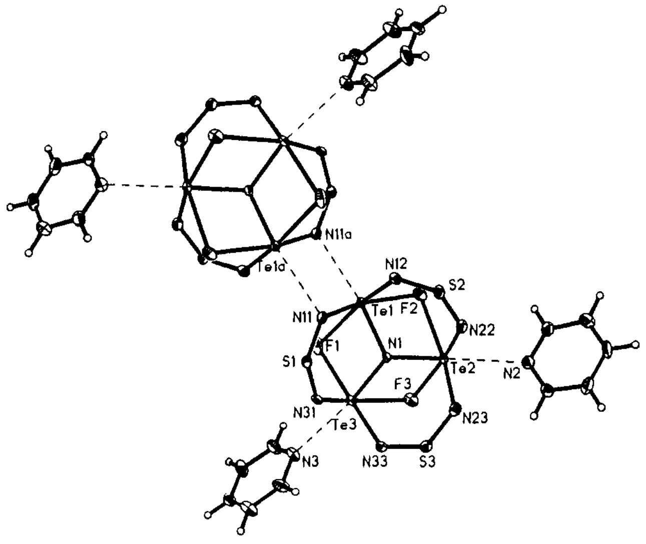

Figure 1. Structure of 2 showing 50% probability thermal motion ellipsoids. Selected mean bond distances (Å) are as follows: Te–N (NSN) (mean) = 2.048 (3), Te–N(1) (mean) = 2.031 (3), Te–F (mean) = 2.331 (3). The nitride N(1) atom is situated about 0.46 Å above the plane built by the three Te atoms.

fast exchange reactions in solution.

The deviation of the calculated elemental analysis from the values required is due to hydrolytic decomposition of compound **2**.

Although mild ionization methods (field desorption) were used, no fragments related to **2** could be found in the mass spectrum. A peak observed at m/z 256 can be attributed to S_8. Whether this peak is due to sulfur formation in the reaction or due to fragmentation of the molecule in the mass spectrum is unclear. The only byproduct detectable by ^1H NMR spectroscopy is Me_3SiF. No mechanism for the formation of **2** can be given. However, cleavage of S–N bonds is a well-known possibility in reactions of sulfur diimides.[2,9]

Figure 1 shows the content of the unit cell. Atomic parameters are given in Table I. In the crystalline state two molecules of **2** are connected by two intermolecular coordinations between N and Te atoms with Te- - -N distances of 2.955 Å. The distorted-octahedral coordination sphere is completed by short contacts to pyridine molecules. The bond distances do not differ significantly from those in the analogous chloride $(ClTeNSN)_3N$. Te–F bonds are significantly longer than the bridging and nonbridging Te–F bonds in tellurium(IV) fluoride (2.331 compared to 2.17 or 1.86 Å, respectively).[10] A structure similar to **2** and $(ClTeNSN)_3N$ is found in the phosphazene $(ClPNPCl_2N)_3N$.[11]

Compound **2** is the second structurally characterized tellurium nitride. Binary nitrides of tellurium are known from the literature, but they are—due to their violently explosive nature—only poorly characterized.[12,13] $(ClTeNSN)_3N$ and **2** are rather stable molecules, except that **2** may decompose violently under mechanical strain. The surprising kinetic stability of these two products can be explained with the steric shielding of the nitride N(1) atom by the sulfur diimide units on the one side and the halogen atoms on the other side of the molecules. Although a brown solid was obtained from a reaction involving tellurium(IV) fluoride and **1** in 1:4 molar ratio in THF, no information with respect to its identity could be obtained since it was found to explode violently on treatment with a spatula.

Table I. Atomic Parameters ($\times 10^4$) and Equivalent Isotropic Temperature Factors ($Å^2 \times 10^3$) for **2**[a]

