Acknowledgment. We would like to thank IBM Corp. and National Science Foundation for financial support of this work and Dr. Sahyun at 3 M for a helpful discussion. Joe Kuczynski would like to thank Dr. S. P. Pappas for the synthetic procedures for (3-(9-anthracenyl)propyl)diphenylsulfonium hexafluoroantimonate.

Registry No. Ph_3SSbF_6, 57840-38-7; Ph-An(H^+), 62726-22-1; (3-(9-anthracenyl)propyl)diphenylsulfonium, 132399-87-2; sodium bromocresol green, 67763-24-0; pyrene, 129-00-0; anthracene, 120-12-7; (3-(9-anthracenyl)propyl)diphenylsulfonium hexafluoroantimonate, 132399-88-3.

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Hydrothermal Synthesis of K_2PdSe_{10}. Coexistence of Two Large Interpenetrating Three-Dimensional Frameworks of $[Pd(Se_4)_2]^{2-}$ and $[Pd(Se_6)_2]^{2-}$

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Abstract: The hydrothermal reaction of $PdCl_2$ with K_2Se_4 in the presence of different KOH and water in the 1:5:5:40 ratio produced $K_2[PdSe_{10}]$ (I), the first metal polychalcogenide compound containing two interpenetrating three-dimensional frameworks. The black rectangular chunky crystals of I are insoluble in water and most organic solvents. They crystallize in the orthorhombic space group $I2_12_12_1$ (no. 24) with a = 15.872 (9) Å, b = 15.922 (9) Å, c = 12.885 (8) Å, V = 3256 (6) Å^3. I is composed of two independent interpenetrating 3D diamond-like frameworks of $[Pd(Se_4)_2]^{2-}$ and $[Pd(Se_6)_2]^{2-}$. Each framework contains large tunnels running parallel to the a- and b-crystallographic axes. The coordination geometry around Pd is square-planar. The average Pd-Se and Se-Se distances are 2.462 (5) and 2.35 (2) Å, respectively. The closest distances between K^+ ions and Se atoms are in the range of 3.27 (2) and 3.75 (2) Å.

Introduction

Recently, we showed that hydrothermal synthesis can be applied to the preparation of some novel Mo polychalcogenide compounds.[1] In order to explore the generality of the technique for other metals, we turned our attention to the more chalcophilic late transition elements. The reactions of such elements with Q_x^{2-} ligands have been explored to some extent at ambient temperature and pressure and were found to produce a number of interesting *molecular* compounds.[2] In the case of the cation/M/Q (M = Cu, Ag; Q = S, Se) and cation/Hg/Te systems it was found that some cations stabilize *polymeric* one-dimensional structures such as those of $(Ph_4P)[Ag(Se_4)]$,[3] $(Me_4N)[Ag(Q_5)]$[4] (Q = S[4], Se[3]), $(H_3NCH_2CH_2NH_3)[Cu(S_5)]_2$,[5] and $(Ph_4P)_2[Hg_2Te_5]$[6]. This raises possibilities for other interesting polychalcogenide-based polymeric structures containing various metals. A particular set of counterions from which it is difficult to obtain pure crystalline products from solution are the alkali metals. Furthermore, based on a significant body of experimental evidence the importance of counterion in determining the structure of the $[MQ_x]^{n-}$ anion has now been recognized.[2,3] For example, we have shown that minor modifications in the size of the counterion (R_4N^+, Ph_4P^+, K^+, Cs^+) can cause major geometric and electronic reorganization of the anionic structure of silver and gold polyselenide compounds.[3] This motivated us to explore new counterions (e.g., alkali ions) in search of new $[MQ_x]^{n-}$ structure types, particularly polymeric. Since we often encounter difficulties in isolating crystalline polymeric compounds using ambient temperature and pressure conditions, we resorted to the hydrothermal technique. The palladium chemistry seemed particularly attractive for such exploration because its first polysulfide compound $(NH_4)_2[PdS_{11}]$[7] was reported to have a polymeric layered structure with S_5^{2-} and S_6^{2-} ligands. The corresponding polyselenide chemistry is unknown, and based on previous experience we expect it to be different. Herein we report the hydrothermal synthesis and structural characterization of $K_2[PdSe_{10}]$ (I), a compound with an intriguing three-dimensional (3D) architecture composed of two unprecedented interpenetrating frameworks. The extended structure of I is rationalized on the basis of anion-cation packing interactions.

