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Crystal and Molecular Structure of Bis[2-(aminocarbonyl)anilinium] Dimethyltetrachlorostannate(IV) Dihydrate at 138 K: A Hydrogen-Bonded Network Lattice

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Bis[2-(aminocarbonyl)anilinium] dimethyltetrachlorostannate(IV) dihydrate, $C_{16}H_{28}N_4O_4SnCl_4$, forms triclinic crystals (mp 98–99 °C) in the triclinic space group $P\bar{1}$ with $a = 7.389$ (3) Å, $b = 11.965$ (5) Å, $c = 6.995$ (2) Å, $\alpha = 102.29$ (3)°, $\beta = 90.87$ (3)°, $\gamma = 84.74$ (4)°, $V = 601.56$ Å^3, $Z = 1$, and $\rho_{calcd} = 1.66$ g cm^{-3}. The structure was solved by the heavy-atom method and refined to an R value of 0.019 for the 1662 reflections collected at 138 ± 2 K with the use of Mo K$\bar{\alpha}$ radiation. The structure is centrosymmetric at the *trans*-dimethylstannate(IV) octahedral units, which are held by a network of hydrogen bonds into a three-dimensional, associated lattice. The equivalent water molecules of the dihydrate are three-coordinated, the two different trans pairs of chlorine atoms are two- and three-coordinated, and the amido nitrogen of the benzamido unit is three-coordinated through hydrogen bonding, which symmetrically dimerizes the benzamide units and internally bonds the anthranilamide units. The hydrogen-bonding network effectively fills space while delocalizing the anilinium cationic and dimethyltetrachlorotin(IV) dianionic charges through the lattice.

We are pursuing a new-found interest in the fate of organotin(IV) species in water. Knowledge of aqueous organotin(IV) chemistry has bearing on environmental questions concerning the commercial use of organotin biocides[1-3] in agriculture and marine antifouling applications. The structures of organotin(IV) hydrates and hydrolysates are largely unknown.[5]

Thus, we were interested in the title compound, which is the fortuitous product of the action of anthranilamide with dimethyltin(IV) dichloride in chloroform. Its structure, reported here, helps to elucidate the interaction of polychloroorganotin(IV) anions with the water medium.

Experimental Section

Synthesis of Bis[2-(aminocarbonyl)anilinium] Dimethyltetrachlorostannate(IV) Dihydrate, $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$. To a chloroform solution (50 mL) of dimethyltin(IV) dichloride (13.18 g, 30.00 mmol) was added anthranilamide (8.16 g, 30.0 mmol) and the mixture refluxed for 1 h and filtered to form a clear solution, which after cooling overnight gave the product as tan crystals (8.01 g, 45.4%). Anal. Calcd for $C_{16}H_2N_4O_4SnCl_4$: C, 31.97; H, 4.66; Sn, 19.77. Found: C, 29.30; H, 3.98; Sn, 19.01. Physical and spectroscopic data are listed in Table I.

Crystal Data. Tan crystals of the title compound were obtained by slow evaporation of a chloroform solution. A prismatic crystal of dimensions 0.38 × 0.14 × 0.15 mm was used for the determination of the cell parameters and subsequent data collection.

The space group was determined by diffractometry studies to be triclinic, $P\bar{1}$. The cell dimensions were determined by least squares from the $+2\theta$ and -2θ values of 48 reflections spaced throughout reciprocal space. All measurements were performed on an Enraf-Nonius CAD-4 automatic counter diffractometer controlled by a PDP8/e computer and fitted with a low-temperature device. Crystal data are listed in Table II.

Data Collection and Structure Determination. The data collection parameters are given in Table III. The intensities were corrected for Lorentz and polarization effects, but not for absorption. The ratio of the maximum and minimum transmission factors is 1.53.

