From Discrete to Extended Guests: A Novel Supramolecular $[Hg_6As_4](AgCl_3)_2$ Assembly Featuring One-Dimensional $(AgCl_3)^{2-}$ Anions

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Keywords: Arsenic / Host–guest systems / Mercury / Silver / Supramolecular chemistry

A novel supramolecular assembly $[Hg_6As_4](AgCl_3)_2$ has been prepared and its structure determined. It crystallizes in the monoclinic system, space group $I2/a$ (no. 15), with the unit-cell dimensions a = 14.690(1), b = 9.1851(7), c = 20.285(1) Å, β = 93.17(1)°, and Z = 4. The crystal structure comprises a three-dimensional cationic host framework, $[Hg_6As_4]^{4+}$, built of mercury and arsenic atoms and one-dimensional $^1_\infty(AgCl_3)^{2-}$ guest anions built of corner-sharing $[AgCl_4]$ tetrahedra. The title compound is the first example of a perfectly ordered supramolecular architecture having an infinite chain anion as a guest moiety embedded into a mercury–pnicogen cationic framework.

Introduction

The formation of supramolecular assemblies is usually controlled by the size, shape, and, where appropriate, charge of guest species that template the host framework.[1] The use of rigid guests enables prediction and successful realization of the desired host-framework topology. Supramolecular architectures form spontaneously through a self-assembly process; the resulting structures typically exhibit perfect ordering of the guests within the cavities of the framework. Such an ordering is achieved due to a reasonably large number of weak host–guest interactions.[2] The process of self-assembly is hard to follow and control in the case of high-temperature ampoule synthesis,[3] therefore only knowledge of typical host frameworks and guest units that can form in a given system can be used for predicting new architectures. Such knowledge is frequently based merely on trends in oxidation states, coordination numbers, and bond lengths.[4]

A broad family of supramolecular assemblies based on the mercury–pnicogen cationic frameworks has been reported in the past decade.[2,5] In these frameworks, mercury atoms possess linear coordination while pnicogen atoms have a tetrahedral environment. There are many possible arrangements of mercury and pnicogen atoms, leading to different topologies of the cationic framework guided by the size, shape, and charge of the guest anions. The guests that take their "assigned" positions within the cavities of the framework have been represented so far by discrete anions only. Octahedral,[5] tetrahedral,[6] pyramidal,[7] and triangular[2] anions have been successfully embedded into closed cavities of host frameworks of complementary topology. We therefore tried to find out if infinite anions could be accommodated by a mercury–pnicogen cationic matrix. Group 11 elements seemed to be a logical choice for such work since they frequently form infinite anions through condensation of different building units.[8] In particular, the $AgX_n^{(n-1)-}$ anions are well known[9,10] to form chains of condensed tetrahedra in the presence of different inorganic and organic cations.

In this article, we report the first supramolecular architecture in which a one-dimensional (1D) anionic chain, instead of a discrete anion, serves as a guest unit in an $[Hg_6As_4](AgCl_3)_2$ supramolecular assembly.

Results and Discussion

$[Hg_6As_4](AgCl_3)_2$ was prepared by a stoichiometric ampoule synthesis at elevated temperatures (see Exp. Sect. for details) as a bright-red, polycrystalline solid that is stable in moist air. It crystallizes in the monoclinic space group $I2/a$. The crystal structure comprises a three-dimensional (3D) host framework built of mercury and arsenic atoms and one-dimensional (1D) guest anions built of corner-sharing $[AgCl_4]$ tetrahedra (Figure 1).

There are six crystallographic positions occupied by mercury atoms (Table 1), each having linear coordination by two arsenic atoms. The Hg–As distances (Table 2) are similar to those reported in the literature.[5–7,11] The As–Hg–As angles (Table 3) deviate significantly from 180° only for the Hg(1) and Hg(2) atoms, each experiencing an influence of two distant chlorine atoms. The corresponding

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  DOI: 10.1002/ejic.200400205