Experimental Section

All work was done under a nitrogen atmosphere. $PdCl_2$ was purchased from Alpha Products Inc. Potassium tetraselenide K_2Se_4 was prepared in liquid ammonia from potassium metal and elemental selenium in a 2:4 ratio as described previously.[3] The X-ray powder diffraction patterns were recorded with a Phillips XRG-3000 computer controlled powder

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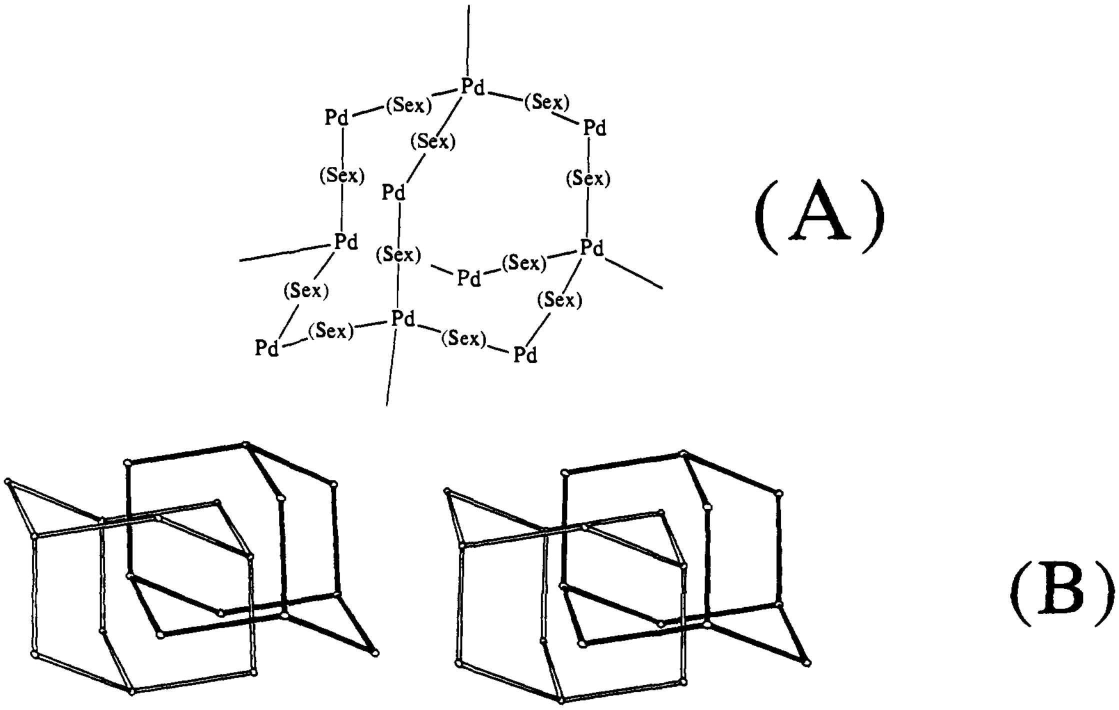

Figure 1. (A) Schematic representation of a single 3D framework of $[Pd(Se_x)_2]^{2-}$. The tetrahedral depiction of Pd atom signifies the disposition and connectivity pattern of the Se_x^{2-} ligands, not their actual coordination geometry. (B) Schematic view of the interpenetrating behavior (stereoview) of $[Pd(Se_4)_2]^{2-}$ and $[Pd(Se_6)_2]^{2-}$ frameworks. This model is topologically equivalent to the $Cd(CN)_2$ structure in which the Se_x^{2-} chains occupy the CN sites and the Pd atoms the Cd sites.

Table I. Data for Crystal Structure Analysis[a] of $K_2[PdSe_{10}]$

formula	$K_2[PdSe_{10}]$
fw	974.20
a, Å	15.872 (9)
b, Å	15.922 (9)
c, Å	12.885 (8)
V, Å^3, Z	3256, 8
space group	$I2_12_12_1$
d_{calcd}, g/cm^3	3.97
μ (Mo Kα), cm^{-1}	237
2θ range, deg	3-45
data collected	1976
no. of unique data	1845
data used	881 ($F_o^2 > 3\sigma(F_o^2)$)
min, max abs. correction	0.99
no. of variables	119
no. of atoms per asym unit	14
final R/R_w (%)	0.065/0.078

[a] At 23 °C, $R = \sum(|F_o| - |F_c|)/\sum|F_o|$. $R_w = \{\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2\}^{1/2}$.

diffractometer operating at 35kV/35mA. Ni filtered, Cu radiation was used. Elemental analyses were performed on a JEOL JSM-35C scanning electron microscope (SEM) equipped with an Energy Dispersive Spectroscopy (EDS) detector.

Synthesis of $K_2[PdSe_{10}]$ (I). $PdCl_2$ (0.050 g, 0.28 mmol), 0.552 g (1.40 mmol) of K_2Se_4, 0.079 g (1.41 mmol) of KOH, and 0.2 mL distilled water were charged in a Pyrex tube. The tube was sealed off under vacuum and heated at 110 °C for a day. The shiny black rectangular chunky crystals (70% yield) were isolated by filtration and washed with a copious amount of water. These crystals are insoluble in water and most organic solvents.