Table I. Physical and Spectroscopic Properties of $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$

mp: 98–99 °C

^{119}Sn Mössbauer (77 K), mm s^{-1}: IS = 1.19 ± 0.03, QS = 4.09 ± 0.06, $\Gamma_1 = 1.02 \pm 0.03$, $\Gamma_2 = 1.03 \pm 0.03$ (ρ = QS/IS = 3.44)

infrared (Nujol/CsI), 2000–200 cm^{-1}: 1965 vw, 1650 w, 1608 w, 1446 s, 1366 m, 1153 vw, 1057 vw, 800 w, 744 w, 550 w, 474 vw, 373 w, 315 vw, 240 vw

NMR (CD_3OD): ^{1}H δ H_3CSn 1.13 ($|^2J(^{119}\text{Sn–C–}^1\text{H})| = 92.80$ Hz), δ $H_3NC_6\mathit{H}_4C(O)NH_2$ 7.62–7.93, δ $\mathit{H}_3NC_6H_4C(O)N\mathit{H}_2\cdot 2\mathit{H}_2O$ 4.95 (integration C_6H_4:$(C\mathit{H}_3)_2$Sn calcd 4:6, found 4:5); ^{13}C δ $H_3\mathit{C}$Sn 13.28 ($|^1J(^{119}\text{Sn–}^{13}\text{C})| = 92$ Hz), δ $H_3NC_6H_4\mathit{C}(O)NH_2$ 181.29, δ $H_3N\mathit{C}_6H_4C(O)NH_2$ 113.00, 116.31, 117.79, 118.72, 129.73, 133.77

mass spectrum (12 eV), *m/e* (fragment, rel abund): 369 ($[HNC_6H_4C(O)NH_2O(CH_3)_2SnCl_2]^+$, 10.6), 220 ($[(CH_3)_2SnCl_2]^{+\cdot}$, 14.6), 205 ($[CH_3SnCl_2]^+$, 100.0), 185 ($[CH_3)_2SnCl]^+$, 19.3), 136 ($[H_2NC_6H_4C(O)NH_2]^{+\cdot}$, 90.0), 120 ($Sn^{+\cdot}$, 33.0)

Table II. Crystal Data for $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(\mu\text{-}H_2O)_2$

formula	$C_{16}H_{28}N_4O_4SnCl_4$	V, Å^3	601.7
fw	600.5	space group	$P\bar{1}$
cryst system	triclinic	Z	1
a, Å	7.389 (3)[a]	$F(000)$	308.00
b, Å	11.965 (5)	ρ_{calcd}, g cm^{-3}	1.657
c, Å	6.995 (2)	μ, cm^{-1}	14.1
α, deg	102.29 (3)		
β, deg	90.87 (3)		
γ, deg	84.74 (4)		

[a] Estimated standard deviations in parentheses.

The structure was determined by the heavy-atom method and refined by a full-matrix least-squares program.[6] All the hydrogen atoms in the structure including the two belonging to water oxygen [O(1)] were located from a difference Fourier map. Final refinements were carried out with use of anisotropic thermal parameters for all the non-hydrogen atoms and isotropic thermal parameters for the hydrogen atoms. Anomalous dispersion corrections for Sn and Cl were included in the refinement, which was discontinued when the maximum parameter shift was less than 0.1 of the corresponding standard deviation. The final R value is 0.019, and R_w is 0.025 for

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Table III. Data Collection Parameters for $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$

diffractometer	Enraf-Nonius CAD-4
radiation	Mo K$\bar{\alpha}$ (λ = 0.71069 Å) graphite monochromator
temp, K	138 ± 2
scan technique	θ–2θ
2θ limit, deg	$0 < 2\theta \leqslant 46$
scan time, s	45
scan angle, deg	$(1.00 + 0.20 \tan \theta)$
aperture width, mm	$(4.00 + 0.86 \tan \theta)$
aperture height, mm	6
aperture distance, mm	173
intensity monitor, s	7200
max fluctuation	2%
orientation monitors	200 measurements[a]
no. of unique data	1662
no. of obsd data ($I > 2\sigma(I)$)	1610

[a] A new orientation matrix was obtained if an angular change in the control reflections was more than 0.1°.

Table IV. Final Positional Parameters (×10⁴)[a] for $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$

atom	x	y	z
Sn	5000 (0)	5000 (0)	5000 (0)
Cl(1)	4242 (1)	6976.9 (4)	4037 (1)
Cl(2)	8075 (1)	4784.5 (4)	3086 (1)
N(1)	8252 (3)	7283 (2)	2136 (3)
N(2)	6156 (3)	10688 (2)	2308 (3)
O(1)	8395 (2)	2753 (2)	−556 (3)
O(2)	6094 (2)	8826 (1)	910 (2)
C(1)	6298 (4)	5812 (2)	7579 (4)
C(2)	8318 (3)	8159 (2)	3956 (3)
C(3)	9228 (3)	7847 (2)	5524 (3)
C(4)	9332 (3)	8653 (2)	7256 (3)
C(5)	8538 (3)	9761 (2)	7392 (3)
C(6)	7663 (3)	10062 (2)	5794 (3)
C(7)	7531 (3)	9269 (2)	4040 (3)
C(8)	6537 (3)	9591 (2)	2311 (3)

[a] Estimated standard deviations for last digit in parentheses.