	x	y	z	U(eq)
Te(1)	5385 (1)	1726 (1)	5383 (1)	12 (1)
Te(2)	7971 (1)	3085 (1)	6146 (1)	12 (1)
Te(3)	6985 (1)	4105 (1)	3334 (1)	12 (1)
S(1)	7212 (1)	904 (1)	3197 (1)	14 (1)
S(2)	8257 (1)	−439 (1)	7186 (1)	17 (1)
S(3)	10606 (1)	3320 (1)	4161 (1)	18 (1)
F(1)	4600 (3)	3628 (2)	3618 (2)	23 (1)
F(2)	5522 (3)	2576 (2)	6832 (2)	21 (1)
F(3)	7241 (3)	5259 (2)	4645 (2)	20 (1)
N(1)	7221 (3)	2552 (3)	4940 (2)	11 (1)
N(11)	6236 (3)	496 (3)	4296 (2)	15 (1)
N(12)	6777 (4)	−232 (3)	6547 (2)	17 (1)
N(22)	8929 (3)	804 (3)	7070 (2)	16 (1)
N(23)	10187 (3)	2886 (3)	5425 (3)	18 (1)
N(31)	7610 (3)	2372 (3)	2697 (2)	14 (1)
N(33)	9415 (3)	3803 (3)	3191 (2)	17 (1)
N(2)	10081 (4)	2638 (3)	7877 (3)	21 (1)
C(1)	9565 (5)	2213 (5)	8938 (3)	28 (2)
C(2)	10587 (6)	1476 (5)	9888 (3)	33 (2)
C(3)	12196 (5)	1167 (5)	9722 (3)	31 (2)
C(4)	12793 (5)	1589 (4)	8618 (3)	26 (2)
C(5)	11677 (5)	2337 (4)	7728 (3)	21 (1)
N(3)	6828 (4)	5376 (3)	1069 (3)	21 (1)
C(6)	7964 (5)	5031 (4)	316 (3)	27 (1)
C(7)	7698 (5)	5699 (5)	−838 (3)	31 (2)
C(8)	6208 (5)	6756 (4)	−1242 (3)	26 (2)
C(9)	5002 (5)	7110 (5)	−460 (3)	30 (2)
C(10)	5387 (5)	6408 (4)	682 (3)	26 (1)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Attempts to generate an analogous bromide to **2** however were unsuccessful. Tellurium(IV) bromide does not react either with **1** or sulfur N,N'-bis(trimethylstannyl)diimide under similar conditions.

Other Te compounds containing sulfur diimide units are known from the literature,[14-16] but except $(ClTeNSN)_3N$ no other compound containing a nitride N atom has been reported. Compound **2** is one of the first fluorides of tellurium(IV) to be structurally characterized.

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Registry No. 1, 18156-25-7; 2, 140874-49-3; 2·2(pyridine), 140874-50-6.

Supplementary Material Available: Listings of positional parameters, anisotropic displacement parameters, and bond distances and angles for $(FTeNSN)_3N$ (3 pages); a listing of structure factor amplitudes for $(FTeNSN)_3N$ (14 pages). Ordering information is given on any current masthead page.

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A Chloroalkane–Silver Complex with a Monodentate RCl→Ag(I) Interaction

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Introduction

During the last 4 years a number of metal complexes containing chloroalkane ligands have been prepared and structurally char-

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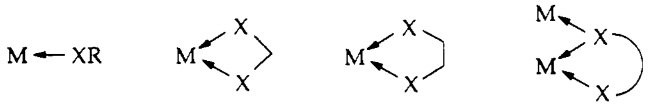
Table I. Crystallographic Data for Ag(1,2,3-$C_3H_5Cl_3$)$OTeF_5$

chem formula	$C_3H_5AgCl_3F_5OTe$
fw	493.9
space group	$P\bar{1}$ (No. 2)
unit cell dimens	
a	7.823 (4) Å
b	8.044 (4) Å
c	9.217 (4) Å
α	75.18 (4)°
β	78.12 (3)°
γ	79.33 (4)°
unit cell vol	538 (1) $Å^3$
Z	2
calcd density	3.05 g cm^{-3}
T	−125 (1) °C
radiation (λ)	Mo Kα (0.7107 Å)
μ	53.5 cm^{-1}
R $((\sum\|(F_o - F_c)\|)/(\sum F_o))$	0.032
R_w $((\sum w^{1/2}\|(F_o - F_c)\|)/(\sum w^{1/2}F_o))$	0.068

acterized.[1-3] In these complexes of Tl(I),[1a] Ag(I),[1b-d] and Ru(II),[2] the chloroalkane ligand has been either dichloromethane or 1,2-dichloroethane. These new complexes have demonstrated the existence of intermolecular $RCl \rightarrow ML_n$ bonds in condensed phases and have helped dispel the notion of the noncoordinating solvent.