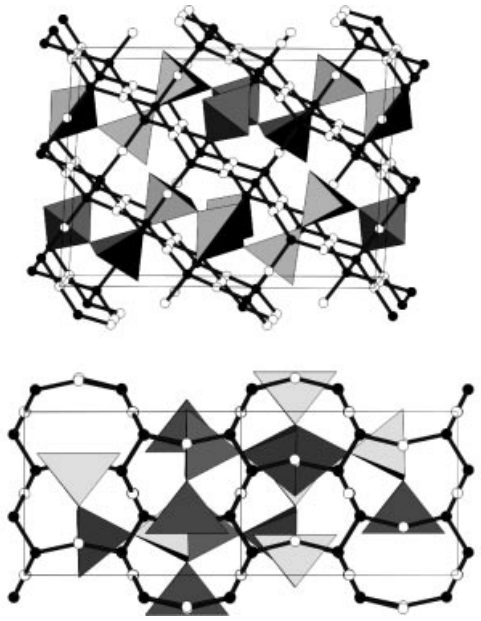

Figure 1. Crystal structure of $[Hg_6As_4](AgCl_3)_2$: (top) view almost along the *c* axis; (bottom) projection onto the (101) plane; the mercury and arsenic atoms composing the cationic framework are shown as open and filled spheres, respectively; $[AgCl_4]$ anions are represented by tetrahedra

Table 1. Atomic positions for $[Hg_6As_4](AgCl_3)_2$

Atom	Position	*x/a*	*y/b*	*z/c*	U_{eq} [Å^2][a]
Hg(1)	8f	0.0814(1)	0.2071(1)	0.3324(1)	0.0103(1)
Hg(2)	4e	1/4	0.8067(1)	0	0.0103(1)
Hg(3)	4b	0	0	1/2	0.0093(1)
Hg(4)	4a	0	0	0	0.0093(1)
Hg(5)	8f	0.1663(1)	0.0000(1)	0.1686(1)	0.0094(1)
Hg(6)	8f	0.3338(1)	0.0009(1)	0.3343(1)	0.0095(1)
As(1)	8f	0.2157(1)	0.1416(1)	0.2687(1)	0.0039(2)
As(2)	8f	0.1173(1)	0.86334(9)	0.0671(1)	0.0044(2)
As(3)	8f	0.9508(1)	0.1420(1)	0.3999(1)	0.0039(2)
Ag(1)	8f	0.1036(1)	0.7197(1)	0.3320(1)	0.0383(2)
Ag(2)	4e	1/4	0.3427(2)	0	0.0493(4)
Cl(1)	8f	0.9950(2)	0.3881(3)	0.2370(1)	0.0200(5)
Cl(2)	8f	0.1722(1)	0.5760(3)	0.9240(1)	0.0145(5)
Cl(3)	8f	0.1379(2)	0.4823(3)	0.3873(1)	0.0191(5)
Cl(4)	4c	1/4	3/4	1/4	0.0156(7)
Cl(5)	8f	0.1211(2)	0.2545(2)	0.0633(1)	0.0210(5)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} *tensor.*

Hg···Cl interactions (2.80−2.87 Å) are much longer than typical Hg−Cl covalent bonds of 2.27−2.29 Å,[12] but do not exceed the Hg···Cl host−guest separations of 3.07−3.34 Å observed in other supramolecular architectures of general formula $[Hg_6Pn_4](MCl_6)Cl$ (Pn = P or As; M = Ti, Mo, or In).[13]

All three crystallographically independent arsenic atoms display similar tetrahedral coordination. They each form one As−As bond and three Hg−As bonds. The As−As bond length (Table 2) is typical for a single bond,[14] which leads to the assignment of a −2 oxidation state to the arsenic atoms. Linear coordination of the mercury atoms with

Table 2. Selected interatomic distances [Å] for $[Hg_6As_4](AgCl_3)_2$

As(1)−As(1)	2.375(1)
As(1)−Hg(1)	2.4909(9)
As(1)−Hg(5)	2.4855(9)
As(1)−Hg(6)	2.4892(9)
As(2)−As(3)	2.379(1)
As(2)−Hg(2)	2.4928(9)
As(2)−Hg(4)	2.4775(9)
As(2)−Hg(5)	2.4843(9)
As(3)−As(2)	2.379(1)
As(3)−Hg(1)	2.4907(9)
As(3)−Hg(3)	2.4875(9)
As(3)−Hg(6)	2.4881(9)
Hg(1)···Cl(1)	2.802(2)
Hg(1)···Cl(3)	2.867(2)
Hg(2)···Cl(2)	2.825(2) × 2
Ag(1)−Cl(3)	2.491(3)
Ag(1)−Cl(1)	2.494(3)
Ag(1)−Cl(2)	2.795(3)
Ag(1)−Cl(4)	2.804(1)
Ag(2)−Cl(5)	2.481(3) × 2
Ag(2)−Cl(2)	2.843(3) × 2