Table VI. Hydrogen-Bond Parameters in $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$[a]

D	A	D···A, Å	D-H, Å	H···A, Å	D-H-A, deg
N(1)	Cl(2)	3.211 (2)	0.80 (3)	2.42 (3)	170 (2)
N(1)	O(2)	2.603 (3)	0.88 (3)	1.82 (3)	147 (3)
N(1)	O(1)[b]	2.721 (3)	1.05 (3)	1.67 (3)	174 (2)
N(2)	O(2)[c]	2.904 (2)	0.78 (2)	2.13 (2)	179 (2)
O(1)	Cl(1)[d]	3.147 (2)	0.82 (3)	2.33 (3)	173 (3)
O(1)	Cl(2)	3.119 (2)	0.74 (4)	2.38 (4)	170 (3)

[a] Estimated standard deviations for the last digit in parentheses. [b] $(2 - x, 1 - y, -z)$. [c] $(1 - x, 2 - y, -z)$. [d] $(1 - x, 1 - y, -z)$.

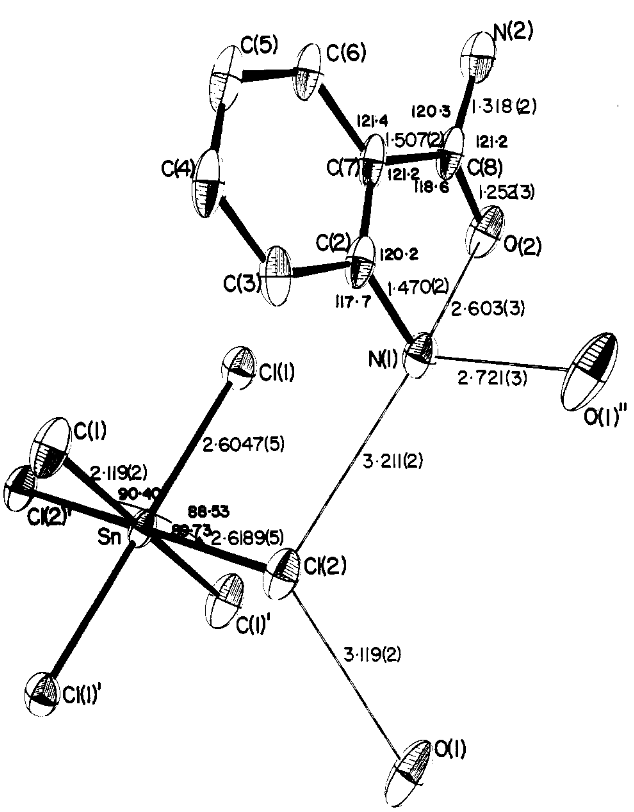

Figure 1. ORTEP plot (20% probability thermal ellipsoids) showing atom numbering and selected bond lengths and angles. Dashed lines indicate hydrogen bonds. Standard deviations of bond angles: Cl–Sn–Cl, 0.02°; C–Sn–C, 0.07°; and all others 0.2°.

Table VII. Hydrogen-Bonding Distances in Sn-Cl- - -H-N Systems in Tin(IV) Salts and Adducts

compd	d(Cl- - -H-N), Å	ref
$[NH_4]^+{}_2[SnCl_6]^{2-}$	3.552, 3.550	10 11
$[4\text{-}ClC_5H_5NH]_2{}^+[SnCl_6]^{2-}$	3.244, 3.344	12
$[C_5H_5NH]_2{}^+[(CH_3)_2SnCl_4]^{2-}$	3.243, 3.312	9
$[2,3\text{-}(CH_3)_2pyr]_2(CH_3)_2SnCl_2$[b]	3.53, 3.73	14
$[quin\text{-}H]^+[(CH_3)_2SnCl_3]^-$[c]	3.18	13
$[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2\text{-}[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$	3.211 (2)	a

[a] This work. [b] pyr = pyrazole. [c] quin = quinoline.