Halocarbon–metal bonds have long been suspected[4] to be important in organometallic chemistry (for example, in oxidative addition[5]) and in biochemistry (in enzyme inhibition[6]). Their existence now firmly established, one can reasonably expect new theoretical treatments of halocarbon–metal bonding and reactivity to be published in the near future. As in one such recent study of iodoalkane–rhenium bonding,[7] these will undoubtedly rely on available structural data.

The bonding modes shown in II–IV are well established for more traditional ligands (e.g., X = O^-, OR, N^{2-}, NR^-, NR_2, S^-, SR, PR^-, PR_2, etc.) and have also been observed in chloroalkane



I II III IV

complexes (X = Cl).[1,2] From a theoretical standpoint, it is unfortunate that structure I has not yet been reported for X = Cl,

Table II. Atomic Coordinates (×10^4) and Isotropic Thermal Parameters ($Å^2$ × 10^3) for Ag(1,2,3-$C_3H_5Cl_3$)$OTeF_5$

	x	*y*	*z*	U^a
Ag	1091 (1)	3725 (1)	3640 (1)	23 (1)
Te	1309 (1)	8434 (1)	2539 (1)	17 (1)
F1	1179 (5)	7248 (4)	1088 (4)	29 (1)
F2	3762 (4)	7957 (5)	2148 (5)	38 (1)
F3	1535 (5)	9928 (4)	3723 (4)	37 (1)
F4	−1097 (5)	9253 (5)	2670 (5)	36 (1)
F5	1577 (5)	10307 (5)	915 (4)	34 (1)
O1	1063 (5)	6619 (5)	4104 (4)	25 (1)
Cl1	−1469 (2)	4237 (2)	1884 (2)	31 (1)
Cl2	−5933 (2)	2231 (2)	4975 (1)	23 (1)
Cl3	−6278 (2)	3428 (2)	1266 (2)	26 (1)
C1	−3534 (7)	4120 (7)	3190 (7)	26 (2)
C2	−4070 (7)	2371 (7)	3417 (6)	21 (2)
C3	−4528 (7)	1914 (7)	2061 (6)	22 (2)

[a] The equivalent isotropic *U* is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

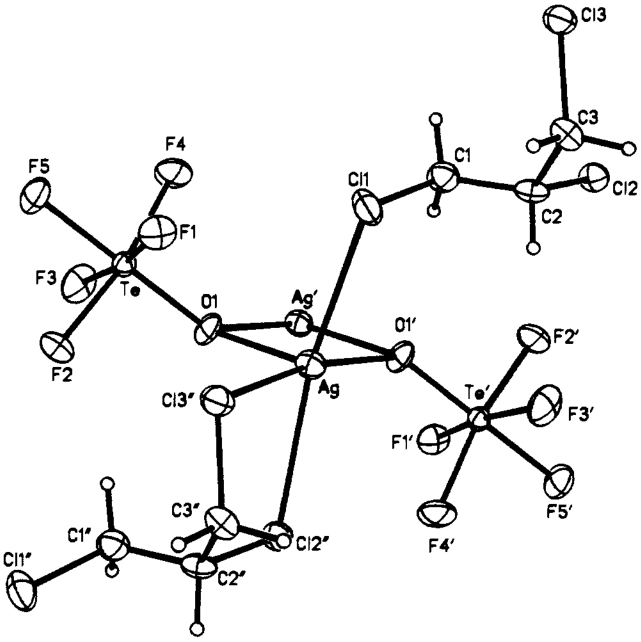


Figure 1. Part of the infinite chain of centrosymmetric [Ag(1,2,3-$C_3H_5Cl_3$)$OTeF_5$]$_2$ dimeric moieties (50% ellipsoids except for hydrogen atoms, which are drawn as unlabeled spheres of arbitrary radius). The chloroalkane ligands coordinated to Ag′ have been omitted for clarity. Selected distances (Å) and angles (deg): Ag–Cl1, 2.722 (2); Ag–Cl2″, 2.770 (1); Ag–Cl3″, 2.698 (1); C1–Cl1, 1.802 (5); C2–Cl2, 1.816 (5); C3–Cl3, 1.788 (5); Ag–Cl1–C1, 105.9 (2); Ag–Cl2″–C2″, 105.6 (2); Ag–Cl3″–C3″, 105.5 (2); O1–Ag–O1′, 84.0 (1); O1–Ag–Cl3″, 105.6 (1); O1–Ag–Cl2″, 94.2 (1); O1–Ag–Cl1, 104.3 (1); O1′–Ag–Cl3″, 168.6 (1); Cl1–Ag–Cl2″, 160.3 (1); Cl1–Ag–Cl3″, 92.5 (1); Cl2″–Ag–Cl3″, 75.6 (1).