In order to ensure product homogeneity, the X-ray powder diffraction (XRD) diagram of the product was compared with, and found to be identical with that calculated from the single-crystal data. The XRD powder pattern was calculated using the atom coordinates determined from the single crystal data using the program POWD 10.[8] A comparison table between the calculated and observed d_{hkl} spacings is deposited with the supplementary material.

X-ray Crystallographic Studies. The crystallographic data were collected on a Rigaku AFC6S diffractometer using a $\omega/2\theta$ scan mode and

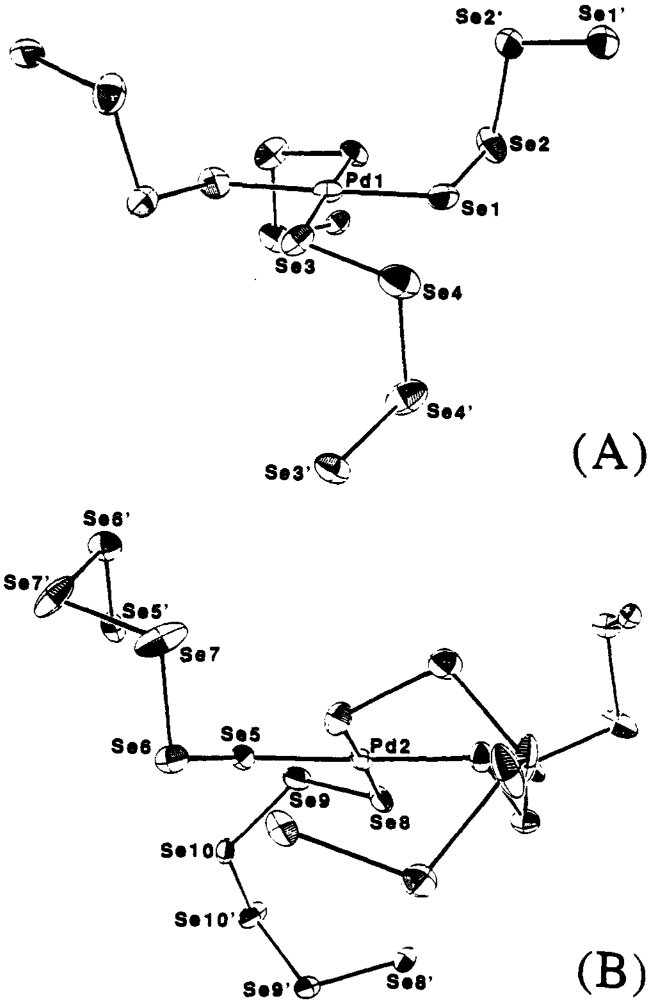

Figure 2. (A) ORTEP representation and labeling scheme of the repeating unit in $[Pd(Se_4)_2]^{2-}$ and (B) ORTEP representation and labeling scheme of the repeating unit in $[Pd(Se_6)_2]^{2-}$.

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[8] Smith, D. K.; Nichols, M. C.; Zolensky, M. E. *POWD 10: A FORTRAN Program for Calculating X-ray Powder Diffraction Patterns*; Version 10, Pennsylvania State University, 1983.

