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Complex Fluids Based on the Flexible One-Dimensional Mineral Polymers $[K(MPS_4)]_\infty$ (M = Ni, Pd): Autofragmentation to Concave, Cyclic $(PPh_4)_3[(NiPS_4)_3]$**

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In memory of Jean Rouxel

The discovery of the lyotropic nematic phase of $LiMo_3Se_3$ in *N*-methylformamide,[1] a rare example of a liquid crystal based on a mineral core, was achieved 70 years after the pioneering work of the German physicist H. Zocher.[2] This event, soon followed by the study of the nematic behavior of V_2O_5 ribbons[3] and smectic clays[4] in water, has aroused acute interest in the development of solution-phase chemistry of charged, all-inorganic molecules. The motivation for such investigations is the prospect of unraveling the organic/inorganic interfacial chemistry and physics of unprecedented anisotropic fluids based on one- and two-dimensional, charged, extended mineral polymers.[5, 6]

The solid-state chemistry of transition metal chalcogenides[7] is abound with low-dimensional motifs, such as $^1_\infty[Mo_3Se_3]^-$. Their dimensionality arises from the balance between the strong covalent character of the transition metal–chalcogen bonds within the negatively charged chains or slabs and their effective ionic screening by alkali metal cations.[8, 9] A recent example is the series of transition metal chalcogenide phosphates $KMPS_4$ (M = Ni, Pd) with infinite anionic chains $^1_\infty[MPS_4]^-$.[10] Here we demonstrate 1) that $KMPS_4$ compounds are soluble in polar organic solvents such as dimethylformamide (DMF) and dimethyl sulfoxide (DMSO), and form complex fluids made up of flexible inorganic polymers; 2) that $[(NiPS_4)_3]^{3-}$—an unprecedented, concave trimetallic thiophosphate molecular trianion adopting pseudo-C_{3v} symmetry—is formed in DMF at room temperature from the autofragmentation and rearrangement of $^1_\infty[NiPS_4]^-$; and 3) that it is possible to follow the dissolution and fragmentation processes with mass spectrometry, solution-state ^{31}P NMR spectroscopy, and transmission electron microscopy (TEM). Thus, the contrasting behavior and stability of the Ni and Pd phases can be revealed.

In accordance with their highly anisotropic bonding, $KNiPS_4$ and $KPdPS_4$ are soluble in polar organic solvents

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[**] This work was supported by the CNRS. L.M.B. wishes to thank the Région Pays de Loire and the CNRS for a postdoctoral fellowship and a visiting scientist grant, respectively.

  1433-7851/98/3712-1711 $ 17.50+.50/0

such as DMF and DMSO, and rapidly form highly viscous gellike solutions at high concentrations ($[KMPS_4] > 10^{-2}\,mol\,L^{-1}$).[11] However, there is a striking difference in the stabilities of these solutions. The $KPdPS_4$ solution is stable with respect to time and temperature (up to about 323 K), whereas the solution of $KNiPS_4$ changes in color from dark brown to orange-brown, and progressively becomes more fluid; it approaches the solvent fluidity within a few hours at 323 K.

Differences between the $KNiPS_4$ and $KPdPS_4$ solutions can also be observed by optical microscopy under polarized light. Crystals of the solid compounds were sandwiched between a microscope slide and a cover glass, and pure DMF was added, which diffused between the two slides. For both compounds the crystals immediately swelled and rapidly dissolved when they came into contact with the solvent. The birefringence observed in the surrounding solution decreased as the distance from the crystal increased.[12] When the crystals had totally dissolved, the birefringence slowly disappeared, and within a few minutes at room temperature the solutions appeared isotropic. If these solutions were then sheared, for example by applying pressure to the top microscope slide, a strong transient birefringence was immediately observed again (Figure 1).[13] However, as the solution of $KNiPS_4$ aged