since one would like to compare theoretical M–ClR distances and M–Cl–C angles with experimental values that are not constrained by chelation or by bridging. In an earlier study, the compound $AgOTeF_5$ dissolved without further reaction in (potentially) bidentate solvents such as dichloromethane and 1,2-dichloroethane but formed AgCl and other (organic) products with chloroalkanes that might have afforded simple, monodentate $RCl \rightarrow Ag(I)$ bonds, including 1-chlorobutane and 1-chloro-2,2,2-trifluoroethane.[1b] However, the first example of structure I has now been discovered in the new compound Ag(1,2,3-$C_3H_5Cl_3$)$OTeF_5$, which contains a monodentate chloroalkane–silver interaction.

Experimental Section

Preparation of Ag(1,2,3-$C_3H_5Cl_3$)$OTeF_5$. The moisture-sensitive compound $AgOTeF_5$[1a,8] was dissolved in 1,2,3-trichloropropane (mp −14

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Table III. Comparison of Ag–Cl Distances (Å), Ag–Cl–C Angles (deg), and C–Cl Distances (Å) for Monodentate (Ag–ClR) and Bidentate ($Ag–Cl–CHR–CH_2–Cl$) Chloroalkane–Silver Complexes

compd	R	Ag–Cl	Ag–Cl–C	C–Cl
	Monodentate			
$Ag(1,2,3\text{-}C_3H_5Cl_3)$-$OTeF_5$[a]	CH_2CH-$ClCH_2Cl$	2.722 (2)	105.9 (2)	1.802 (5)
	Bidentate			
$Ag(1,2,3\text{-}C_3H_5Cl_3)$-$OTeF_5$[a]	CH_2Cl	2.698 (1)	105.5 (2)	1.788 (5)
		2.770 (1)	105.6 (2)	1.816 (5)
$Ag(1,2\text{-}C_2H_4Cl_2)OTeF_5$[b]	H	2.626 (3)	107.8 (3)	1.80 (1)
		2.640 (3)	105.0 (4)	1.81 (1)
		2.705 (3)	97.2 (4)	1.79 (1)
$Ag_2(1,2\text{-}C_2H_4Cl_2)_4Pd$-$(OTeF_5)_4$[c]	H	2.645 (1)	108.4 (2)	1.763 (4)
		2.655 (1)	99.6 (2)	1.793 (3)
		2.840 (1)	104.3 (1)	1.791 (5)
		2.928 (1)	96.7 (1)	1.795 (5)

[a] This work. [b] References 1b and 1d. [c] Reference 1b.

°C, bp 156 °C) that had been purified by fractional distillation. The resulting mixture was filtered. After hexanes (20% by volume) were layered on top of the solution, it was stored at −20 °C for 4 h, at which time a crop of well-formed crystals had appeared.

Crystallographic Study. A Siemens R3m diffractometer equipped with an LT-1 variable-temperature accessory was used. Crystals of $Ag(1,2,3\text{-}C_3H_5Cl_3)OTeF_5$ were examined under argon at −18 °C using an apparatus previously described.[1b] A suitable crystal was embedded in Halocarbon 25-5S grease at the end of a glass fiber and quickly placed in the cold nitrogen stream of the LT-1 unit. Centering of 24 reflections allowed least-squares calculation[9] of the cell constants given in Table I, which also contains other experimental parameters. The intensities of control reflections 100, 020, and 001, monitored every 97 reflections, showed no significant trend during the course of the data collection. An empirical absorption correction, based on intensity profiles for 16 reflections over a range of setting angles (ψ) for the diffraction vector, was applied to the observed data. Transmission factors ranged from 0.702 to 0.853. Lorentz and polarization corrections were applied to the data.

The structure was solved by interpretation of the Patterson map.[9] Final refinement involved anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were included in calculated positions (C–H = 0.96 Å, $U(H) = 1.2\ U_{iso}(C)$). Neutral-atom scattering factors (including anomalous scattering) were taken from ref 10. The weighted (weights calculated as $(\sigma^2(F) + |g|F_o^2)^{-1}$) least-squares refinement on F converged (average shift/esd = 0.005 over the last two refinement cycles). In the final difference Fourier maps, the maximum and minimum electron densities were 0.71 e $Å^{-3}$ (0.87 Å from Ag) and −0.98 e $Å^{-3}$. Analysis of variance as a function of Bragg angle, magnitude of F_o, reflection indices, etc. revealed no significant trends. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table II.