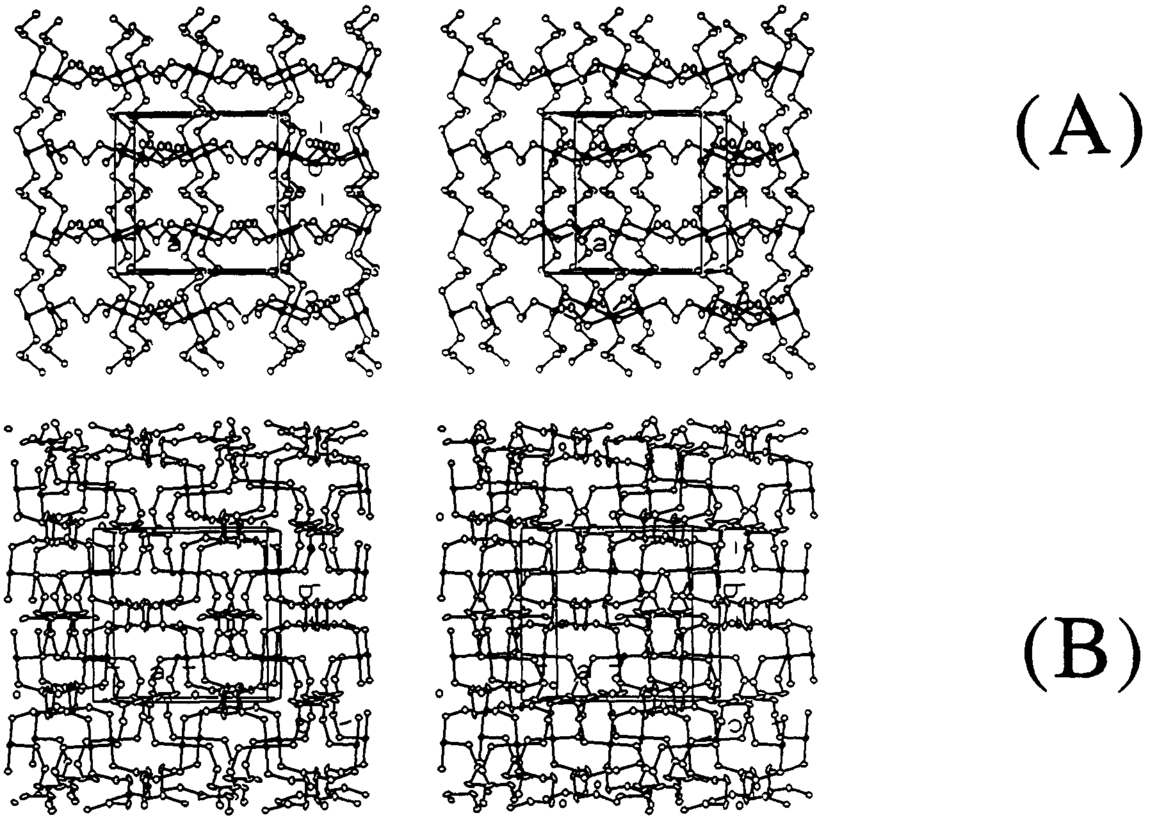

Figure 3. Stereoview of the individual substructures of (A) $[Pd(Se_4)_2]^{2-}$ looking down the *c*-axis and (B) $[Pd(Se_6)_2]^{2-}$ looking down the *c*-axis. Filled circles represent Pd atoms.

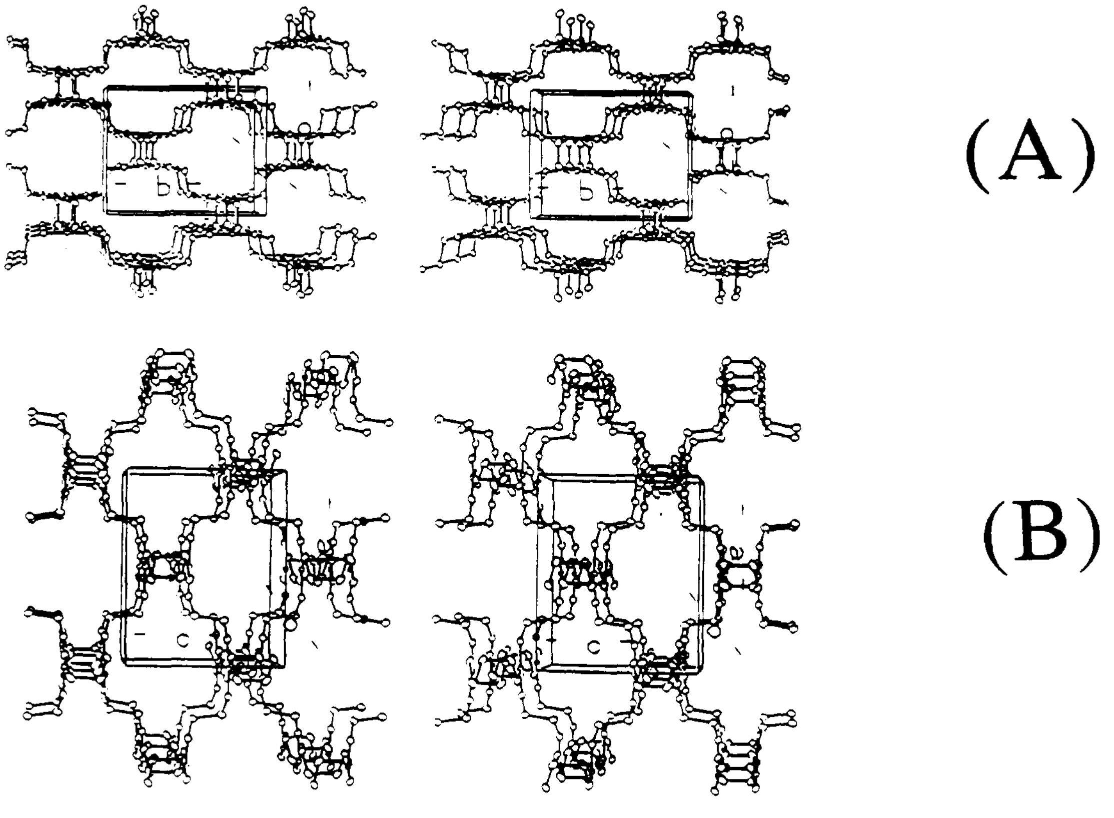

Figure 4. Stereoview of the $[Pd(Se_4)_2]^{2-}$ substructure looking down (A) the *a*-axis and (B) the *b*-axis, illustrating the large one-dimensional channels.

