Coordination Modes

The Preparation and X-ray Crystal Structure of $[(\mathrm{AgI}_2)_n]\cdot n\,\mathrm{MF}_6$ (M = Sb, As): Diiodine Acting as a Donor in the Planar Polymeric $[(\mathrm{AgI}_2)_n]^{n+}$**

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Many of the more electronegative elements can be oxidized to homopolyatomic cations, for example, S_4^{2+}, I_3^+.[1] Partial positive-charge transfer can also be effected by coordination of these elements to a Lewis acid cation (e.g. Ag^+) in $[(S_8)_2Ag]^+$,[2a,b] $[S_8Ag]^+$,[2b] $[(Se_6Ag)_n]^{n+}$,[3] $[(Se_6Ag_2)_n]^{2n+}$,[3] and $[(P_4)_2Ag]^+$.[4a] The nature of the bonding in these cations, especially in the P_4[4b] and in the related CO[5] complexes, has been of the subject of recent controversies. The LUMOs and HOMOs of P_4 and I_2 have similar energies,[6] and consistently we were able to prepare a salt containing diiodine coordinated to Ag^+ ions, the first example of a dihalogen molecule coordinated to a simple metal cation in the solid state, that is, a metal dihalogen homoleptic species, the subject of this paper. Recently the related $AuCl_2^+$ was identified experimentally in the gas phase, and high-level calculations imply a geometry (angle Au-Cl-Cl: 102°) similar to that of the $\{AgI_2\}$ portion of $[(AgI_2)_n]^{n+}$ in the solid phase.[7] The $[(AgI_2)_n]^{n+}$ ion is also the first example, as far as we are aware, of a polymeric chain $[(AY_2)_n]^{n+}$ in which A and Y are any elements. Cations $[Ag_2X]^+$,[8a] $[Ag_3X]^{2+}$[8b] and anions $[AgX_3]^{2-}$,[8c] $[Ag_2X_4]^{2-}$[8d] (X = halogen) containing Ag–X, but not X–X bonds, have been structurally characterized.

Molecular diiodine forms a large number of charge-transfer complexes by accepting donor (D) lone pairs or π electrons into its low lying acceptor σ* orbitals.[9] The resulting $D^{\delta+}\rightarrow I_2^{\delta-}$ adducts are linear with an I–I distance much longer than that in I_2 (g) (2.667(2) Å[10]). Recently F. A. Cotton et al.[11] reported that the Lewis acid $[Rh_2(O_2CCF_3)_4]$ reacts with diiodine to give a zigzag polymer $[Rh_2(O_2CCF_3)_4I_2]\cdot I_2$. The 95.31(2)° (av) Rh-I-I angle implies that diiodine donates electrons from its antibonding π* HOMO orbitals probably into the corresponding vacant Rh–Rh $p_zd_{z^2}$ hybrid orbitals (see the Supporting Information). Back donation of electrons into the I_2 σ* orbitals may occur (as shown in the Supporting Information) and it seems that this is more important than the donation from the I_2 antibonding π* orbitals as the I–I interatomic distance (2.7202(6) Å) is longer than that in I_2 (g) and the I–I distance of 2.666(2) Å of the clathrated I_2 in the same compound.

We report here the ready synthesis of $[(AgI_2)_n]\cdot n\,MF_6$ (M = Sb, As) by the reaction of excess $AgMF_6$ with I_2 (s) in liquid SO_2 according to Equation (1), giving purple–black, thermally stable, but highly moisture sensitive crystals.

$$AgMF_6 + I_2 \xrightarrow{SO_2\,(l)} 1/n\,[(AgI_2)_n]\cdot n\,MF_6\ (M = Sb,\ As) \qquad (1)$$

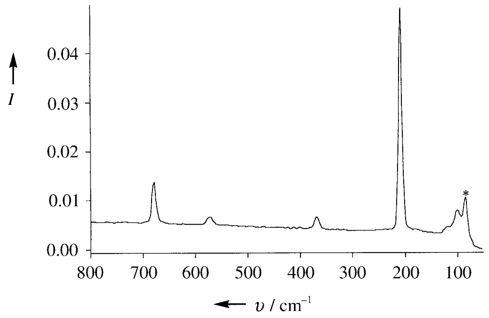

Figure 1. Raman spectrum of $[(AgI_2)_n]\cdot n\,AsF_6$ at room temperature. Scans: 1000; resolution: 4 cm^{-1}. The peak due to the glass of the sample container is marked with an asterisk.