Results and Discussion

The structure of $Ag(1,2,3\text{-}C_3H_5Cl_3)OTeF_5$, shown in Figure 1, consists of chains in which centrosymmetric $Ag_2(\mu\text{-}OTeF_5)_2$ moieties are linked by 1,2,3-trichloropropane ligands via Ag–Cl bonds. Each Ag(I) ion is coordinated in monodentate fashion by Cl1 of one 1,2,3-trichloropropane molecule and in bidentate fashion by Cl2 and Cl3 of another. The latter interactions give rise to five-membered $Ag–Cl–CHR–CH_2–Cl$ chelate rings (R = CH_2Cl), similar in many respects to those observed in $Ag(1,2\text{-}C_2H_4Cl_2)OTeF_5$[1b,d] and in $Ag_2(1,2\text{-}C_2H_4Cl_2)_4Pd(OTeF_5)_4$[1b] (R = H).

The chains of dimeric $Ag_2(1,2,3\text{-}C_3H_5Cl_3)_2(OTeF_5)_2$ moieties are cross-linked by weak, secondary Ag–F3 interactions of 2.986 (4) Å, which are close to the expected van der Waals limit for silver and fluorine of 3.15 ± 0.08 Å.[11] The pseudooctahedral AgO_2Cl_3F coordination sphere is shown in Figure S-1 (supplementary material). The atom next closest to Ag is F1, which, at 3.179 (3) Å, is clearly not bonded to silver. For comparison, the Ag–F distances in $AgSbF_6$ are ~2.62 Å.[12]

The most significant feature of this structure is the presence of an example of the previously unknown monodentate $RCl\rightarrow M$ interaction. The results, listed in Table III, demonstrate surprisingly that the Ag–Cl distance and the Ag–Cl–C angle for an unconstrained (i.e., monodentate, nonbridging) $RCl\rightarrow Ag(I)$ interaction are within the range of values observed for bidentate $Ag–Cl–CHR–CH_2–Cl$ moieties. For example, the monodentate Ag–Cl bond distance in $Ag(1,2,3\text{-}C_3H_5Cl_3)OTeF_5$, 2.722 (2) Å, lies between the bidentate Ag–Cl bond distances in this compound, 2.698 (1) and 2.770 (1) Å. The monodentate Ag–Cl–C bond angle, 105.9 (2)°, is the same (within experimental error) as the bidentate Ag–Cl–C angles in $Ag(1,2,3\text{-}C_3H_5Cl_3)OTeF_5$, 105.5 (2) and 105.6 (2)°. Furthermore, when bond distances and angles in the compounds $Ag(1,2\text{-}C_2H_4Cl_2)OTeF_5$[1b,d] and $Ag_2(1,2\text{-}C_2H_4Cl_2)_4Pd(OTeF_5)_4$[1b] are included in the comparison, the ranges of the relevant distances and angles for bidentate 1,2-dichloroalkane ligands become 2.626 (3)–2.928 (1) Å and 96.7 (1)–108.4 (2)°, respectively.

In five-membered chelate rings, Ag–Cl–C bond angles cannot be much greater than ~110° without either lengthening of the Ag–Cl bonds or drawing together the chelate ring chlorine atoms (note that Cl2···Cl3 is 3.352 (2) Å, less than the van der Waals limit of ~3.6 Å[11]). The monodentate Ag–Cl–C bond angle of ~106° observed here bears on the nature of $RCl\rightarrow Ag$ bonds in particular and $RCl\rightarrow M$ bonds in general. Potentially these bonds can vary from being largely covalent to being largely ion–dipolar in nature.

