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Structural diversity in silver salts of hexahalogenocarborane anions, $Ag(CB_{11}H_6X_6)$ (X = Cl, Br or I)

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Crystal structures have been determined for three $Ag(CB_{11}H_6X_6)$ compounds (X = Cl, Br or I) crystallized from arene solvents. They are all one-dimensional co-ordination polymers in the solid state with hexahalogenocarborane anions acting as bridging ligands. The co-ordination geometries of these three salts are, however, unique. The type and number of donors to silver(I) varies from one to another and is not particularly predictable.

Icosahedral carboranes constitute a new class of very weakly co-ordinating anions.[1,2] The 7–12 hexahalogenated derivatives $CB_{11}H_6X_6^-$ (X = Cl, Br or I), are larger, more thermodynamically stable, more chemically inert, less co-ordinating and less nucleophilic than the parent $CB_{11}H_{12}^-$ ion.[3] They play a very important role in stabilizing co-ordinatively unsaturated cations such as the silylium ion (R_3Si^+)[4–7] and the four-co-ordinate 5,10,15,20-tetraphenylporphyrinatoiron(III) ion, $[Fe(tpp)]^+$.[8] Silver(I) salts of carborane anions are very useful halide metathesis reagents for introducing weakly co-ordinating anions into complexes and they are finding immediate applications in metathesis, catalysis and oxidation chemistry.[2–12] They are also of structural interest in their own right because they illustrate the subtlety and diversity of co-ordination chemistry when anions have comparable donor ability to solvent molecules. Examples of crystallographically characterized silver(I) carborane complexes include $Ag(CB_{11}H_{12})\cdot 2C_6H_6$ **1**,[13] $Ag(CB_{11}H_{11}Br)\cdot C_6H_6$ **2**,[14] $Ag(CB_{11}H_{11}F)\cdot 2C_6H_6$ **3**,[15] $Ag(CB_9H_5Br_5)\cdot C_6H_5Me$ **4**[3] and $Ag(CB_9H_{10})$ **5**.[3] Each structure has a different co-ordination motif which could not be predicted from a knowledge of the constituent parts. Some patterns are, however, beginning to emerge. In the five known structures the carborane anion co-ordinates to Ag^+ *via* lone pairs on bromine atoms and/or *via* σ complexation of B–H bonded pairs from different positions of the cage. Three-co-ordinate silver is found in **1** with unusual η^1 co-ordination of benzene and 7,12 bridging of monodentate B–H bonds of the carborane. Four-co-ordinate silver is found in **2**, again with η^1 co-ordination of benzene but now with monodentate B–Br *and* B–H contacts from the bridging carboranes. On changing from the 12-bromo to the 12-fluoro carborane anion, **3** takes on a formally six-co-ordinate stereochemistry with two benzene ligands in asymmetric η^2 fashion and two bridging B–H bonds from the anions. Five-co-ordinate silver in **4** is attached to η^2-toluene, a bidentate dibromo interaction with one $CB_9H_5Br_5^-$ ion and a monodentate bromo interaction with a neighbouring anion. Compound **5** excludes arenes upon crystallization and the co-ordination geometry approximates linear two-co-ordination except that the bridging anions in this one-dimensional polymeric structure provide η^2-like co-ordination from adjacent pairs of B–H bonds.

As part of our attempt to understand the systematics of silver–carborane structural chemistry, we now report the crystal structures of the silver salts of three isostructural hexahalogenocarboranes, $Ag(CB_{11}H_6Cl_6)\cdot C_6H_4Me_2$-*p* **6**, $Ag(CB_{11}H_6Br_6)$ **7** and $Ag(CB_{11}H_6I_6)\cdot 0.5C_6H_6$ **8**.

Results and Discussion

Preparation

The compound $Ag(CB_{11}H_6Cl_6)$ is the most soluble of the three salts in arene solvents and X-ray-quality crystals were obtained by slow evaporation of a *p*-xylene solution. By contrast, $Ag(CB_{11}H_6I_6)$ and $Ag(CB_{11}H_6Br_6)$ are almost insoluble in pure arene solvents and had to be recrystallized from more polar solvents or from solvent mixtures. It is interesting that the hexaiodo species **8** crystallizes without co-ordinated (or lattice) MeCN from a mixture of benzene and MeCN, and the hexabromo species **7** crystallizes without $SiPr^i_2(H)Cl$ from a mixture of $SiPr^i_2(H)Cl$ and toluene. These results suggest that while the solubilizing action of MeCN and $SiPr^i_2(H)Cl$ may involve co-ordination, the interaction is labile and insufficiently strong to prevail in the solid state. Solubility presumably controls which structural motif is observed in the solid state.