Table 1: FT Raman frequencies (cm^{-1}) for I_5AsF_6,[17a,b] $[(AgI_2)_n]\cdot n\,SbF_6$ and $[(AgI_2)_n]\cdot n\,AsF_6$.[a]

I_5AsF_6[17a,b]	$[(AgI_2)_n]\cdot n\,SbF_6$	$[(AgI_2)_n]\cdot n\,AsF_6$	Assignments
413(0.2)			$2\nu_1$(I-I)
321(0.2)			ν_1(I-I) + ν_2(I-I-I)
228(0.8)			$2\nu_2$(I-I-I)
207(1)	207(10)	208(10)	ν_1(I-I)
114(10)	99(0.5)	101(0.5)	ν_2(I-Ag-I/I-I-I)
679(2)	645(2)	679(2)	$\nu_1(AsF_6^-/SbF_6^-)$
573(0.1)	574(0.2)	573(0.2)	$\nu_2(AsF_6^-/SbF_6^-)$
366(0.2)	281(0.6)	368(0.3)	$\nu_5(AsF_6^-/SbF_6^-)$

[a] Relative intensities in parentheses.

The products were characterized by FT Raman spectroscopy (Figure 1 for M = As and Table 1), and X-ray diffraction.[12] The $[(AgI_2)_n]^{n+}$ ion (Figure 2) has a similar zigzag structure to $\{(Rh_2I_2)_n\}$ chain in $[Rh_2(O_2CCF_3)_4I_2]\cdot I_2$ except that $[(AgI_2)_n]^{n+}$ is planar, the {Rh–Rh} unit is replaced by Ag^+ and the I–I distance in $[(AgI_2)_n]\cdot n\,MF_6$ (2.6744(18) Å M = Sb; 2.661(2) Å M = As) is shorter than that of I_2 (2.7202(6) Å) coordinated to Rh in $[Rh_2(O_2CCF_3)_4I_2]\cdot I_2$. The $[(AgI_2)_n]^{n+}$ ion is simpler and the fluorine–cation distances allow for an estimate of the positive charge residing on the Ag and I atoms.[15] The overall structure consists of planar $[(AgI_2)_n]^{n+}$ chains stacked down the *c* axis separated by sheets of $[MF_6]^-$ ions, linked by fluorine–cation contacts (Figure 3 for M = Sb; $[AsF_6]^-$ is disordered). The net charge on the silver as deduced by the contact to anionic fluorines is +0.472 (M = Sb).[15] The charge on each of the iodine atoms is +0.230 (M = Sb), which implies that I_2 acts as a donor resulting in

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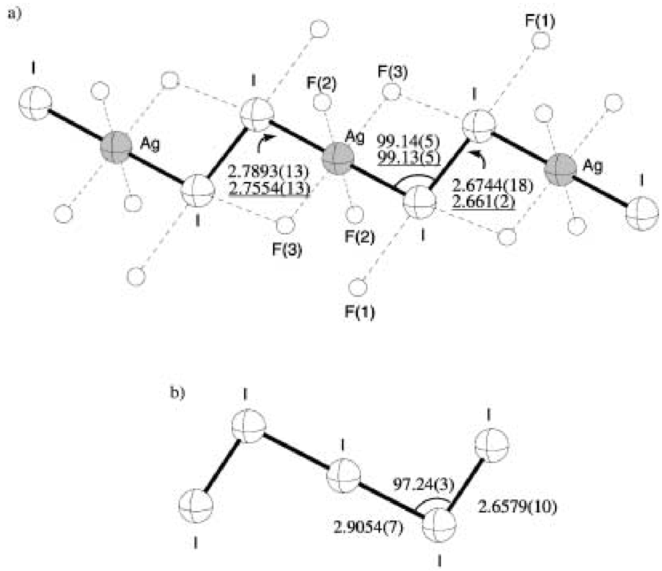

Figure 2. a) $[(AgI_2)_n]^{n+}$ ions in $[(AgI_2)_n]\cdot n\,MF_6$ (M = Sb, As) with cation–anion contacts (M = Sb) for Ag···F less than 2.92 Å, I···F less than 3.45 Å. Thermal ellipsoids were drawn at the 50% probability level. Selected distances [Å] and angles [°]: I···F(1) 2.95 (1), I···F(3) 3.44 (1), Ag···F(2) 2.58(1), Ag···F(3) 2.60 (1), I-Ag-I 180.0 (0). In $[(AgI_2)_n]\cdot n\,AsF_6$, $[AsF_6]^-$ ions are disordered. b) Structure of I_5^+ in I_5AsF_6.[17a,b]