In potassium tetrafluorophthalate, a fluorine atom coordinates to the metal without being part of a chelate ring, and the resulting K–F–C bond angle is 161 (1)°.[13] A large angle such as this is apparently dictated by electronic and not steric forces (i.e., the interaction is largely ion–dipole). A theoretical study predicted that $[Li–F–CH_3]^+$ and $[Na–F–CH_3]^+$ bond angles should both be 180° and that $[Li–Cl–CH_3]^+$ and $[Na–Cl–CH_3]^+$ bond angles should be 138 and 157°, respectively.[14] Clearly, these results, involving hard metal ions, do not provide relevant benchmarks for complexes in which the M–XR bonds are expected to have significant covalent character. In four complexes containing iodocarbons bound in monodentate fashion to soft metal ions, M–I–C angles were found to be 106.9 (8)° (M = Ir(III); average of two values),[3h] 102.5 (5)° (M = Re(I)),[3d] 101.8 (2)° (M = Ru(II)),[3a] and 104.9 (7)° (M = Ru(II)).[3c] These results are in harmony with a theoretical study[15] which predicted an angle of 100° for a $CH_3–I–Pt(II)$ linkage (the recent theoretical study[7] of $RI\rightarrow Re(I)$ bonding mentioned earlier used interatomic distances and angles from an X-ray crystallographic study and did not allow the geometries to vary). The relative affinities of metal ions for haloalkanes also depend on whether the metal ion is hard or soft—the available data suggest that the trend in bond strengths is $RI–M^{n+} > RBr–M^{n+} > RCl–M^{n+}$ for the soft metal centers Ir(III), Re(I), and Ru(II).[3] However, for the hard metal center Li(I), gas-phase binding energies follow the order $RF–Li^+ > RCl–Li^+ \sim RI–Li^+$.[16]

In conclusion, structural results for a monodentate interaction between a chloroalkane and the soft metal ion Ag(I) suggest that this interaction is significantly covalent with perhaps only a minor ion–dipole component. Forthcoming papers from this laboratory will deal with reactions of haloalkanes with metal ions, including

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(9) Calculations for diffractometer operations were performed by using software supplied with the Siemens R3m diffractometer. All structural calculations were performed on a Data General Eclipse S/140 computer in the X-ray laboratory at Colorado State University with the SHELXTL program library written by Professor G. M. Sheldrick and supplied by Siemens Analytical X-ray Corp.

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simple adduct formation as well as oxidative addition and halide abstraction.

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Supplementary Material Available: Figure S-1, showing the full Ag(I) ion coordination sphere, and Tables S-I–S-V, listing crystallographic data, bond distances, bond angles, anisotropic thermal parameters, and hydrogen atom coordinates and thermal parameters (4 pages); Table S-VI, listing observed and calculated structure factors (15 pages). Ordering information is given on any current masthead page.

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Synergism in a Transition Metal Cluster Compound. Crystal and Molecular Structure of a Polysilver Cluster Molecule with an Unusual Bridging Sulfur Atom, $Ag_{11}S(Et_2dtc)_9$

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One of our current research interests is to understand the synergistic effect in transition metal cluster compounds through the studies of synthetic routes, stereo- and electronic structures, and their chemical and physical properties.[1] Dialkyldithiocarbamates (R_2dtc^-) acting as either bridging or terminal ligands play an important role in modern coordination chemistry.[2–6] The dialkyldithiocarbamato molecules of the univalent coinage metals (Cu, Ag, and Au) form a class of compounds where steric restrictions strongly influence the crystal structures.[2] We had prepared the cubane-like structure compound of Mo(W)–Fe–R_2dtc and various structural types of Mo(W)–Cu–R_2dtc compounds.[3,4] In an attempt to prepare a heterometal Mo(W)–Ag cluster molecule similar to the Mo(W)–Cu–R_2dtc system, we obtained a polysilver compound whose molecular structure is completely different from that of silver(I) diethyldithiocarbamate or its β-modification.[2,5,6] Herein we report the synthesis and crystal structure of $Ag_{11}S(Et_2dtc)_9$.

Experimental Section

All operations were carried out under a pure dinitrogen atmosphere.