Mo Kα radiation. The crystal was mounted on the tip of a glass fiber. Crystal data and details for intensity data collection and refinement are shown in Table I. The intensities of three check reflections were monitored every 150 reflections. No decay was observed during the data collection period. The data were collected using 4 deg/min scan speed at 23 °C. An empirical absorption correction based on ψ scans for several reflections was applied to all data. An additional absorption correction following the DIFABS[9] procedure was applied to isotropically refined data. Due to the close similarity of the *a*- and *b*-axes we looked carefully for possible tetragonal symmetry without success.

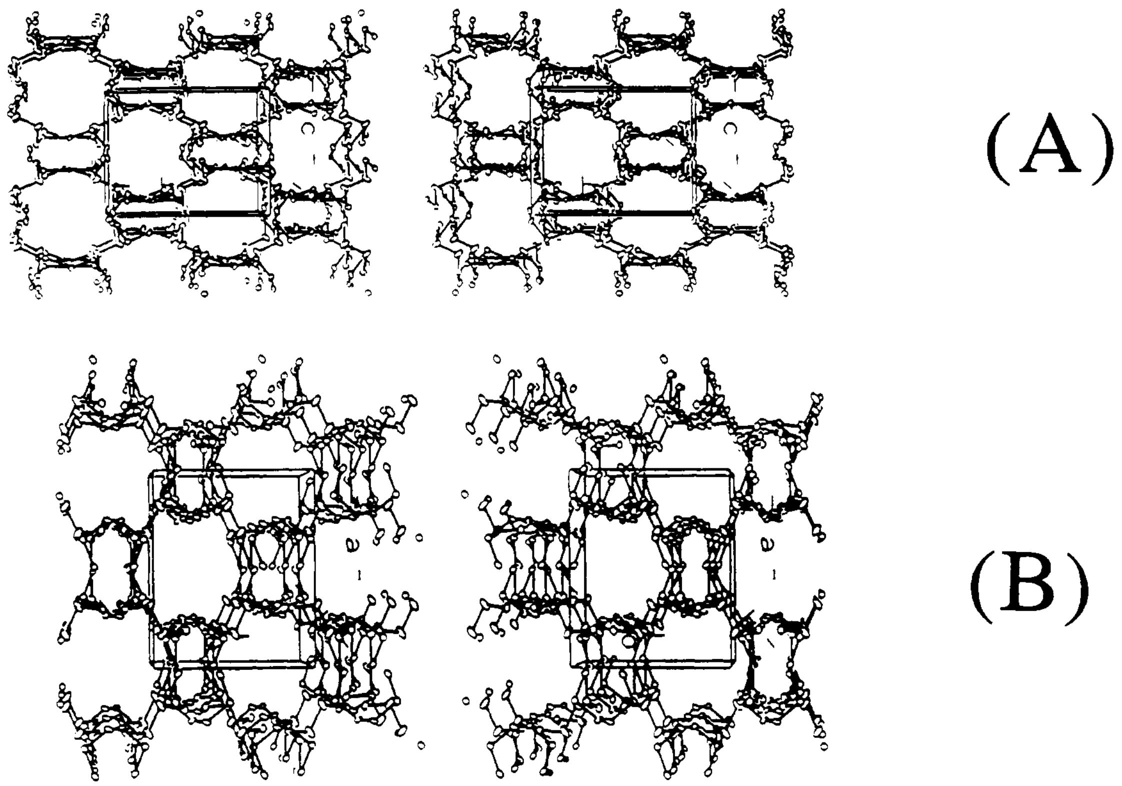

Figure 5. Stereoview of the $[Pd(Se_6)_2]^{2-}$ substructure looking down (A) the *a*-axis and (B) the *b*-axis, illustrating the large one-dimensional channels.

Structure Solution. The structure was solved with direct methods using SHELXS-86[10] and was refined with the TEXSAN package of crystallographic programs[11] running on a VAXstation 3100/76 computer. No disorder was found in the unit cell of I. All atoms were refined anisotropically. The final R and R_w were 6.5% and 7.8%, respectively. The enantiomorph was also refined to completion but gave slightly higher R values (~0.3%) and estimated standard deviations (esds). There were no significant residual peaks in the final electron density difference map. The final coordinates and thermal parameters and their esds of all atoms are deposited in the supplementary material.