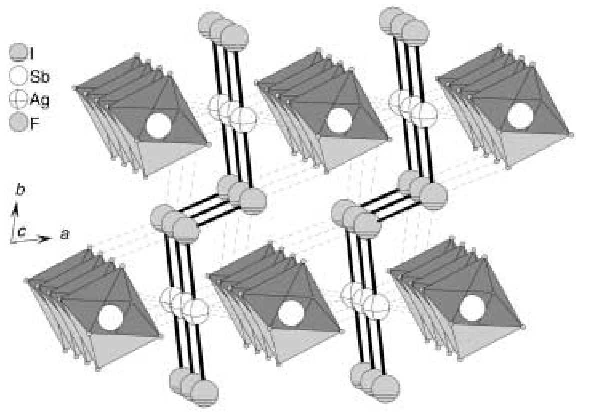

Figure 3. A view of $[(AgI_2)_n]\cdot n\,SbF_6$, the $[SbF_6]^-$ ions shown as polyhedrons. The structure of $[(AgI_2)_n]\cdot n\,AsF_6$ is similar except that the $[AsF_6]^-$ ions are disordered.

about equal positive charges on Ag and I_2. All estimated charges in one {AgI_2} fragment add up to about 1 valence unit (for M = Sb $0.472 + 2\times 0.230 = +0.932$), in good accord with the formal charge of +1. The I–I interatomic distance in $[(AgI_2)_n]\cdot n\,MF_6$ (2.6744(18) Å M = Sb; 2.661(2) Å M = As) is similar to that in I_2 (g) (2.667(2) Å[10]), I_3^+ (2.660(2), 2.669(2) Å) in I_3AsF_6[16] and the terminal I–I (2.6579(10) Å) in the I_5^+ ion in I_5AsF_6[17a,b] (Figure 2), and therefore the I–I bond in $[(AgI_2)_n]\cdot n\,MF_6$ has a bond order of 1. The Ag–I bond length (2.7893(13) Å M = Sb; 2.7554(13) Å M = As) implies 0.331 valence units for M = Sb and 0.362 for M = As.[15] The geometry of the {I-I-Ag-I-I} portion of the $[(AgI_2)_n]^{n+}$ chain is very similar to that of the I_5^+ ion in I_5AsF_6[17] (Figure 2). The Raman spectra of these compounds are also similar, thus aiding the assignments of $[(AgI_2)_n]^{n+}$ given in Table 1. The $\tilde{\nu}$ (I–I) stretching frequency (207 cm^{-1} M = Sb, 208 cm^{-1} M = As) is lower than I_2^+ (238 cm^{-1}) in $I_2(Sb_2F_{11})$[18a] and very similar to $\tilde{\nu}$ (I_2 (g), 215 cm^{-1}).[18b] The symmetric $\tilde{\nu}$ (I-Ag-I) can be compared to the corresponding $\tilde{\nu}$ (I-I-I, ~110 cm^{-1}) in I_3^-.[9a] In valence bond terms the structure of I_5^+ can be described by the resonance structures given in Figure 4a and a′ and that of $[(AgI_2)_n]^{n+}$ (Figure 4b, c, and c′). This model implies an I–I bond order of 1 in $[(AgI_2)_n]^{n+}$ and the presence of positive charge on silver and the diiodine molecule. Alternatively the bonding in the chain can be viewed as arising from the monopole on Ag^+ and the induced dipole on the adjacent iodine atom and by specific donation of electrons from the π* I_2 HOMO ($E = -0.342$ atomic units (a.u.))[6] to the 5s Ag^+ LUMO ($E = -0.278$ a.u.)[6] and the empty 5p orbital as shown in Figure 5a–c. Some back donation from the Ag^+ $4d^{10}$ HOMO ($E = -0.788$ a.u.)[6] to the I_2 σ* LUMO ($E = -0.080$ a.u.)[6] can occur (Figure 5d), but is likely to be less favorable as the energy gap is larger, although the reduction of charge on Ag^+ reduces this gap, that is, the interactions are synergic. However, based on this model, an I–I distance of slightly less than 2.667 Å (I_2, g) would be anticipated.[19]