Table 3. Selected bond angles [°] for $[Hg_6As_4](AgCl_3)_2$

As(3)−Hg(1)−As(1)	152.06(4)	Hg(5)−As(2)−Hg(2)	111.03(3)
As(2)−Hg(2)−As(2)	155.88(4)	As(2)−As(3)−Hg(3)	109.00(4)
As(3)−Hg(3)−As(3)	180.0	As(2)−As(3)−Hg(6)	108.22(4)
As(2)−Hg(4)−As(2)	180.0	Hg(3)−As(3)−Hg(6)	108.70(3)
As(2)−Hg(5)−As(1)	178.75(3)	As(2)−As(3)−Hg(1)	107.38(4)
As(3)−Hg(6)−As(1)	179.41(3)	Hg(3)−As(3)−Hg(1)	112.13(3)
As(1)−As(1)−Hg(5)	106.72(5)	Hg(6)−As(3)−Hg(1)	111.31(3)
As(1)−As(1)−Hg(6)	108.11(5)	Cl(3)−Ag(1)−Cl(1)	153.11(10)
Hg(5)−As(1)−Hg(6)	109.19(3)	Cl(3)−Ag(1)−Cl(2)1	103.31(7)
As(1)−As(1)−Hg(1)	108.99(5)	Cl(1)−Ag(1)−Cl(2)	97.75(9)
Hg(5)−As(1)−Hg(1)	110.24(3)	Cl(3)−Ag(1)−Cl(4)	102.21(6)
Hg(6)−As(1)−Hg(1)	113.36(3)	Cl(1)−Ag(1)−Cl(4)	92.70(6)
As(3)−As(2)−Hg(4)	107.29(4)	Cl(2)−Ag(1)−Cl(4)	93.98(6)
As(3)−As(2)−Hg(5)	107.77(4)	Cl(5)−Ag(2)−Cl(5)	141.88(13)
Hg(4)−As(2)−Hg(5)	111.10(3)	Cl(5)−Ag(2)−Cl(2)	105.12(7) × 2
As(3)−As(2)−Hg(2)	109.21(4)	Cl(5)−Ag(2)−Cl(2)	103.38(7) × 2
Hg(4)−As(2)−Hg(2)	110.32(3)	Cl(2)−Ag(2)−Cl(2)	82.16(10)

no Hg−Hg bonds present points to a +2 oxidation state of mercury. Consequently, the host framework carries a +4 charge per formula unit that can be formulated as $[Hg_6As_4]^{4+}$. The formula of the cationic framework is the same as in the series of supramolecular complexes with general formula $[Hg_6As_4](MX_6)Y$ (M = Ti, Mo, Cr, Fe, In, Sb, Hg; Y = Cl, Br, I, Hg).[5] However, the frameworks of the latter assemblies have a different topology (Figure 2). They possess two types of symmetric cavities capable of trapping guests of two different sizes. The guests carry a total charge of −4 to compensate for the positive charge of the framework. In the case where M is a trivalent metal, this compensation is achieved by a combination of two guest anions, $M^{III}X_6^{3-}$ and Y^-, occupying the larger and smaller cavities, respectively. If M is a divalent metal, the octahedral $M^{II}X_6^{4-}$ anions do not require additional charge-compensating guests, whereas the smaller cavities either trap zerovalent mercury or remain empty. In the title compound, all the cavities are almost of the same size and less symmetric.

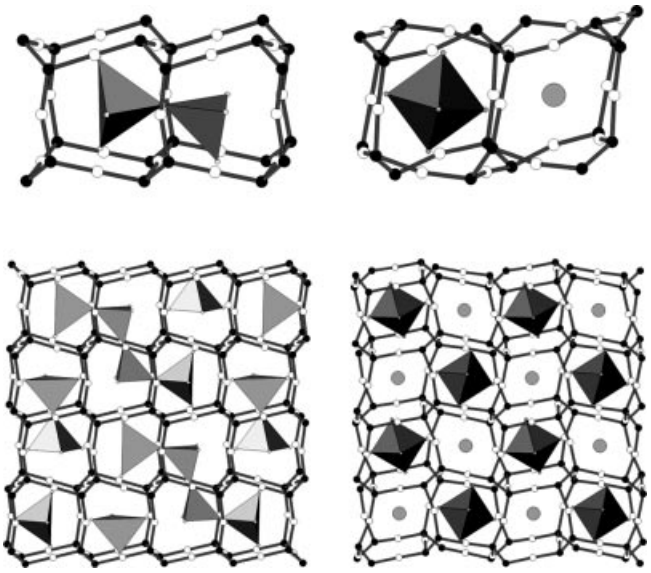

Figure 2. Topology of the $[Hg_6As_4]$ framework in the title compound (left) and in the $[Hg_6Pn_4](MX_6)Y$ complexes (right): two adjacent cages (top) and a 4×4 grid (bottom); mercury: open circles; pnicogen: filled circles; guest units are shown in polyhedral presentation save for the monoatomic guest shown as large gray circles