Table VIII. Tin-119m Mössbauer Parameters for $[R_2SnCl_4]^{2-}$ Salts and Predicted C-Sn-C Angles

compd	IS, mm/s	QS, mm/s	predicted C–Sn–C, deg
$[(CH_3)_2SnCl_4]^{2-}Cs^+{}_2$	1.63,[a] 1.47[b,c]	4.32,[a] 4.28[b,c]	180
$[(CH_3)_2SnCl_4]^{2-}[C_5H_5NH]^+{}_2$	1.59[d]	4.32[d]	180
$[(C_2H_3)_2SnCl_4]^{2-}[(CH_3)_4N]^+{}_2$	1.38[e]	3.90[e]	158
$[(C_2H_5)_2SnCl_4]^{2-}[(CH_3)_4N]^+{}_2$	1.64[a]	3.99[a]	163
$[(C_6H_5)_2SnCl_4]^{2-}[C_5H_5NH]^+{}_2$	1.44[d]	3.80[d]	180
$[(CH_3)_2SnCl_4]^{2-}\text{-}[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2(H_2O)_2$	1.19[f]	4.09[f]	172

[a] Reference 23. [b] Measured at 4.2 K. [c] Reference 24. [d] Reference 25. [e] Reference 26. [f] This work.

189 parameters and all 1662 reflections (S = 1.14). The final difference map was featureless with peaks ranging between +0.3 and −0.3 e Å^{-3}. The scattering factors and anomalous dispersion corrections for all non-hydrogen atoms were taken from ref 7 and scattering factors for the hydrogen atoms from ref 8.

The final positional and thermal parameters of non-hydrogen atoms are listed in Tables IV and IX, respectively. The atom-numbering schemes followed in these listings are identified in Figure 1. Selected bond distances and angles are also shown in Figure 1. Figure 2 shows a stereoview of the unit cell contents. The C–C distances and C–C–C angles in the phenyl rings are listed in Table V (supplementary material). Hydrogen-bond parameters are listed in Table VI. Table X lists hydrogen positional parameters and Table XI the least-squares planes (supplementary material). The tin-119m Mössbauer parameters for $[R_2SnCl_4]^{2-}$ salts with the predicted C–Sn–C angles are listed in Table VIII.

Results and Discussion

Adventitious moisture hydrolyzes dimethyltin(IV) dichloride to the oxide, liberating HCl,

$$(CH_3)_2SnCl_2 + H_2O \rightarrow (CH_3)_2SnO + 2HCl \qquad (1)$$

and precipitates an anthranilinium salt of the dimethyltetra-

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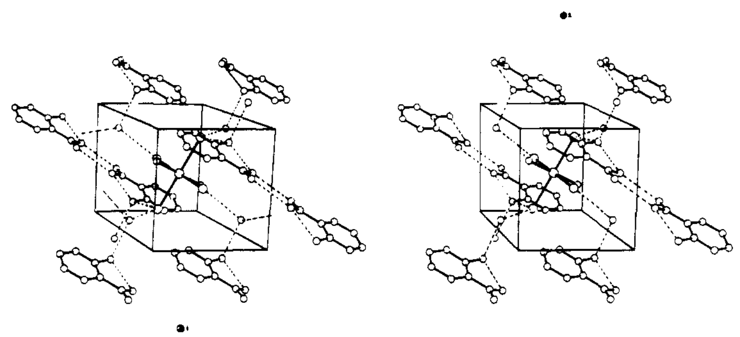

Figure 2. Stereoview of the unit cell of $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[\textit{trans}\text{-}(CH_3)_2SnCl_4]^{2-}(H_2O)_2$.

chlorotin(IV) dianion as a dihydrate on addition of anthranilamide:

$$(CH_3)_2SnCl_2 + 2HCl + 2H_2O + 2(2\text{-}H_2NC_6H_4C(O)NH_2) \rightarrow [2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2 \quad (2)$$

The crystalline product is a complex associated network of hydrogen bonds in which all but one hydrogen atom on each of the two kinds of nitrogen (anilinium and benzamide) are utilized (see Figures 1 and 2). The tin atoms lie on centrosymmetric positions with trans methyl groups. Pairs of trans chlorine atoms are two- and three-coordinated by hydrogen bonds to the water oxygen (all four chlorine atoms) and the anilinium nitrogen (one pair of chlorine atoms). The anthranilamide units are internally hydrogen bonded as shown in Figure 1, and the benzamide units are dimerized as shown in Figure 2.