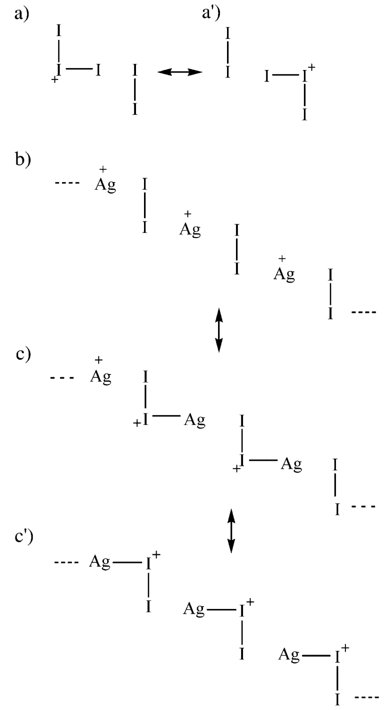

Figure 4. Valence-bond structures of I_5^+ (a and a′) and a portion of $[(AgI_2)_n]^{n+}$ (b–c′).

Whatever the nature of the bonding between Ag^+ and I_2 in the planar polymeric cation, its geometry, the similarity of the {I-I-Ag-I-I} fragment in $[(AgI_2)_n]^{n+}$ to I_5^+, and the presence of positive charge on all atoms imply that molecular I_2 acts as a donor to Ag^+ in $[(AgI_2)_n]^{n+}$. Thus $[(AgI_2)_n]^{n+}$ provides a simple and rare (the second) example of diiodine acting as a donor.

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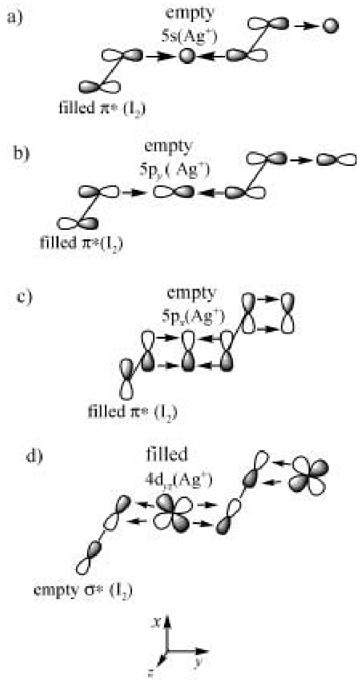

Figure 5. Schematic representation of the covalent $I_2\cdots Ag$ bonding in the $[(AgI_2)_n]^{n+}$ ion.

Experimental Section

All manipulations were performed by using grease-free metal apparatus and dry-box techniques under a dry N_2 atmosphere as previously described.[22] Reactions were carried in a two-bulb (pyrex), two-valve (teflon in glass) vessel, incorporating a medium frit and a teflon-coated stirring bar magnet. FT Raman spectra were recorded at 293 K on a Bruker IFS66 FTIR equipped with a Bruker FRA106 FT Raman accessory by using an Nd-YAG laser (emission wavelength, 1064 nm; maxium laser power, 3009 mW; used laser power, 1.8%). Samples were sealed in melting point capillaries, and data were collected in the backscattering mode (180° excitation; resolution 4.0 cm^{-1}). Chemical analyses were performed by Galbraith Laboratories, Inc. (U.S.). Analyses by scanning electron microscopy (JEOL-6400 SEM) were obtained with EDAX Genesis EDS X-ray analyzer. $AgSbF_6$ (SynQuest. Labs. Inc.) was dissolved in liquid SO_2 and the insoluble impurities were discarded. $AgAsF_6$ was prepared as described in the literature.[23] The purity of $AgMF_6$ was checked by FT Raman spectroscopy. Iodine (BDH analytical reagent) was further purified by sublimation. Sulfur dioxide (Matheson, anhydrous, 99.85%) was vacuum-distilled and stored over CaH_2 before use.