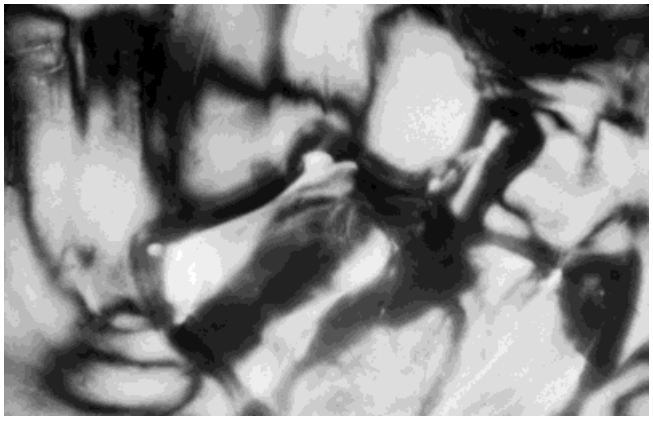

Figure 1. Transient birefringence (reminiscent of nematic threaded textures) observed by optical microscopy under polarized light (polarizers crossed) from a sheared concentrated gel of $KPdPS_4$ in DMF.

and became more fluid, the intensity of this transient birefringence decreased to zero. This is in stark contrast to the unlimited reproducibility of the transient birefringence of $KPdPS_4$ at room temperature. Such flow birefringence is commonly observed for solutions of lyotropic mesophases at concentrations lower than the isotropic/anisotropic transition concentration and is proof of transient anisotropy in the fluid.[14] This behavior can arise from either a preferred orientation of anisotropic rigid molecules induced by flow (analogous to logs aligned with the flow of a river), or from stretching of interdigitated, folded, flexible polymers (like drawn spaghetti). Both models imply the persistence of $^1_\infty[MPS_4]^-$ chains in solution.

Transmission electron microscopy of $KPdPS_4$ obtained after evaporation of the solvent (Figure 2)[15] confirms that the chains remain intact in solution. The separation between the

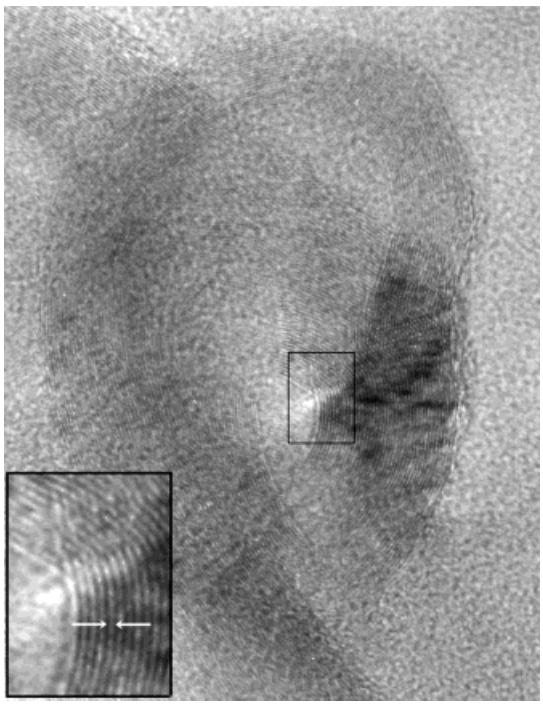

Figure 2. TEM micrograph of the chains in $KPdPS_4$. The sample was prepared by directly depositing one drop of a solution in DMF ($[KPdPS_4] = 10^{-6}\,mol\,L^{-1}$) onto a carbon grid and drying overnight. The distance between the arrows is 3.5(1) Å, which indicates a hexagonal stacking of the chains.

chains, indicated in Figure 2 by the arrows, is 3.5(1) Å, which is consistent with a hexagonal packing of chains that are 7.0 Å apart. This distance is approximately equal to the calculated van der Waals radius of a PdS_4 square-planar unit and is slightly greater than the size of a tetrahedral PS_4 group. Another striking feature of the micrograph is that the chains appear to be highly flexible. A curvature radius as low as 50 Å was measured, which supports the spaghettilike model. X-ray experiments are in progress to determine the coherence length of the chains.