Results and Discussion

$K_2[PdSe_{10}]$ is the first metal polychalcogenide compound featuring interpenetrating frameworks. The spectacular structure of $K_2[PdSe_{10}]$ is composed of K^+ ions and two remarkable macroanions of $[Pd(Se_4)_2]^{2-}$ (**Ia**) and $[Pd(Se_6)_2]^{2-}$ (**Ib**). Therefore, $K_4[Pd(Se_4)_2][Pd(Se_6)_2]$ is a more descriptive formulation. Both **Ia** and **Ib** resemble diamond-like frameworks having the intriguing property of existing one inside the other. Polyselenide ligands Se_4^{2-} and Se_6^{2-} serve as long bridges between square-planar Pd metal centers. As shown in Figure 1A, the individual $[Pd(Se_x)_2]^{2-}$ is topologically equivalent to the structure of cristobalite SiO_2 (Pd^{2+} atoms occupy the Si sites and Se_x^{2-} ligands occupy the O sites). It is also interesting to note that the square-planar Pd atoms in a single framework are arranged in space in the same way as carbon atoms in diamond. The average distance between Pd metal centers in both frameworks is 8.577 (6) Å. The interpenetrating behavior of **Ia** and **Ib** is shown in Figure 1B. In this architecture the $PdSe_4$ square planes from one framework stack directly above and below those of the other, spaced 6.44 Å apart along the *c*-axis. There are no covalent or ionic bonds between the two frameworks, and thus one can never cross, through bonds, from one to the other. The shortest contact between frameworks is van der Waals type at 3.45 (1) Å, between Se(4) and Se(9). The angles around the Pd atoms are 90°. The average Pd–Se and Se–Se distances are 2.462 (5) and 2.35 (2) Å, respectively. The closest distances between K^+ ions and Se atoms are in the range of 3.27 (2) and 3.75 (2) Å. Selected distances and angles are given in Table II. The repeating units in **Ia** and **Ib** are shown in Figure 2. Since it is not easy to verbally convey the proper impression of the gordian crystal arrangement of the Se_x^{2-} chains and Pd atoms in I we show two stereoviews of each lattice separately in Figure 3(A,B). A clear view of the entire crystal structure was not possible. The diamondoid arrangement of metal centers and polyselenide ligands in I explains the lack of center of symmetry in the structure.

Table II. Selected Bond Distances (Å) and Angles (deg) for the $[Pd(Se_4)_2]^{2-}$ and $[Pd(Se_6)_2]^{2-}$ Substructures

	$[Pd(Se_4)_2]^{2-}$		
Pd(1)–Se(1)	2.468 (8)	Se(2)–Se(2′)	2.34 (2)
Pd(1)–Se(3)	2.461 (9)	Se(3)–Se(4)	2.32 (1)
Se(1)–Se(2)	2.33 (1)	Se(4)–Se(4′)	2.38 (2)
Se(1)–Pd(1)–Se(1′)	178.3 (6)	Pd(1)–Se(1)–Se(2)	108.6 (4)
Se(1)–Pd(1)–Se(3)	90.0 (3)	Se(1)–Se(2)–Se(2′)	106.3 (4)
Se(1)–Pd(1)–Se(3′)	90.0 (3)	Pd(1)–Se(3)–Se(4)	108.8 (4)
Se(3)–Pd(1)–Se(3′)	177.9 (6)		
	$[Pd(Se_6)_2]^{2-}$		
Pd(2)–Se(5)	2.458 (7)	Se(7)–Se(7′)	2.36 (2)
Pd(2)–Se(8)	2.459 (8)	Se(8)–Se(9)	2.37 (1)
Se(5)–Se(6)	2.33 (1)	Se(9)–Se(10)	2.37 (1)
Se(6)–Se(7)	2.36 (1)	Se(10)–Se(10′)	2.37 (2)
Se(5)–Pd(2)–Se(5′)	177.0 (5)	Se(6)–Se(7)–Se(7′)	104.1 (5)
Se(5)–Pd(2)–Se(8)	89.3 (3)	Se(3)–Se(4)–Se(4′)	105.7 (4)
Se(5)–Pd(2)–Se(8′)	90.8 (3)	Pd(2)–Se(8)–Se(9)	108.7 (4)
Se(8)–Pd(2)–Se(8′)	177.4 (6)	Se(8)–Se(9)–Se(10)	106.2 (4)
Pd(2)–Se(5)–Se(6)	109.3 (4)	Se(9)–Se(10)–Se(10′)	104.2 (5)
Se(5)–Se(6)–Se(7)	106.1 (4)		

When considered alone, framework **Ia** is perforated with mutually perpendicular large tunnels running parallel to both the *a*- and *b*-axes. The approximate dimensions of these tunnels are 8 by 11 Å, large enough to fit small organic molecules. The tunnels