Crystal structures

The co-ordination geometries of compounds **6**–**8** are shown in Fig. 1. Fig. 2 shows stereoscopic views of their crystal packings. Selected bond distances and angles are listed in Table 1. A feature common to all three compounds is the one-dimensional co-ordination polymeric structure formed with halogen donor atoms from the bridging anions. Two of the three structures retain an arene molecule in the lattice but only in one of them, *p*-xylene in **6**, the arene is co-ordinated. These aromatic molecules fill the space between the zigzag polymeric chains. The shortest non-bonded $Ag\cdots X$ distance is 4.302 Å for **6** [X = Cl(9)], 4.961 Å for **7** [X = Br(11)] and 4.231 Å for **8** [X = I(12)], respectively. The co-ordination details are unique to each structure.

Compound **6** has a five-co-ordinate silver atom in a propeller arrangement of one η^2-*p*-xylene and two bidentate bridging $CB_{11}H_6Cl_6^-$ ions (considering the η^2-*p*-xylene as one co-ordination ligand site). The asymmetric η^2 fashion of *p*-xylene bonding, with Ag–C bond distances of 2.506(3) and 2.481(4) Å, is typical of many silver–arene complexes.[3,16] Arene bonding probably persists in solution and may contribute to the good solubility of $Ag(CB_{11}H_6Cl_6)$ in *p*-xylene. The Ag–Cl distances range from 2.640(1) to 2.926(1) Å which are comparable to those found in dichloroalkane complexes of silver(I).[17–20] The average B–Cl distance, 1.789(3) Å, is similar to the 1.803(9) Å average in $(Pr^i_3Si)CB_{11}H_6Cl_6$.[7] The longest B–Cl bond is associated with the shortest Ag–Cl(11) distance, but the extension is

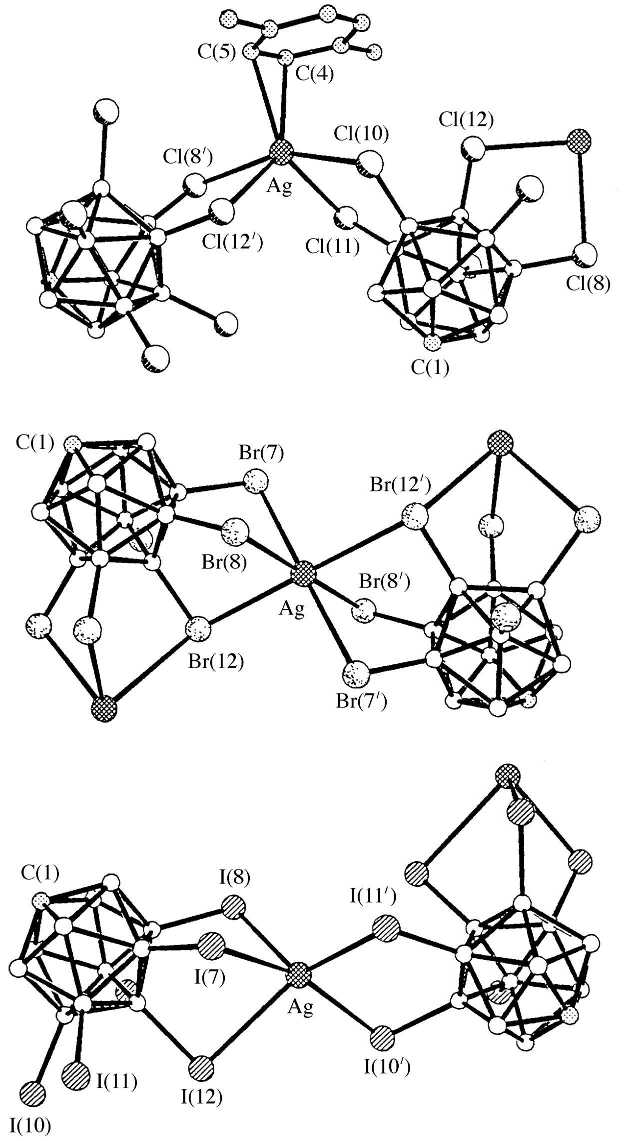

Fig. 1 Molecular structures of compounds **6** (top), **7** (middle) and **8** (bottom, solvate benzene is not shown) revealing only a portion of the infinite anion–silver–anion–silver zigzag chain

very small (*ca.* 0.02 Å). This is consistent with weak binding of the anion to silver.