Recrystallization of the original $KMPS_4$ compounds was not possible from the initial solutions under various conditions; only amorphous compounds were obtained. However, again, there is a difference in the behaviors of aged and freshly prepared solutions of $KNiPS_4$/DMF: A crystalline compound can be isolated from the aged solution upon replacing K^+ by PPh_4^+ through a metathesis reaction. The structure of the corresponding phase[16] $(PPh_4)_3[Ni_3P_3S_{12}]$ discloses the unprecedented inorganic anionic ring $[Ni_3P_3S_{12}]^{3-}$ (Figure 3). In light of these observations, the loss of transient birefringence in aged solutions of $KNiPS_4$/DMF can be understood in terms of autofragmentation of the $^1_\infty[NiPS_4]^-$ chains to form $[Ni_3P_3S_{12}]^{3-}$ cycles. This would also account for the inability to recrystallize freshly prepared samples, as the solutions predominately consist of tangled chains, which cannot be induced to arrange in an ordered fashion necessary for crystallization. This is analogous to the behavior of an organic polymer.

We followed the evolution of these solutions with ^{31}P NMR spectroscopy and mass spectrometry. The mass spectrum of

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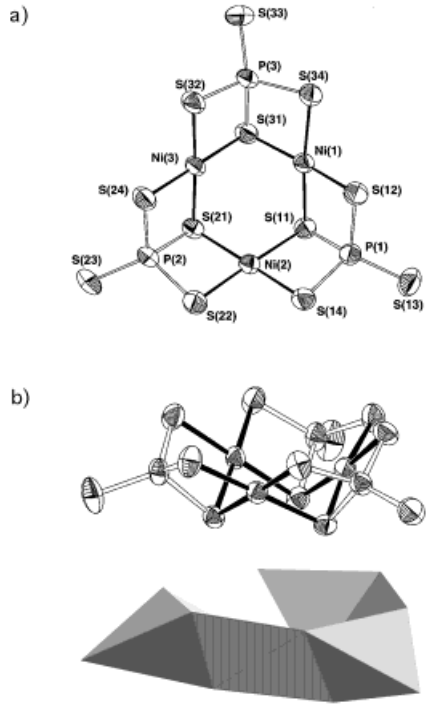

Figure 3. The concave $[(NiPS_4)_3]^{3-}$ cyclic anion: a) ORTEP representation showing the pseudo-C_{3v} symmetry adopted in the solid state; b) ORTEP (top) and polyhedra representation (bottom) exemplifying the bowllike configuration. The Ni–S bond lengths are homogeneous (av 2.217(10) Å). There are three types of P–S bond lengths: P–(μ_3-S) 2.112(5), P–(μ_2-S) 2.046(8), P–(μ_1-S) 1.949(7) Å. A Mulliken analysis based on extended-Hückel calculations indicates that the negative charge is essentially located on the μ_1-S ligands at the periphery of the anion.

$KPdPS_4$/DMF consists of a broad peak with some superimposed sharp peaks. The extremely broad background is characteristic of a polydispersed distribution of chain lengths in solution. The peaks of low intensity can be attributed to a small concentration of $[Pd_3P_3S_{12}]^{3-}$ anions with different types and numbers of charge-compensating cations (H^+ or K^+) and/or solvent molecules. The solution-state ^{31}P NMR spectrum of $KPdPS_4$ remains unchanged even after the sample has been heated at 323 K for several months; a single peak is observed at $\delta = 146.4$, which we assign to the $^1_\infty[PdPS_4]^-$ chain by comparison with the solid-state ^{31}P NMR chemical shift of $KPdPS_4$. Since there is no evidence for the fragmentation of the Pd chains at this temperature (decomposition occurs above 368 K), we believe that the $[Pd_3P_3S_{12}]^{3-}$, $[(Pd_4P_4S_{16})K_3]^-$, and $[PdPS_4]^-$ anions observed in small concentrations in the mass spectrum are produced in situ upon vaporization of the sample. In contrast, the mass spectrum of aged $KNiPS_4$/DMF shows only a series of sharp peaks, of which the three of greatest intensity can be assigned to $[Ni_3P_3S_{12}]^{3-}$ with different types and numbers of cations. This formula is consistent with that of the cyclic anion described above. Other species formed in smaller quantities are tentatively assigned as larger anions of the type $[NiPS_4]^{n-}_n$ ($n = 4-6$) and as smaller anions. Again, the latter are probably produced in situ upon vaporization of the sample, as no evidence for their existence was observed by ^{31}P NMR spectroscopy. The changes at 300 K in the solution-state ^{31}P NMR spectrum of $KNiPS_4$ in $[D_7]$DMF, which was previously dissolved by rigorous stirring at 263 K in order to avoid fragmentation, is shown in Figure 4. Initially, a peak at $\delta = 122$ is observed[17] which decreases in intensity with time while