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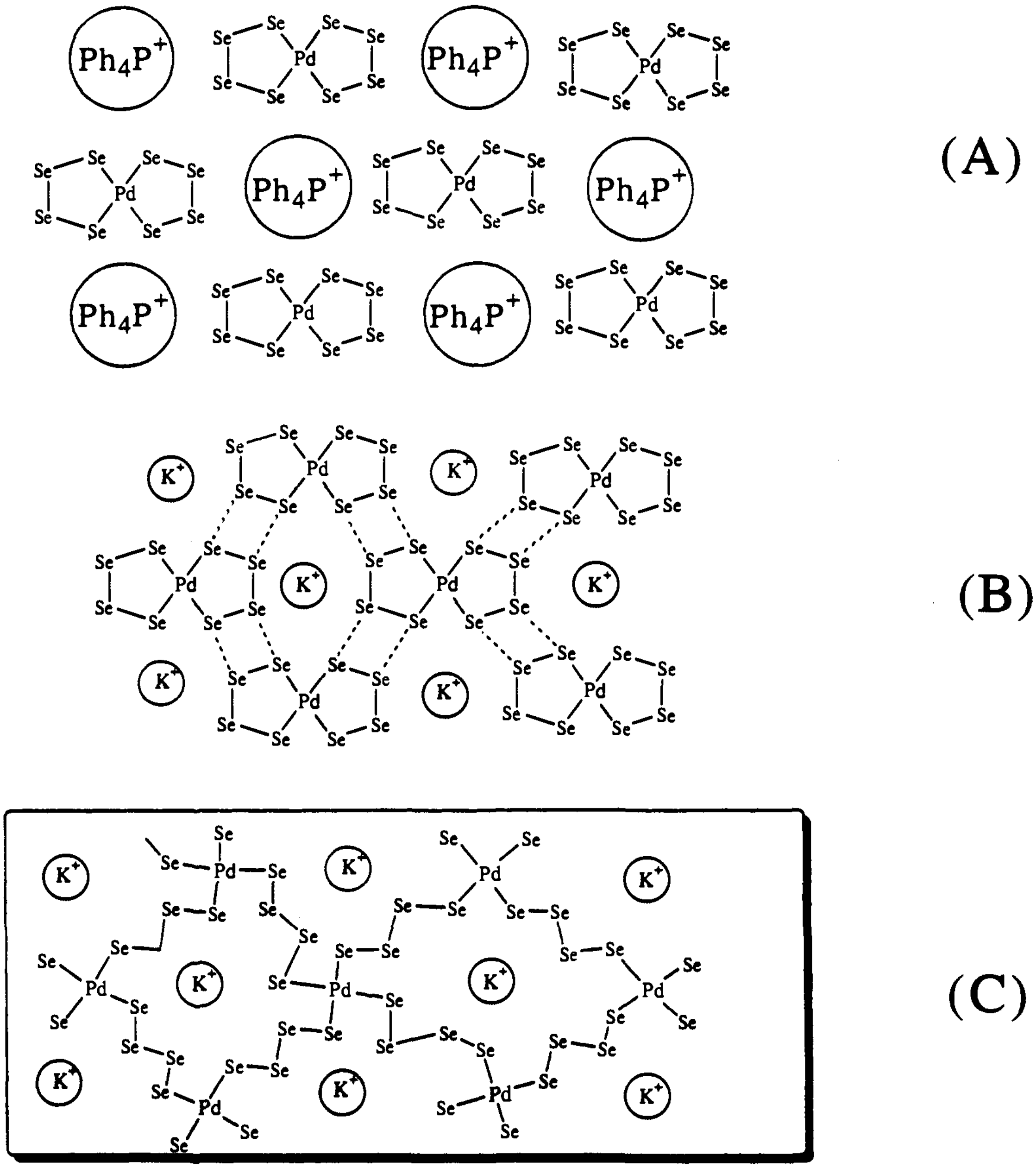

Figure 6. (A) Stable assembly of mutually screened Ph_4P^+ cations and $[Pd(Se_4)_2]^{2-}$ anions. (B) Substitution of large Ph_4P^+ for K^+ results in short anion–anion contacts (poor screening) developing destabilizing repulsive interactions. The latter are represented by dotted lines. (C) A stable assembly is possible by converting chelated Se_x^{2-} ligands to bridging. The repulsive interactions disappear, and an extended lattice of lower energy is formed by the association of Se_x^{2-} and Pd^{2+} ions.

of **Ia** are shown in Figure 4. Framework **Ib** is also perforated with mutually perpendicular tunnels running parallel to both the *a*- and *b*-axes. These tunnels are shown in Figure 5.