The $CB_{11}H_6Br_6^-$ ion in compound **7** occupies a site of symmetry *m*, and the silver atom sits at an inversion centre. The six-co-ordinate geometry about silver is very similar to that found in the complex ion $[Ag(CB_{11}H_6Br_6)_2]^-$,[8] with the metal atom bonding to three bromine atoms from each of two bridging $CB_{11}H_6Br_6^-$ ions. The bromine atom attached to B(12) is shared by two silver ions, perhaps reflecting that this bromine atom is the most basic one. Reactivity patterns in the electrophilic halogenation of $CB_{11}H_{12}^-$ strongly suggest that the boron atom antipodal to carbon, *i.e.* B(12), is the most electron-rich site in the icosahedral cage.[14] The average Ag–Br distance of 2.862(2) Å is comparable to the 2.879(3) Å average in $[Ag(CB_{11}H_6Br_6)_2]^-$ [8] and lies in the range found in dibromoalkane complexes of silver(I): 2.816(2)–3.081(2) Å.[17] It is significantly longer than the 2.800(6) Å average in $Ag(CB_9H_5Br_5)\cdot C_6H_5Me$[3] and the 2.642(1) Å in $Ag(CB_{11}H_{11}Br)\cdot C_6H_6$,[14] indicating that the interaction of the anion with silver is relatively weak. The average B–Br distance, 1.97(2) Å, falls in the range 1.92–2.05 Å found in $(Pr^i_3Si)CB_{11}H_6Br_6$[7] and $[Ag(CB_{11}H_6Br_6)_2]^-$. The co-ordination polymer of **7** can dissociate in solution into a smaller unit, $[Ag(CB_{11}H_6Br_6)_2]^-$; in the presence of halide anions it reacts with [Fe(tpp)Br] in toluene to generate the ionic complex, $[Fe(tpp)][Ag(CB_{11}H_6Br_6)_2]$.[8]

Table 1 Selected bond distances (Å) and angles (°) for compounds **6**–**8**

	6 (chloro)	**7** (bromo)	**8** (iodo)
Ag–X(7)		2.870(2)	3.120(7)
Ag–X(8)	2.926(1)	2.777(2)	2.857(2)
Ag–X(9)			
Ag–X(10)	2.873(1)[a]		2.856(4)[b]
Ag–X(11)	2.640(1)[c]		2.927(9)[d]
Ag–X(12)	2.679(1)	2.940(1)	3.256(14)
Ag–C(4)	2.506(3)		
Ag–C(5)	2.481(4)		
B(7)–X(7)	1.793(4)	2.001(21)	2.165(20)
B(8)–X(8)	1.799(3)	1.975(18)	2.198(23)
B(9)–X(9)	1.769(4)		2.183(21)
B(10)–X(10)	1.786(4)		2.179(23)
B(11)–X(11)	1.803(3)	1.948(28)	2.158(21)
B(12)–X(12)	1.786(3)	1.964(30)	2.172(18)
C(1)–B	1.687(5)–1.713(6)	1.704(24)–1.724(38)	1.671(28)–1.748(28)
B–B	1.751(5)–1.801(4)	1.719(31)–1.792(28)	1.739(31)–1.838(28)
Ag–X(7)–B(7)		92.6(6)	88.5(5)
Ag–X(8)–B(8)	102.9(1)	94.1(5)	95.0(6)
Ag–X(9)–B(9)			
Ag–X(10)–B(10)	102.1(1)[e]		99.5(6)[f]
Ag–X(11)–B(11)	106.5(1)[e]		98.8(6)[f]
Ag–X(12)–B(12)	108.6(1)	91.0(3)	89.1(7)

Symmetry transformations: a $-\frac{1}{2}+x, \frac{1}{2}-y, z$; b $\frac{1}{2}+x, \frac{1}{2}-y, z$; c $-x, y, \frac{1}{2}+z$. [a] X(10) = Cl(10a). [b] X(10) = I(10c). [c] X(11) = Cl(11a). [d] X(11) = I(11c). [e] Ag = Ag(b). [f] Ag = Ag(c).