The difference in the framework topology reflects their different hierarchical origin. The title compound framework originates from a 4-connected As net having a lonsdaleite[15] topology in which three of four edges are expanded[16] by mercury atoms. On the other hand, the framework in the $[Hg_6As_4](MX_6)Y$ compounds originates from the γ-Si[17] topology. As a consequence, while the compositions of the frameworks are the same, their topologies are different.

The guest part of the structure is the 1D anionic chain composed of $AgCl_4$ tetrahedra sharing the corners (Figure 3). Two types of tetrahedra compose each chain; however, the environment of the Ag(1) and Ag(2) atoms centering the tetrahedra appears to be very similar. Each silver atom is surrounded by four chloride ions so that there are two short (2.48−2.49 Å) and two long (2.80−2.84 Å) Ag−Cl interactions involving terminal and bridging chlorine atoms, respectively. The Ag−Cl bond lengths fall in the range of 2.37−2.92 Å reported in the literature[9,18] for similar anions. The tetrahedral coordination of a silver atom by chloride ions is very common; moreover, there are examples of corner-sharing $[AgCl_4]$ tetrahedra in Cs_2AgCl_3 and Rb_2AgCl_3, for instance,[9] although the resulting chains are straight rather than spiral as in the title compound. The oxidation states for the silver and chlorine atoms apparently are +1 and −1, respectively, giving the formulation of the anionic chain as $^1_\infty(AgCl_3)^{2-}$. Hence, the notation of the supramolecular assembly as $[Hg_6As_4](AgCl_3)_2$ indicates the host−guest charge balance.

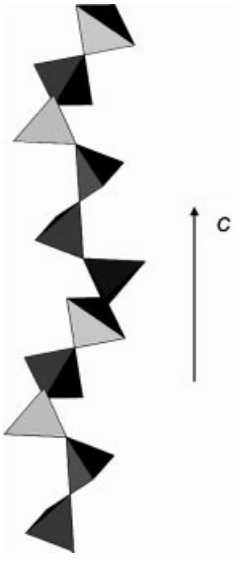

Figure 3. View of the 6-step $(AgCl_3)^{2-}$ anionic chain

The conformation of the 1D $(AgCl_3)^{2-}$ anion is best described as a 6-step spiral chain of tetrahedra,[19] reflecting the repeat unit (Figure 3). The chains are chiral; both enantiomers are present in the centrosymmetric structure. At this stage we cannot explain the observed conformation of the anionic chains. It is clear, though, that there are several factors that control the geometry and conformation of the guest unit. Firstly, a high negative charge of a single $AgCl_4^{3-}$ anion favors condensation of tetrahedra in a vertex-sharing fashion. Secondly, three of five independent chlorine atoms [Cl(1), Cl(2), and Cl(3)] have rather distant contacts with the mercury atoms of the host framework, ensuring that the host and guest units assemble into an ordered supramolecular architecture through weak host−guest interactions. The dimensions of the $[AgCl_4]$ tetrahedra and their fitting into the cavities of the framework should also be taken into account.

In general, this research confirms that it is possible to accommodate infinite anions within a host cationic framework. The title compound is the first example of a supramolecular architecture having a chiral tetrahedral chain as a guest moiety embedded into a mercury−pnicogen framework. The lack of structural data does not allow us to explain the observed features or to predict similar architectures. Nevertheless, the principal direction of a search for new compounds having infinite anions embedded into a mercury−pnicogen framework is clear. It is based on the propensity of the Group 11 elements to form condensed polyhedral anions of flexible geometry, with variable coordination and oxidation number of the central atom. A search for new compounds of this type is currently underway.