The situation at the tin atom can be directly compared with that in the analogous pyridinium salt,[9] in which the tin atom also sits at a centrosymmetric position with trans methyl groups and D_{4h} symmetry. The distance d(Sn–C) in this material at 2.109 Å is shorter by 0.01 Å, and the distances d(Sn–Cl) at 2.603 and 2.625 Å brackets the ones found here. Hydrogen bonding is also a feature of the pyridinium salt structure, but in that case pairs of cis chlorines form bifurcated bonds with each $[C_5H_5NH]^+$ unit at distances d(N–H--Cl) of 3.243 and 3.312 Å. Thus, each chlorine is two-coordinated. The N–H--Cl distances in our structure, on the other hand, involve three-coordinated chlorine and tetrahedral nitrogen atoms of the anilinium unit. The distance d(N–H--Cl) at 3.211 Å is shorter by 0.067 (2) Å, with the tendency toward lengthening of the chlorine bonds owing to their increased coordination number apparently overcome by the single, rather than bifurcated, nature of the hydrogen bonds and the differences between the anilinium and pyridinium systems.

Another difference in the structures is that by hydrogen bonding pairs of pyridinium cations symmetrically to the dimethyltetrachlorotin(IV) dianions, a crystal composed of discrete, neutral ion triplets is produced, while in the title compound the ligand anthranilinium amide cations are, respectively, both intramolecularly and intermolecularly hydrogen bonded to bind the ortho atoms together and pairs of ligands into amide dimers. These ligand dimers then bridge the octahedral tin units to form a three-dimensional network. Additional binding in the network is provided by hydrogen bonding by the equivalent water molecules, each of which utilizes both their hydrogen atoms for this purpose.

The hydrogen bonds formed by the Sn–Cl--H–N systems found in four related tin(IV) salts[9-13] and one adduct[14] whose structures are known are compared in Table VII. Hydrogen bonding through Cl--H–N systems is well-known,[15-17] with structural data for 87 examples being available with an average d(Cl--H–N) of 3.23 Å.[17]

The $[R_2SnCl_4]^{2-}$ dianions are all expected to adopt the ubiquitous *trans*-R_2Sn O_h configuration.[5] In both the pyridinium[9] and the title salts, the tin is at a center of symmetry with the C–Sn–C angle precisely 180°. In other cases the tin-119m Mössbauer quadrupole splitting (QS) values can be related through a point-charge model to the C–Sn–C angle, and this approach has been shown to work in various dimethyl-[18,19] and diphenyltin(IV)[20] systems for which structural data are available.[5] In this treatment it is assumed that the contribution to the QS from the other attachments will be small compared to those of the R groups and that the splitting will reach ca. 4.0 mm s^{-1} in a linear C–Sn–C array. We assume that QS values in excess of 4.0 mm s^{-1}, which we have recently observed in diorganotin(IV) systems, also correspond to linear R_2Sn configurations.[21,22] The $[R_2SnCl_4]^{2-}$ salts for which QS values are known[23-26] are listed in Table VIII. Only the diethyl (163.3°) and divinyl derivatives are predicted to have C–Sn–C angles much different from 180°.

Some enhancement of the magnitude of the QS would be expected in the title compound since the chlorine atoms are two- [Cl(1)] and three-coordinated [Cl(2)] in trans pairs. The former chlorine atoms, which are hydrogen bonded only to adjacent water molecules, are held by the tin atom at d[Sn–Cl(1)] = 2.6047 (5) Å, while the latter, which are hydrogen bonded both to water oxygen and to anilinium nitrogen atoms, lie at a distance, d[Sn–Cl(2)] = 2.6139 (5) Å, longer by 0.0142 (5) Å.

The structure also contains two disparate nitrogen atoms. The anilinium NH_3^+ is four-coordinated through intramolecular hydrogen bonding to the oxygen atom of the benzamide group that is located ortho to it and is intermolecularly hy-

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drogen bonded both to the chlorine of an adjacent $[(CH_3)_2SnCl_4]^{2-}$ unit and to the oxygen atom of an adjacent water molecule. These N–H--O hydrogen bonds are at 2.603 (3) Å (intramolecular) and 2.721 (3) Å (intermolecular) vs. 2.89 Å for 989 known examples.[17] The benzamido nitrogen atom, on the other hand, uses only one of its hydrogens to make an adjacent benzamide into a dimeric unit and produces a three-coordinated situation. These N–H--O=C hydrogen bonds are longer, at d(N–H--O) = 2.904 (2) Å. Thus, only one potential hydrogen-bonding site at the benzamide nitrogen [H(14)] is left unutilized in the structure.