In the co-ordination structure of compound **8** the silver atom is complexed by iodine atoms to one anion in bidentate fashion and to another in tridentate fashion. The five-co-ordinate structure adopts a square-pyramidal arrangement. It is closely related to that of **7** except that six-co-ordination in the bromo compound is reduced to five-co-ordination in the iodo derivative by virtue of longer bond lengths. The benzene solvate appears only to fill space within the lattice. The shortest Ag ··· C separation is 6.25 Å. The Ag ··· I(12′) distance is 4.23 Å which is much longer than the 3.66 Å sum of the van der Waals radii for Ag and I.[21] So unlike compound **7**, the most electron-rich iodine atom, I(12), is not shared by two silver ions. Moreover, the bond formed from I(12) to silver is not the shortest. Apparently, crystal-packing forces and dimensional differences between the bi- and tri-dentate bites of the iodo anion disfavour stronger co-ordination by the most basic iodine atom. The Ag–I bond distances vary from 2.856(4) to 3.256(3) Å which fall in the range 2.777(4)–3.306(5) Å for diiodoalkane complexes of silver(I).[22,23] The average B–I distance, 2.179(30) Å, is indistinguishable from the 2.180(20) Å average in $(Pr^i_3Si)CB_{11}H_6I_6$[7] reflecting little perturbation of the anion by the cation in both structures.

It has recently been established by NMR spectroscopy that the dipolar charge distribution in these anions, reflected in the ^{11}B chemical shifts within the CB_{11} unit, is dependent upon the nature of the substituents on the cage.[7] The data in Table 2 show, however, that the size of the CB_{11} cage is not systematically affected by halogen substituents. The bond distances and angles within the CB_{11} cage stay within a very small range despite different electronegativities of the halogen substituents.

Conclusion

A number of conclusions can be drawn from this study. First, the bridging anion motif is common to all silver(I)–carborane structures determined to date. Often this creates a one-dimensional co-ordination polymer, but higher dimensionalities are also observed. This helps explain the low solubility of certain salts and may help to explain why certain silver salt metathesis reactions to not proceed to completion.[25] The for-