Experimental Section

Synthesis: A stoichiometric mixture of mercury(I) chloride, gray arsenic, and silver powder (all > 99.99% purity, total weight 0.5 g) was sealed in a silica tube, annealed at 680 K for 6 d, and then furnace-cooled. A comparison of the diffraction pattern [STADI-P (Stoe), Cu-$K_{\alpha 1}$ radiation] of the red, air-stable product with the theoretical one calculated on the basis of the data obtained during the crystal structure refinement revealed that the product was a desired new phase. However, the profile analysis of the diffractogram enabled us to suggest that only slight admixtures of mercury(II)

chloride and silver chloride were present. For a single-crystal preparation, a mixture of Hg_2Cl_2, As, and Ag taken in a 3:4:2 molar ratio was annealed in a silica tube at 680 K for 5 d. Red crystals were found in the furnace-cooled product.

Crystal Structure Determination: A red crystal with approximate dimensions 0.09 mm × 0.04 mm × 0.03 mm was selected from the reaction product for X-ray crystallographic analysis. The X-ray intensity data were recorded at 173(2) K (Bruker KRYO-FLEX) with a Bruker SMART APEX CCD-based X-ray diffractometer system equipped with graphite-monochromated Mo-K_α radiation (λ = 0.71073 Å) operated at 1800 W. The detector was placed at a distance of 6.140 cm from the crystal. The data collection covered approximately a hemisphere of the reciprocal space. A total of 1850 frames were collected with a scan width of 0.3° in ω and an exposure time of 20 s/frame. The frames were integrated with the Bruker SAINT software package[20] using a narrow-frame integration algorithm. The integration of the data using a monoclinic unit-cell yielded a total of 11582 reflections to a maximum 2θ angle of 56.45° (0.75 Å resolution), of which 3204 were independent (R_{int} = 0.0389). The final cell constants are based upon the refinement of the *XYZ*-centroids of 4394 reflections above $20\sigma(I)$. Analysis of the duplicate reflections showed negligible decay during data collection. Data were corrected for absorption effects using the empirical method (SADABS),[21] the ratio of minimum/maximum apparent transmission was 0.3865. The structure was solved and refined by a full-matrix least-squares procedure on $|F^2|$ in the *I*2/*a* space group using the Bruker SHELXTL (Version 6.12) software package.[22] The positions of six mercury and three arsenic atoms were found in direct-method *E* maps. The remaining atoms were located from a combination of least-squares refinement and difference Fourier synthesis. The isotropic refinement of the obtained model led to $R1$ = 0.078 with the slightly enlarged thermal displacement parameters of both silver atoms. However, refinement of the occupancies of these atoms proved full occupation for the corresponding positions. Attempts to refine the structure with split positions of the silver atoms did not lead to satisfactory results. Final anisotropic refinement led to the composition $[Hg_6As_4](AgCl_3)_2$, that is in excellent agreement with synthesis data, and to the atomic and structural parameters listed in Tables 1 and 4, respectively. Further details of the crystal structure determination may be obtained from the Fachinformationzentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany, on quoting the depository number CSD-413821.

Table 4. Data collection and structure refinement parameters for $[Hg_6As_4](AgCl_3)_2$

Empirical formula	$Ag_2As_4Cl_6Hg_6$
T [K]	173(2)
Space group	*I*2/*a*
a [Å]	14.690(1)
b [Å]	9.1851(7)
c [Å]	20.285(1)
β [°]	93.170(1)
V [Å^3]	2732.8(4)
Z	6
Density (calcd.) [Mg/m^3]	7.042
μ [1/mm]	60.523
λ [Å]	0.71073
Data collection range [°]	$2.01 < \theta < 28.23$
Reflections collected	11582
Independent reflections	3204 (R_{int} = 0.0389)
Parameters refined	129
Transmission factors	
Extinction coefficient	
$R1$,[a] $wR2$[b] [$I > 4\sigma(I)$]	0.0387, 0.1023
$R1$, $wR2$ (all data)	0.0509, 0.1100
Largest difference peak/hole [e/Å^3]	3.801/−5.111
Quality-of-fit[c]	1.026

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. [c] Quality-of-fit = $\{\Sigma[w(F_o^2 - F_c^2)^2]/(N_{obs} - N_{params})\}^{1/2}$, based on all data.

Acknowledgments

This work was supported by the U. S. Civilian Research & Development Foundation for the Independent States of the Former Soviet Union (award No. 12735), the Russian Foundation for Basic Research (grant No. 04-03-32351a), and the European Commission 5th Framework Program (contract HPRN-CT-2002-00193). E. V. D. is grateful to the National Science Foundation for funding the CCD diffractometer (CHE-0130985) at the University of Albany. A. V. S. thanks the Russian Science Support Foundation for a research award.

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Received March 13, 2004
Early View Article
Published Online August 13, 2004

