing for the van der Waals dimensions of the phenyl groups, is about 12 Å, and the volume is thus 1728 $Å^3$. The central cavity in **1**, as synthesized, contains Ph_3SnOH, acetonitrile, and water molecules in a disordered arrangement (Figure 4). The Ph_3SnOH molecule is disordered about a threefold axis. Three Ph_3SnOH molecules, together with water and acetonitrile, are disordered about a tetrahedral site. The occupancy of Ph_3SnOH was determined from elemental analysis to be one molecule per formula unit. The occupancies of the acetonitrile and water molecules were refined with C, N, and O isotropic thermal parameters constrained to be equal. The refined occupancies correspond to about 0.8 MeCN and 0.2 H_2O per formula unit. The presence of water molecules in the structure was confirmed by infrared spectroscopy.

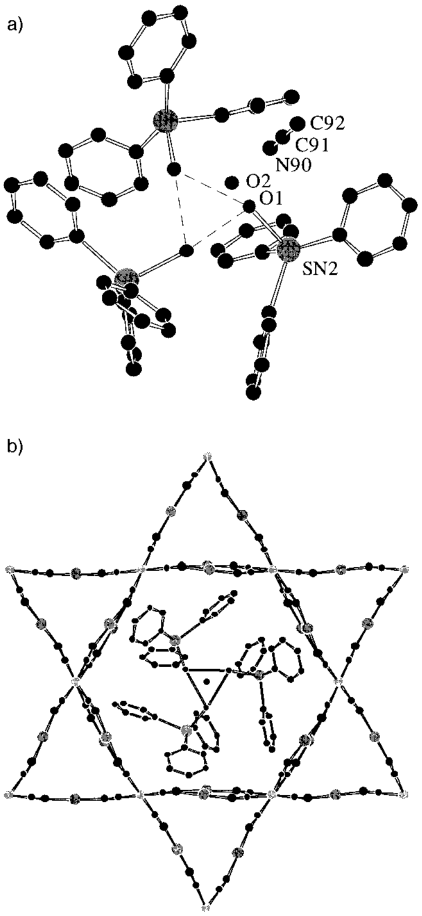

Figure 4. a) Model for the arrangement of the $(Ph_3SnOH)_3$ trimer and the solvent molecules in the central cavity in **1**. b) The arrangement of the guest molecules in the framework, viewed down the (111) direction; phenyl groups on the framework are omitted for clarity.

The infrared spectrum of **1** shows a C≡N stretching band at 2150 cm^{-1} which is blue shifted from the corresponding band for $K_2[Ni(CN)_4]$, as observed in other organotin–cyanometalate compounds.[16,22,23] Thermogravimetric analysis of **1** shows a weight loss beginning immediately on heating due to rapid loss of water and acetonitrile molecules (see Experimental Section). The weight remains constant from 100 °C to about 200 °C, and powder X-ray diffraction data indicate that the structure is maintained. Reabsorption of water/acetonitrile occurred at room temperature for samples that had been heated to 160 °C. Above 200 °C complete decomposition of the framework and loss of the included nonframework molecules occurred in a single step. The residue was shown by powder X-ray diffraction to be a mixture of NiO and SnO_2.

Microcrystalline **1** was also obtained by direct reaction of Ph_3SnCl and $K_2[Ni(CN)_4]$ in water/acetonitrile. The purity of the product was confirmed by comparison of its X-ray powder diffraction pattern with a pattern simulated using the parameters from the single-crystal X-ray structure analysis. Changing the ratio of Ph_3SnCl to $K_2[Ni(CN)_4]$ from 3:1 to 2:1 in the reaction had no effect on composition and purity, indicating that **1** is the stable phase. The microcrystalline form of **1** and the larger crystals formed by diffusion are both stable under ambient conditions.

Compound **1** is the only example reported to date of a three-dimensional framework structure formed by $[Ni(CN)_4]^{2-}$ anions and R_3Sn^+ cations. Two-dimensional layer structures are formed with R groups other than phenyl. For example, our preliminary results show that the compounds $[(R_3Sn)_2Ni(CN)_4]$ (R = ethyl, *n*-butyl) have layered structures. A different layered structure is adopted by $[Ni(CN)_2 \cdot 2Me_3SnCN \cdot (nBu_4N)OH]$.[24] The compounds $[(R_3Sn)_2M(CN)_4]$ (M = Pd, Pt; R = Me, Bu, Ph) have been reported also to have layered structures,[25] but were not completely characterized.