Preparation of $[(AgI_2)_n]\cdot n\,MF_6$ (M = Sb, As): $AgSbF_6$ (3.240 g, 9.429 mmol) and I_2 (0.714 g, 2.813 mmol) were placed into one bulb. The mixture quickly became pale yellow, thus indicating that a reaction had occurred in the solid state. About 8 g liquid SO_2 was condensed onto the mixture through the frit, giving a dark-red solution over a small amount of colorless precipitate that was tentatively identified by scanning electron microscopy and Raman spectroscopy as Ag_2ISbF_6, or $Ag_3I(SbF_6)_2$, or a mixture of both.[8] The solution was stirred for 1 day. A 40% yield of isolated dark-purple crystals was obtained from the filtrate held at 5 °C. The salt $[(AgI_2)_n]\cdot n\,AsF_6$ was prepared similarly (yield of isolated crystals: 60%). Elemental analysis calcd (%) for $[(AgI_2)_n]\cdot n\,AsF_6$: Ag 19.59, I 46.10, As 13.61, F 20.70; found: Ag 20.78, I 45.87, As 14.42, F 22.52. A sample of $[(AgI_2)_n]\cdot n\,AsF_6$ was heated at about 140 °C for one hour in a tube connected to a collection vessel in a static vaccum. No iodine was obtained.

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[12] a) X-ray structure determination of $[(AgI_2)_n]\cdot n\,SbF_6$: Rigaku AFC5R diffractometer with graphite-monochromated $Mo_{K\alpha}$ radiation, $T = 143 \pm 0.25$ K, unit cell determination: 24 reflections, corrections: Lorentz, polarization, and empirical absorption correction, direct methods with SHELX-97, refinement with SHELXL-97.[13] Space group: $P\bar{1}$ (no. 2), $Z = 1$, $a = 6.026(2)$, $b = 6.558(2)$, $c = 5.652(2)$ Å, $\alpha = 92.44(3)$, $\beta = 91.06(3)$, $\gamma = 74.43(3)°$, $V = 215.0(1)$ Å^3, $\rho_{calcd} = 4.614\ g\,cm^{-3}$,

$2\theta_{max}=60.1°$, reflections: 1365 collected, 1256 unique, 920 observed, 50 parameters, $R_1=0.057$, $wR_2=0.173$. b) X-ray structure determination of $[(\mathrm{AgI_2})_n]\cdot n\,\mathrm{AsF_6}$: Rigaku AFC5R diffractometer with graphite-monochromated $\mathrm{Mo_{K\alpha}}$ radiation, $T=183\pm1$ K, unit cell determination: 24 reflections, corrections: Lorentz, polarization, and absorption correction, direct methods with SHELXS-86,[14] refinement with SHELXL-97. Space group: $P\bar{1}$ (no. 2), $Z=1$, $a=5.915(3)$, $b=6.489(2)$, $c=5.444(2)$ Å, $\alpha=91.92(3)$, $\beta=90.21(4)$, $\gamma=77.23(3)°$, $V=203.7(1)$ Å^3, $\rho_{\text{calcd}}=4.488\ \mathrm{g\,cm^{-3}}$, $2\theta_{max}=60.1°$, reflections:1306 collected, 1201 unique, 760 observed, 69 parameters, $R_1=0.049$, $wR_2=0.143$. c) It is sometimes difficult to distinguish emphatically between silver and iodine atoms by X-ray diffraction criteria alone. The elemental analysis of the bulk material (M = As) clearly confirms the presence and empirical formula of the $\{\mathrm{AgI_2}\}$ fragment and the spectroscopic data (both salts) further confirms the identities of the silver and iodine atoms in the structure. d) Further details on the crystal structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: (+49) 7247-808-666; e-mail: crysdata@fiz-karlsruhe.de), on quoting the depository numbers CSD-413710 ($[(\mathrm{AgI_2})_n]\cdot n\,\mathrm{SbF_6}$) and CSD-413711 ($[(\mathrm{AgI_2})_n]\cdot n\,\mathrm{AsF_6}$).

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[19] However some I–I bond lengthening possibly occurs as: 1) The shortest I···F (1) contacts (2.95(1) Å) to I_2 have an I-I···F angle of 177.6° (M = Sb)[20] and are geometrically well positioned for $F^{\delta-}$ from $[\mathrm{SbF_6}]^-$ to donate some electron density into the low-lying I_2 σ* LUMO orbital; 2. The result of electrostatic attraction and repulsion between atoms in the linear $\mathrm{F(1)^{-0.115}\cdots I^{+0.23}{-}I^{+0.23}\cdots F(1)^{-0.115}}$.[21]

[20] For M = Sb, the shortest I–F contacts are also the only significant interactions between I and F. The I–F3 (3.44(1) Å) is almost at the I–F van der Waals distance (3.45 Å) (Figure 1). In the case For M = As, I-I···F is 169.5 or 169.4° relative to two disordered F atoms (50 % occupancy each; I–F distance: 2.9678(12) and 2.9887(11) Å).

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