Interpenetrating 3D framework structures are rare in nature, examples include Cu_2O, $M(CN)_2$ (M = Cd, Zn), $Cu[(NC(CH_2)_4CN)_2]NO_3$, and some alkali borates.[12,13] A remarkable example of multiple identical diamondoid frameworks comes from the organic compound adamantane-1,3,5,7-tetracarboxylic acid.[14] A key difference in $K_2[PdSe_{10}]$ is that its frameworks are chemically and structurally inequivalent, whereas in the known examples the frameworks are identical. The conformational flexibility of the polychalcogenide chains is beautifully manifested by the fact that two very different polyselenide ligands, Se_4^{2-} and Se_6^{2-} each in different frameworks serve as bridges between *equally* spaced Pd atoms. This means that the potentially larger $[Pd(Se_6)_2]^{2-}$ framework must "fold" or contract (by folding of the Se_6^{2-} chains) in order to fit inside the smaller $[Pd(Se_4)_2]^{2-}$ framework.

The distance between Pd atoms in the 3D diamond-like framework structure determine the size of adamantane cage. Compared to the distance between O atom centers in the Cu_2O structure (3.7 Å),[11] the distance between Pd centers in **Ia** and **Ib** (8.6 Å) is much longer creating a much larger adamantane cage. In order to avoid the disadvantage of creating too much empty space in the structure, two frameworks come together and coexist in such a way as to fill each other's empty cages and tunnels. Alternatively, each individual framework can be viewed as a host framework, raising the intriguing possibility of microporosity in this type of frameworks. Since the K^+ ion is too small to fit inside the adamantane cage and separately stabilize each framework, it would be more appropriate to use larger cations as templating agents.[15]

A molecular form of $[Pd(Se_4)_2]^{2-}$ was reported recently to

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crystallize as its Ph_4P^+ salt.[16] The structure of this complex is simple, composed of a square-planar Pd^{2+} center chelated by two Se_4^{2-} ligands. Although this illustrates the importance of counterion in determining anion structure, it does not explain why the K^+ salt of the same composition is polymeric. The origin of such an explanation must lie in the packing arrangement of the molecular $(Ph_4P)_2[Pd(Se_4)_2]$. In the latter, the anions and cations pack in such a way as to screen each other. When the large Ph_4P^+ cation is changed to the drastically smaller K^+, the $[Pd(Se_4)_2]^{2-}$ anions can no longer be effectively screened, resulting in destabilizing repulsions. If the stoichiometry is to remain the same, one way for the system to respond and avoid these destabilizing repulsions is to convert the Se_4^{2-} ligands from chelating one Pd atom to bridging two neighboring Pd atoms. This is shown schematically in Figure 6. In this fashion the number and type (i.e., Pd–Se, Se–Se) of bonds remains constant, and thus there is no cost in enthalpy. However, the repulsive interactions transform into attractive ones leading to a lattice energy gain (in the Madelung sense). This concept can be used to predict or eliminate possible structures for charged molecular or polymeric anions upon changing the nature and size of the corresponding noninteracting counterion. It should be broadly applicable in cases where the structural framework of interest is relatively labile.

$(NH_4)_2[PdS_{11}]$[7] (**II**), the polysulfide analogue of I, is polymeric and among the earliest known metal polychalcogenide compounds. However, $[PdS_{11}]^{2-}$ *is not isostructural* to I and possesses a unique layered structure composed of square-planar Pd^{2+} ions and disordered S_5^{2-} and S_6^{2-} ligands. NH_4^+ ions fill voids in the tetragonal network of $[PdS_{11}]^{2-}$. **II** exhibits no interpenetrating networks. Given the similar dimensions of K^+ and NH_4^+, the different architectures of $K_2[PdSe_{10}]$ and $(NH_4)_2[PdS_{11}]$ underscore the ever increasing conviction that polysulfide and polyselenide chemistry are divergent more often than not.

The reaction reported here should be general. The combination of Q_x^{2-} species with proper counterions and divalent cations which prefer square-planar (i.e., Ni, Pd) or tetrahedral (e.g., Zn, Cd, Hg, In) coordination geometries should give new compounds with related three-dimensional structures.

Even though microporous metal chalcogenide compounds do not exist yet, they are of considerable interest. The combination of microporosity and unique properties of metal chalcogenides such as catalytic, semiconducting, or photoconducting[17] could result in new types of multifunctional materials. Attempts toward this goal were reported recently with the syntheses of (Me_4N)-GeS_x,[18] $(Me_4N)[Sb_3S_5]$,[19] and $(Et_4N)_4[Hg_7Se_9]$[20] compounds.

Acknowledgment. Financial support from the National Science Foundation for a Presidential Young Investigator Award, CHE-8958451, is gratefully acknowledged. The X-ray instrumentation used in this work was purchased in part with funds from NSF Grant CHE-8908088. We also thank the Center of Electron Optics at Michigan State University for using the electron microscopy facilities.

Supplementary Material Available: Tables of atomic coordinates and anisotropic thermal parameters of all atoms and calculated and observed powder patterns (5 pages); a listing of calculated and observed $(4F_o/4F_c)$ structure factors (7 pages). Ordering information is given on any current masthead page.

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