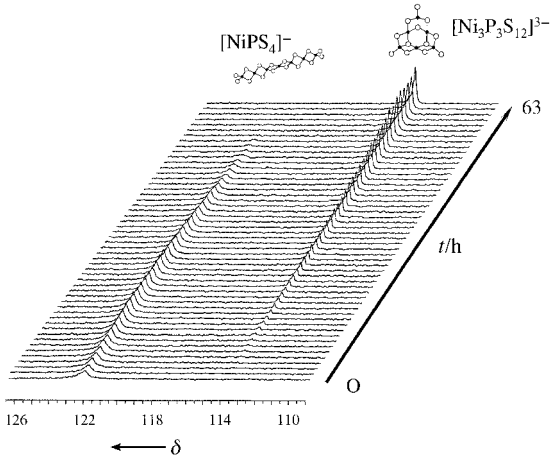

Figure 4. Changes with time t in the solution-state ^{31}P NMR spectrum of $KNiPS_4$ in $[D_7]$DMF at 300 K. The chain undergoes autofragmentation and rearrangement to form the cyclic anion.

another peak appears and grows at $\delta = 114$. After 63 h the peak at higher frequency has completely disappeared, and the intensity of the other peak remains constant. Based on the solid-state ^{31}P NMR shifts of $KNiPS_4$ and $(PPh_4)_3[Ni_3P_3S_{12}]$ we assign the peaks at $\delta = 122$ and 114 to the $^1_\infty[NiPS_4]^-$ chain and the cyclic anion $[Ni_3P_3S_{12}]^{3-}$, respectively. This data confirms that $^1_\infty[NiPS_4]^-$ initially exists in solution, and that the infinite mineral polymer autofragments to form the cyclic structure. The rate of fragmentation of $KNiPS_4$ in DMF is highly dependent on temperature. Upon raising the temperature by just 20 K to 320 K, the conversion of the chain into the cyclic anion is complete after only 12 h.

Why the $[Ni_3P_3S_{12}]^{3-}$ anion is favored over any other anion is an interesting question. To our knowledge, cyclic anions of this type have never been reported to form in solid-state reactions. Finally, it appears that the remarkable dispersion/autofragmentation/rearrangement sequence disclosed here extends the concept of mere excision in solution of monomeric molecular motifs from low-dimensional inorganic solids.[18]

Experimental Section

$KNiPS_4$[10] (2 g) was dissolved in DMF (50 mL, 1.56×10^{-1} mol L^{-1}); complete dissolution was obtained after stirring the solution for a few hours. The cation was exchanged by adding Ph_4PBr (2 g, 4.77 mmol, ca. 20% excess) to an aged (2 d at ambient temperature) solution of $KNiPS_4$ in DMF. This mixture was stirred for 30 min, and then Et_2O (500 mL) was added slowly. A brown precipitate formed, which was isolated by filtration and recrystallized from a minimum of CH_3CN (yield 75%); elemental analysis calcd (found) for $C_{24}H_{20}P_2NiS_4$: C 47.64 (47.95), H 3.33 (3.71).

The mass spectra were collected with a VG BioTech spectrometer equipped with a Quatro II triple quadrupole detector. The solutions of $KNiPS_4$ and $KPdPS_4$ in DMF were heated at 323 K for 5 d before being analyzed.

Samples were prepared for solution-state ^{31}P NMR spectroscopy by vigorous stirring of the compounds in $[D_7]$DMF for 18 h at 263 K. Spectra

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were collected on a Bruker AM200 spectrometer operating at a frequency of 81 MHz, and were recorded with a $\approx 20^\circ$ pulse length of 2 μs and a recycle time of 3 – 5 s. No proton decoupling was used, and so the relative intensities of the signals are quantitative. Spectra were referenced to phosphoric acid as the standard ($\delta = 0$). The data were processed with the PC version of WINNMR (Bruker).