Preparation of $Ag_{11}S(Et_2dtc)_9$. A direct electrochemical synthetic method similar to that of Casey[7] was used to prepare the title compound. The crystal thus obtained could be used for the measurement of the crystal unit cell but was not suitable for the crystal structure determination.[8] A chemical synthetic method was then developed. A mixture of 0.34 g of $AgNO_3$ (2 mmol) and 0.45 g of $Na(Et_2dtc)\cdot 3H_2O$ (2 mmol) in 40 mL of DMF was added with $(NH_4)_2MoS_4$ (0.5 mmol) at 25 °C. After being stirred for 24 h, the dark red solution was filtered and the filtrate was diluted with 20 mL of acetone. The solution was kept at 4 °C for several days, and the orange prismlike crystals were collected,

Table I. Crystallographic Data for $Ag_{11}S(Et_2dtc)_9$

chem formula: $C_{45}H_{90}Ag_{11}N_9S_{19}$
fw = 2553.07
space group: *R3c*
$a = 18.661\ (6)$ Å
$c = 43.227\ (8)$ Å
$V = 13036$ Å^3
$Z = 6$
$T = 296$ K
$\lambda = 0.71069$ Å
$d_{calcd} = 1.951$ g cm^{-3}
$\mu(\text{Mo K}\alpha) = 28.726$ cm^{-1}
$R^a = 0.044$
$R_w{}^b = 0.045$

[a] $R = \sum(|F_o| - |F_c|)/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum(|F_o|)^2]^{1/2}$.

Table II. Atomic Coordinates and Thermal Parameters for $Ag_{11}S(S_2CNEt_2)_9$

atom	*x*	*y*	*z*	*B*,[a] Å^2
Ag(1)	0.02181 (8)	0.1512 (1)	0.0628 (0)	5.37 (4)
Ag(2)	−0.12086 (8)	0.03507 (8)	0.10469 (4)	5.00 (3)
Ag(3)	−0.1383 (1)	0.02824 (9)	0.03268 (4)	6.33 (4)
Ag(4)	0.0000 (0)	0.0000 (0)	0.13348 (7)	4.91 (5)
Ag(5)	0.0000 (0)	0.0000 (0)	0.01196 (7)	7.21 (6)
S(1)	0.0000 (0)	0.0000 (0)	0.0698 (2)	4.1 (1)
S(11)	0.1087 (3)	0.2255 (2)	0.1116 (1)	3.7 (1)
S(12)	−0.0286 (2)	0.1092 (2)	0.1548 (1)	3.6 (1)
S(21)	−0.0869 (3)	0.1759 (3)	0.0358 (1)	4.7 (1)
S(22)	−0.1854 (3)	0.1222 (3)	0.0954 (1)	5.5 (2)
S(31)	0.1459 (3)	0.2591 (3)	0.0268 (1)	6.0 (1)
S(32)	−0.1312 (3)	−0.0440 (3)	−0.0234 (1)	6.5 (1)
N(10)	0.0194 (7)	0.2689 (7)	0.1467 (3)	3.9 (3)
N(20)	−0.1647 (8)	0.2461 (7)	0.0597 (4)	4.8 (4)
N(30)	0.091 (1)	0.285 (1)	−0.0251 (4)	11.2 (6)
C(10)	0.0310 (8)	0.2049 (8)	0.1385 (3)	3.1 (3)
C(11)	−0.051 (1)	0.258 (1)	0.1647 (4)	6.6 (6)
C(12)	−0.028 (1)	0.271 (1)	0.1981 (5)	7.8 (8)
C(13)	0.072 (1)	0.3534 (8)	0.1353 (4)	4.0 (4)
C(14)	0.040 (1)	0.367 (1)	0.1046 (5)	6.1 (6)
C(20)	−0.1483 (9)	0.1850 (8)	0.0643 (4)	3.7 (4)
C(21)	−0.146 (1)	0.294 (1)	0.0301 (4)	5.8 (5)
C(22)	−0.060 (2)	0.372 (1)	0.0333 (6)	8.2 (8)
C(23)	−0.209 (1)	0.270 (1)	0.0846 (5)	7.4 (7)
C(24)	−0.295 (1)	0.237 (2)	0.0812 (7)	9.5 (9)
C(30)	−0.224 (1)	−0.124 (1)	−0.0095 (4)	8.0 (6)
C(31)	0.057 (2)	0.271 (3)	−0.0599 (8)	17 (2)
C(32)	−0.028 (2)	0.219 (3)	−0.0604 (9)	22 (2)
C(33)	0.098 (2)	0.358 (1)	−0.0157 (9)	17.9 (9)
C(34)	0.187 (2)	0.420 (2)	−0.026 (1)	23 (1)

[a] Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $(4/3)\ [a^2(B(1,1)) + b^2(B(2,2)) + c^2(B(3,3)) + ab(\cos\gamma)(B(1,2)) + ac(\cos\beta)(B(1,3)) + bc(\cos\alpha)(B(2,3))]$.

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