The carbonyl oxygen atoms are also three-coordinated, making hydrogen bonds both intra- and intermolecularly to anilinium and benzamido nitrogen atoms, respectively, as are the oxygen atoms of the water molecules, which utilize both their hydrogen atoms in binding to chlorines and are bound by a hydrogen bond from an adjacent anilinium nitrogen.

The network of hydrogen bonds efficiently fills space with dimeric anthranilamide, water, and dimethyltetrachlorostannate(IV) units, delocalizing the anilinium cation and tin dianion charges through the lattice.

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Registry No. $[2\text{-}H_3NC_6H_4C(O)NH_2]^+{}_2[(CH_3)_2SnCl_4]^{2-}(H_2O)_2$, 88336-79-2; $(CH_3)_2SnCl_2$, 753-73-1; $2\text{-}H_2NC_6H_4C(O)NH_2$, 88-68-6.

Supplementary Material Available: A listing of structure factor amplitudes, Tables V and IX–XI, showing anisotropic thermal parameters, C–C distances, C–C–C angles, least-squares plane parameters, and hydrogen atom parameters, and Figure 3, showing the coordination geometries and dimer dimensions (12 pages). Ordering information is given on any current masthead page.

Contribution from the Laboratoire de Chimie de Coordination du CNRS,
associé à l'Université Paul Sabatier, 31400 Toulouse, France

Synthesis and Structural Characterization of a New Ruthenium–Gold Cluster Complex: $Ru_3(\mu\text{-}AuPPh_3)(\mu\text{-}Cl)(CO)_{10}$

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The tetranuclear mixed-metal cluster complex $Ru_3(\mu_3\text{-}AuPPh_3)(\mu\text{-}Cl)(CO)_{10}$ has been synthesized from the reaction of $Ru_3(CO)_{12}$ with $AuPPh_3Cl$ in refluxing dichloromethane and characterized by analysis, IR, ^1H NMR, and X-ray diffraction at low temperature. X-ray data (−155 °C): monoclinic, space group $P2_1/n$, a = 12.697 (2) Å, b = 16.748 (3) Å, c = 16.078 (3) Å, β = 113.0 (1)°, V = 3147 Å^3, Z = 4. R = 0.022, R_w = 0.024 for 3249 reflections with $F_o^2 > 3\sigma(F_o)^2$. The complex has a butterfly metal framework with the gold atom occupying one of the wing tips. Bond distances of interest: (i) Ru–Ru (unsupported) 2.8197 (7) and 2.8125 (8) Å; (ii) (μ-Au)(μ-Cl)Ru–Ru 2.8742 (6) Å; (iii) Ru–Au 2.7523 (6) and 2.7549 (6) Å. In terms of isolobal analogy, this complex is shown to have a structure similar to that of the hydrido cluster complexes of general formula $M_3(\mu\text{-}H)(\mu\text{-}X)(CO)_{10}$ (M = Ru, Os; X = Cl, Br, I). The complex is shown to have low stability since thermolysis at 80 °C leads to extrusion of the gold atom and affords the complex $Ru_3(\mu\text{-}Cl)_2(CO)_8(PPh_3)_2$.

Introduction

The isolobal relationship between the hydride lignad and the $AuPR_3$ group[1] has been exemplified in a series of syntheses of mixed-metal cluster complexes.[2-16]

While the first osmium–gold cluster complex, $Os_3(\mu\text{-}AuPPh_3)(\mu\text{-}Cl)(CO)_{10}$, was reported in the literature 13 years ago, attempts to prepare the ruthenium analogue were unsuccessful.[2]

As part of a new research program directed to the synthesis of ruthenium-containing mixed-metal clusters,[17] we have now found a facile synthetic route to $Ru_3(\mu\text{-}AuPPh_3)(\mu\text{-}Cl)(CO)_{10}$. The complex has been characterized by spectroscopic and crystallographic techniques.[18] Additional interest in this

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