Received: March 12, 1998 [Z11580IE]
German version: *Angew. Chem.* **1998**, *110*, 1773 – 1776

Keywords: liquid crystals · metathesis · nickel · polymers · thiophosphates

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[11] $KMPS_4$ compounds are soluble in DMF at all concentrations. Highly viscous gels (resembling modelling clay) have been obtained at concentrations as high as 2.2 mol L^{-1} ($KMPS_4 \cdot 6$ DMF).
[12] The difference between the crystal and the solution can be readily determined by uncrossing the polarizers, which reveals a crystal surrounded by a particle-free phase that flows when compressed; this confirms its fluid nature.
[13] Relaxation times are on the order of about 1 s at 10^{-2} mol L^{-1} and about 15 min at 1.2 mol L^{-1} ($KMPS_4 \cdot 11$ DMF).
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[15] Electron micrographs were obtained with a CM30T Philips microscope (LaB_6 cathode) operating at 300 kV. The spherical aberration was 2 mm, and the point resolution was 2.3 Å.
[16] Crystal data for $(PPh_4)_3[Ni_3P_3S_{12}]$: monoclinic, space group $P2_1/c$, $a = 15.828(2)$, $b = 10.099(1)$, $c = 47.112(6)$ Å, $\beta = 91.11(1)^\circ$, 6862 independent reflections with $I \geq 3\sigma(I)$, $RF = 0.0624$, $R_wF = 0.0848$, min./max. residual electron density: $-0.61/0.76$ e Å^{-3}, $S = 1.88$. The data were collected on a Siemens CCD single diffractometer with an automated three-axis goniometer; $Mo_{K\text{-}L2,3}$ radiation was used. The data collected at room temperature were reduced to net intensities, and estimated standard deviations were calculated on the basis of counting statistics; Lorentz polarization corrections were applied, and equivalent reflections were averaged. The structures were solved by direct methods with SHELXTL (G. M. Sheldrick, SHELXTL version 5, Siemens Analytical X-Ray Instruments, Inc. Madison, WI) and refined with JANA98 (V. Petøièek, M. Dušek, JANA98 Crystallographic Computing System, Institute of Physics, Academy of Sciences of the Czeck Republic, Prague, **1998**). Final refinements were performed on F_0 with anisotropic atomic displacement parameters for Ni, P, and S atoms and isotropic parameters for C atoms. The positions of the H atoms were introduced in the structure factors calculations with the same isotropic atomic displacement as the C atoms on which they ride. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-101227. 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).
[17] As only one peak is observed for the chain, we assume that either there is no difference in the chemical shifts of phosphorus atoms in the middle and at the ends of a chain, or that the chains are so long that the intensities of the signals for phosphorus atoms at the ends are negligible with respect to those phosphorous atoms in the central part. The latter is more probable based on the observation from electron microscopy. No other peaks, apart from those from the chain and the cyclic structure, are observed during the in situ NMR study. If we assume that the chemical shift is different for the atoms at the ends and the middle of a chain, then the breaking of the chains must be the rate-limiting step for the cyclization, and hence the concentration of the chain fragments in solution is never high enough to be observed.
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Organometallic Triskelia: Novel Tris(vinylideneruthenium(II)), Tris(alkynylruthenium(II)), and Triruthenium – Triferrocenyl Complexes**

Mitsunari Uno* and Pierre H. Dixneuf*

Dedicated to Professor Warren Roper on the occasion of his 60th birthday

Multitopic carbon-rich metal complexes, with rigid conjugated branches are potentially useful for building carbon-rich networks,[1] supramolecular polymetallic assemblies,[2] and nanoarchitectures for material science.[3] They have found application as the core of metal-containing dendrimers[4, 5] and polymers,[6] and as the basis of liquid crystals.[7] Polymetallic complexes containing multiple identical redox systems are of special interest for electron storage[5, 8] and as modified electrodes[9] due to their ability to provide several electrons at the same potential.

We now report the novel polymetallic complexes containing reversible redox systems, and displaying a C_3-symmetric triskelion shape,[10] formed by activation of the tritopic polyyne 1,3,5-$(HC{\equiv}CC_6H_4C{\equiv}C)_3C_6H_3$ (**1**) namely the first examples of tris(vinylideneruthenium(II)) complexes (**2**),

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[**] This work was supported by Osaka University and CNRS especially through an associate researcher position to Dr. M. Uno.

  1433-7851/98/3712-1714 $ 17.50+.50/0