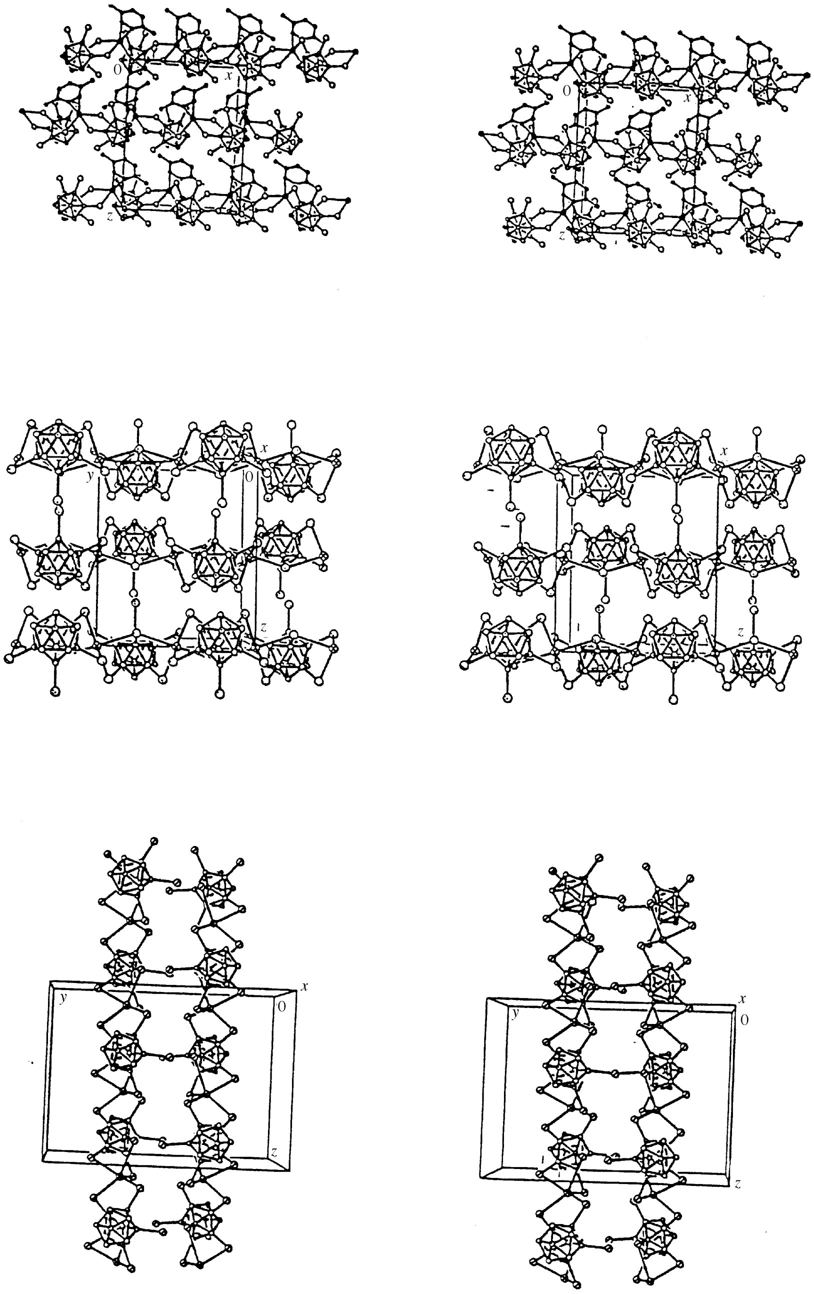

Fig. 2 Stereoscopic views of compounds **6** (top), **7** (middle) and **8** (bottom, solvate benzene is not shown)

Table 2 Diameters (Å) of $\mathrm{CB_{11}}$ cages in some 12-vertex *closo*-carborane derivatives

Compound	C(1)–B(12)	B···B (average)	Ref.
$\mathrm{AgCB_{11}H_{12}}$	3.21	3.39	13
$\mathrm{Ag(CB_{11}H_{11}Br)}$	3.17	3.39	14
$\mathrm{(Pr^i_3Si)CB_{11}H_6Cl_6}$	3.23	3.39	7
$\mathrm{(Pr^i_3Si)CB_{11}H_6Br_6}$	3.21	3.41	7
$\mathrm{(Pr^i_3Si)CB_{11}H_6I_6}$	3.27	3.39	7
$\mathrm{[Fe(tpp)][Ag(CB_{11}H_6Br_6)_2]}$	3.22	3.39	8
$\mathrm{Ag(CB_{11}H_6Cl_6)}$	3.20	3.39	This work
$\mathrm{Ag(CB_{11}H_6Br_6)}$	3.27	3.36	This work
$\mathrm{Ag(CB_{11}H_6I_6)}$	3.22	3.41	This work
$\mathrm{[NHMe_2Ph][CB_{11}Me_{12}]}$	3.27	3.39	24

mation of double salts can be anticipated in which a silver polymer coexists with other cations.

Despite being isostructural as discrete ions, the three anions adopt different crystal structures as their silver salts. This rather pointedly underscores the trend in co-ordinative flexibility that was becoming evident from existing structures. Three- through six-co-ordination, mono-, bi- and tri-dentate halogen atom binding, η^1 and η^2 π-arene co-ordination, and mono- and bi-dentate σ complexation of B–H bonds have all been observed. Silver has the necessary lability in solution to allow the formation of whatever solid-state structure is the most stable, *i.e.* least soluble. Apparently silver(I) can accommodate almost any reasonable co-ordinative geometry which compensates charge and packs efficiently.

In various structures it is possible to argue for consistencies with hard–soft acid–base principles. However, this does not seem to be universally useful amongst all structures and it is probably best to reserve such a discussion for data obtained in solution, where the packing constraints of polymer formation are absent. Likewise, in some cases we have been able to point out data that are consistent with the expectation of the 12 substituent (antipodal to carbon) being more basic than the 7–11 substituents. However, the 12-halogeno atom is sterically less accessible than those on the 7–11 pentagonal belt and apparently its higher basicity is not always manifest in closer approach to a metal centre. This is particularly true with cations

Table 3 Crystal data and details of data collection and structure refinement for compounds **6**–**8**