The inclusion of Ph_3SnOH molecules appears to be necessary for the formation of **1**. The elemental composition and X-ray refinement indicate that each cavity contains three Ph_3SnOH molecules in a disordered arrangement. The inclusion of a fourth Ph_3SnOH molecule in the remaining tetrahedral position would lead to a very short O–O distance. Consequently, the remaining space in the cavity is occupied by acetonitrile and water molecules. The arrangement of the three Ph_3SnOH molecules shown in Figure 4 can be stabilized by the formation of hydrogen bonds with O–O atom separations of 2.86 Å. It is significant that **1** is recovered upon use of different reactant ratios, confirming the stability of the $(Ph_3SnOH)_3$ hydrogen-bonded trimer. In contrast, crystalline Ph_3SnOH forms infinite $(Sn\text{-}O\text{-}Sn)_n$ chains with five-coordinate Sn atoms.[26] The inclusion of the hydrogen-bonded $(Ph_3SnOH)_3$ trimer probably accounts for the formation of a three- rather than a two-dimensional framework. The presence of the guest molecules also prevents the formation of an interpenetrated lattice. Further studies are in progress to investigate the effects of other combinations of guest molecules and R_3Sn ligands on the preferential formation of three- rather than two-dimensional frameworks.

Experimental Section

1: $K_2[Ni(CN)_4]$ (0.052 g, 0.216 mmol) was dissolved in distilled water (10 mL) and placed in a 30-mL screw-capped tube. A mixture of water/acetonitrile (1/1, 5 mL) was applied as a buffer layer, and a solution of Ph_3SnCl (0.25 g, 0.648 mmol) in acetonitrile (10 mL) was layered on top. Cubic colorless crystals of **1** were observed at the interface after 10 d of interdiffusion. Elemental analysis calcd: C 56.54, H 3.88, N 5.31, Sn 28.13, Ni 4.64; found: C 56.23, H 3.68, N 4.80, Sn 28.39, Ni 4.61; IR (KBr; Galaxy FTIR 5000): $\tilde{\nu}$ = 3443 (m), 3049 (m), 2150 (s), 1481 (m), 1431 (m), 1076 (m), 997 (m), 729 (m), 694 cm^{-1} (s); TGA (TA Instruments 2100, N_2 atmosphere, 5 °C min^{-1}): 5 % solvent loss, 58.3 % loss upon total decomposition, 36.7 % residue (calcd 37.7 %).

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[21] a) X-ray crystal structure analysis of **1**: colorless cubic crystal of size 0.3 × 0.3 × 0.35 mm, cubic, space group $Fd\bar{3}c$, $a = 41.972(2)$ Å, $V =$

73938(4) Å^3, $Z = 48$, $\rho = 1.365$ g cm^{-3}, $2\theta_{max} = 41.22°$, $\mu = 1.542$ mm^{-1}, $T = 223(2)$ K; Siemens Smart CCD diffractometer; Mo$_{K\alpha}$ radiation ($\lambda = 0.71073$ Å); of 25657 reflections measured, 692 were independent, R(int.) = 0.0323. The structure was solved by direct methods and refined by a full-matrix least-squares procedure against F^2 (hydrogen atoms were not refined) with SHELXTL (Version 5.03, G. M. Sheldrick); $R = 0.0558$, $wR^2 = 0.1547$ for 679 reflections with $I > 4\sigma(I)$, 68 parameters, GOF = 1.164; max./min. residual electron density 0.687/ − 0.501 e Å^3. Semiempirical absorption correction applied. All other F-centered space groups (centro and non-centrosymmetric) were investigated, but none remove any of the disorder. b) Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-112388. 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).