	6	**7**	**8**
Empirical formula	$C_9H_{16}AgB_{11}Cl_6$	$CH_6AgB_{11}Br_6$	$C_4H_9AgB_{11}I_6$
Crystal size/mm	0.4 × 0.5 × 0.8	0.3 × 0.3 × 0.2	0.3 × 0.2 × 0.4
M	563.7	724.3	1045.3
Crystal class	Orthorhombic	Orthorhombic	Orthorhombic
Space group	$Pna2_1$ (no. 33)	$Pnma$ (no. 62)	$Iba2$ (no. 45)
a/Å	12.965(1)	11.781(2)	15.063(1)
b/Å	10.487(1)	11.042(2)	19.584(2)
c/Å	16.023(1)	12.821(2)	14.267(2)
U/Å^3	2178.6(11)	1667.8(4)	4208.8(7)
Z	4	4	8
D_c/g cm^{-3}	1.719	2.885	3.300
Data collected	$+h + k - l$	$+h + k + l$	$+h + k + l$
2θ Range/°	2.0–55.0	4.0–108.0	2.0–45.0
μ/mm^{-1}	1.655	15.553	9.745
F(000)	1096	1296	3624
T/K	298	153	157
No. relections collected	2486	1361	1579
No. independent reflections	2486	982	1354
No. observed reflections ($\lvert F \rvert > n/\sigma \lvert F \rvert$)	2201 ($n = 6$)	730 ($n = 4$)	1252 ($n = 4$)
No. parameters refined	244	65	140
Data-to-parameters ratio	9.0:1	11.2:1	8.9:1
Goodness of fit	1.28	1.06	1.11
R_F*	0.029	0.049	0.030
R'	0.039	0.063	0.036
$\Delta\rho_{max}$, $\Delta\rho_{min}$/e Å^{-3}	0.39, −0.43	1.30, −1.71	0.85, −0.97
Largest, mean Δ/σ	0.000, 0.000	0.000, 0.000	0.078, 0.004

* Weighting scheme employed: $w^{-1} = \sigma^2(F) + nF^2$, $n = 0.0002$ **6**, 0.0023 **7** and 0.0004 **8**.

such as R_3Si^+ and $[Fe(\eta^5\text{-}C_5H_5)(CO)_2]^+$ that are bulkier than Ag^+. In these cases co-ordination occurs exclusively at the 7 position.[3–7]

Finally, we comment on the weakness of the co-ordination of the hexahalogenocarborane anions. The bond-length comparisons roughly equate their binding to that of alkyl halides. Similarly, the appearance of co-ordinated arenes in some of the structures puts the binding of carborane anions on a par with arene solvent molecules. We have thus arrived at the point where most of the solvents useful for dissolving ionic or ion-like species compete with the anion binding.

Experimental

All operations were performed under purified N_2 or He with rigorous exclusion of air and water using standard Schlenk vacuum-line and glove-box techniques. Anhydrous silver salts of hexahalogenocarborane anions were prepared by literature methods.[7] Arene solvents were distilled from sodium–benzophenone, and MeCN was distilled from CaH_2 prior to use.

Preparations of compounds 6–8

The compound $Ag(CB_{11}H_6Cl_6)$ was dissolved in *p*-xylene to give a clear colourless solution. Slow evaporation yielded X-ray-quality crystals of **6**. To a suspension of $Ag(CB_{11}H_6Br_6)$ in toluene was added 1.5 equivalents of $SiPr^i_2(H)Cl$. The mixture was then stirred overnight at room temperature to give a clear pale yellow solution. Hexane vapour diffusion resulted in colourless crystals. To a suspension of $Ag(CB_{11}H_6I_6)$ in benzene was added dry MeCN until the solid was completely dissolved. Slow evaporation of this solution gave colourless crystals.

Crystallography

All single crystals were sealed under N_2 and immersed in Paraton-N oil in a thin-walled glass capillary. Data were collected on a MSC/Rigaku RAXIS-IIC imaging plate for compound **6** using Mo-Kα radiation (λ 0.710 73 Å) from a Rigaku rotating-anode X-ray generator operating at 50 kV and 90 mA. An absorption correction was applied by correlation of symmetry-equivalent reflections using the ABSCOR program.[26] A Siemens RA or P4 diffractometer equipped with a LT-2 variable-temperature accessory was used for the data collection of **7** and **8**, using Cu-Kα (λ 1.541 78 Å) or Mo-Kα radiation, respectively. These two data sets were corrected for absorption using an empirical ψ-scan method. All three structures were solved by direct methods and subsequent Fourier-difference techniques, and refined anisotropically for all non-hydrogen atoms in **6** and Ag, halogen and C atoms in both **7** and **8**, by full-matrix least squares, on F using the SHELXTL PLUS program package (personal computer version).[27] All hydrogen atoms were geometrically fixed using the riding model. The direction of the polar axis in **6** was confirmed from the fact that the Rogers parameter η refined toward +1.[28] Crystal data and details of data collection and structure refinement are given in Table 3.

Atomic coordinates, thermal parameters, and bond lengths and angles have been deposited at the Cambridge Crystallographic Data Centre (CCDC). See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1997, Issue 1. Any request to the CCDC for this material should quote the full literature citation and the reference number 186/401.

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