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Templated Synthesis of Vanadium Borophosphate Cluster Anions[**]

Ranko P. Bontchev, Junghwan Do, and
Allan J. Jacobson*

Early transition metals are well known to form complex polyoxoanions with ring and cage structures,[1] including some exceptionally large ring-shaped molybdenum oxide clusters that have recently been reported.[2] Other cluster compounds contain arsenate, organophosphonate, and borate anions in addition to metal oxo species.[3] Several of these cluster anions encapsulate anions,[4] molecules,[5] or cations,[6] and in some cases the size and shape of the cage has been shown to depend on the size of a specific ion present in the synthesis. For example, the Preyssler anion, $[NaP_5W_3O_{110}]^{14-}$, is not formed in the absence of Na$^+$.[7] Similarly, ClO_4^- and N_3^- anions apparently act as templates in the formation of $[HV_{22}O_{54}(ClO_4)]^{6-}$ and $[H_2V_{18}O_{44}(N_3)]^{5-}$.[8]

In the last few years a number of metal borophosphates have been synthesized and structurally characterized.[9] Three new vanadium borophosphates have been described recently: the three-dimensional framework **I**[10] and two compounds containing cluster anions, **II**[11] and **III**.[12] Here we report the

$(H_3NCH_2CH_2NH_3)_2[(VO)_5(H_2O)BO_2(PO_4)_2]\cdot 1.5\,H_2O$ **I**

$(N_2C_6H_{14})_2[VO(PO_3OH)_4(B_3O_3OH)]\cdot 4\,H_2O$ **II**

$(H_3NCH_2CH_2NH_3)_2[Na \supset \{(VO)_2BO_2(PO_3OH)_2\}_5]\cdot 22.5\,H_2O$ **III**

$(C_4H_{12}N_2)_6[(VO)_2BP_2O_{10}]_4\cdot n\,H_2O$ **1** ($n = 2, 6, 14$)

$Na_{14}[Na \supset \{(VO)_2BP_2O_{10}\}_5]\cdot n\,H_2O$ **2**

$A_{17}[A \supset \{(VO)_2BP_2O_{10}\}_6]\cdot n\,H_2O$ **3** (A = NH$_4^+$, K$^+$, Rb$^+$, Cs$^+$)

synthesis and structures of the new vanadium borophosphates **1**, **2**, and **3** containing cluster anions with different ring sizes, and show that the ring size of the anion is determined by the size of the cation used in the synthesis. Compound **III**, which was recently obtained in a low-yield synthesis,[12] contains a cluster anion similar to that found in **2**.

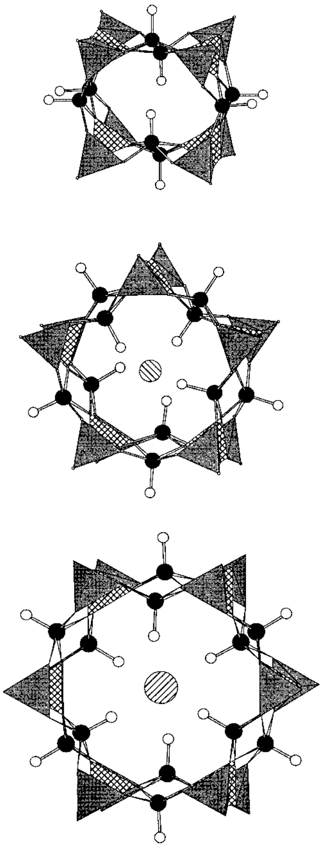

Figure 1. The cyclic cluster anions present in **1** (top), **2** (middle), and **3** (bottom). The PO_4 and BO_4 units are shown as filled and crosshatched tetrahedra, vanadium and oxygen atoms as filled and open circles, and sodium and nitrogen atoms as hatched circles.

Hydrothermal reactions of V_2O_3, H_3BO_3, and the appropriate phosphate salt at 180 °C for three days gave crystals of **1**, **2**, and **3**. The compounds were characterized by single-crystal X-ray diffraction. The structure of **1** ($n = 6$) contains the cluster anion $[(VO)_2BP_2O_{10}]_4^{12-}$ (Figure 1, top).[13] Each tetravalent vanadium atom in the cluster anion is coordinated by five oxygen atoms in a square-pyramidal arrangement with one short V=O distance characteristic of a vanadyl group. Two VO_5 pyramids share a common edge to form V_2O_8 dimers of two distinct types in equal number. In one dimer (V3, V4; Figure 2) the two V=O groups are in a *cis* orientation and point outwards from the center of the cluster. In the other dimer (V1, V2) the V=O groups are in a *trans* orientation. Two oxygen atoms related by a center

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[*] Prof. A. J. Jacobson, Dr. R. P. Bontchev, J. Do
Department of Chemistry
University of Houston
Houston, TX 77204-5641 (USA)
Fax: (+1) 713-743-2787
E-mail: ajjacob@uh.edu

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