Ylide-Metal Complexes. IX.[1] The Preparation and Properties of Metal (Be, Mg, Zn, Cd, Hg, Al, In, Sn, and Pb) Complexes with Methylenetriphenylarsorane

Yoshihisa YAMAMOTO
Faculty of Pharmaceutical Science, Higashi Nippon Gakuen University, Ishikari-Tobetsu, Hokkaido 061-02
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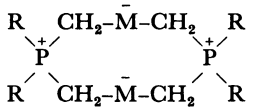
The reactions of methylenetriphenylarsorane, $(C_6H_5)_3As{=}CH_2$ (**L**), with $ZnCl_2$ or $HgCl_2$ in the mole ratio 2:1 yield new monomeric compounds of bis(methylenetriphenylarsorane)metal dichloride, $[(C_6H_5)_3AsCH_2\text{-}M\text{-}CH_2As(C_6H_5)_3]Cl_2$. The reactions of **L** with $BeCl_2$ or $MgCl_2$ in the mole ratio 2:2 yield dimeric trigonal compounds of di-μ-chloro-bis[(methylenetriphenylarsorane)metal] dichloride, $[(C_6H_5)_3AsCH_2\text{-}M\langle^{Cl}_{Cl}\rangle M\text{-}CH_2As(C_6H_5)_3]Cl_2$. The reactions of **L** with $AlCl_3$ or $InCl_3$ in the mole ratio 4:2 yield dimeric tetrahedral compounds of di-μ-chloro-bis[bis(methylenetriphenylarsorane)metal] tetrachloride, $[\{(C_6H_5)_3AsCH_2\}_2\text{-}M\langle^{Cl}_{Cl}\rangle M\text{-}\{CH_2As(C_6H_5)_3\}_2]Cl_4$. The reactions of **L** with $(C_6H_5)_3MCl$ (M: Sn and Pb) in the mole ratio 1:1 yield new monomeric compounds of (methylenetriphenylarsorane)triphenyl-metal chloride, $[(C_6H_5)_3AsCH_2\text{-}M(C_6H_5)_3]Cl$. They are thermally stable complexes and their properties are compared with those of the methylenetriphenylphosphorane metal compounds.

A previous paper,[1] in this series, was concerned with the preparation and properties of Cu, Ag, Au, Zn, Cd, Hg, Ge, Sn, Pb, Pd, and Pt complexes with methylenetris(dimethylamino)phosphorane, $(Me_2N)_3P{=}CH_2$.

Many investigations of organometallic compounds between unstable phosphonium ylides and metals have been reported concerning their preparation, structure, general properties, stability and NMR spectra. The metal compounds with methylenetriphenylphosphorane, $(C_6H_5)_3P{=}CH_2$, have a linear structure,[2-7] *e.g.*,

$$[(C_6H_5)_3\overset{+}{P}CH_2\text{-}\bar{M}\text{-}CH_2\overset{+}{P}(C_6H_5)_3]Cl^- \quad (M: Cu, Ag, Au).$$

On the other hand, the metal compounds with methylenetrimethylphosphorane, $(CH_3)_3P{=}CH_2$, and the related phosphoranes, $R_2(CH_3)P{=}CH_2$ (R: C_2H_5, C_6H_5), are cyclic[8-21] such as



(M: Cu, Ag, Au).

The cyclic structure compound is formed by a rapid intermolecular proton exchange[22] of the phosphoranes.

In the arsenic ylide series, many researchers tried to isolate methylenetriphenylarsorane, $(C_6H_5)_3As{=}CH_2$.[22-25] However, it was not isolated until 1975,[26] because of its sensitivity to air, water, and temperature. Even now, the investigation of organometallic compounds with methylenetriphenyl(or trimethyl)arsorane is limited to the Cu, Ag, Au compounds.[3,28-30] Very little is known of the physical properties and general chemistry of the arsorane metal compounds, in contrast to the information on phosphorane metal compounds. The present paper deals with the preparation and properties of bis(methylenetriphenylarsorane)metal dichloride, $[(C_6H_5)_3AsCH_2\text{-}M\text{-}CH_2As(C_6H_5)_3]Cl_2$ (M: Zn, Cd, Hg), di-μ-chloro-bis[(methylenetriphenylarsorane)metal] dichloride, $[(C_6H_5)_3AsCH_2\text{-}M\langle^{Cl}_{Cl}\rangle M\text{-}CH_2As(C_6H_5)_3]Cl_2$ (M: Be, Mg), di-μ-chloro-bis[bis(methylenetriphenylarsorane)-metal] tetrachloride, $[\{(C_6H_5)_3AsCH_2\}_2\text{-}M\langle^{Cl}_{Cl}\rangle M\text{-}\{CH_2As(C_6H_5)_3\}_2]Cl_4$ (M: Al, In), and (methylenetriphenylarsorane)triphenylmetal chloride, $[(C_6H_5)_3AsCH_2M(C_6H_5)_3]Cl$ (M: Sn, Pb).

Results and Discussion

Zinc, Cadmium, and Mercury Complexes. Bis(methylenetriphenylarsorane)zinc dichloride (**1**) and the corresponding mercury(II) chloride (**3**) have been isolated from a reaction mixture of arsorane, $(C_6H_5)_3As{=}CH_2$ (**L**), and $ZnCl_2$ or $HgCl_2$ in the mole ratio of 2:1. The corresponding cadmium complex (**2**) was not isolated (*cf.* Experimental section). Complexes **1** and **3** are soluble in chloroform, dichloromethane, *N,N*-dimethylformamide (DMF), dimethyl sulfoxide(DMSO) and methanol but insoluble in acetone, ether, pentane, tetrahydrofuran (THF), ethyl acetate, acetic acid, hydrochloric acid and water.

$$2(C_6H_5)_3As{=}CH_2 + MCl_2 \longrightarrow [(C_6H_5)_3AsCH_2\text{-}M\text{-}CH_2As(C_6H_5)_3]Cl_2$$
1, 3
1: M=Zn, **3**: M=Hg

Complex **3** is unstable in dichloromethane at room temperature, but stable at −80 °C. On the other hand, complex **1** was slowly changed to methyltriphenylarsonium chloride $[(C_6H_5)_3AsCH_3]Cl$ (**4**) in dichloromethane at −80 °C. They are highly hygroscopic. The ^1H NMR spectrum of **3** showed a singlet signal for the CH_2As at δ 2.9 and the multiplet signal for the phenyl groups at δ 7.1—9.2 in a ratio of 2:15 in dichloromethane-d_2 at −80 °C as is shown in Table 1. The chemical shift of the methylene protons of **3** is at a lower field than that[26] (δ 0.93) of arsorane (**L**). This property is similar to those of bis(methylenetriphenylarsorane)gold(I) chloride[3] $[(C_6H_5)_3AsCH_2\text{-}Au\text{-}CH_2As(C_6H_5)_3]Cl$ (**5**) and bis(methylenetriphenylphosphorane)metal(II) dichloride,[4] $[(C_6H_5)_3PCH_2\text{-}M\text{-}CH_2P(C_6H_5)_3]Cl_2$ M: Zn, Cd,

Hg (**6**). The arsorane (**L**) is bonded to the metal atom through the carbanionic donor atom. Complexes **1** and **3** have free anion from the equivalent conductance, and the chloride is precipitated by silver nitrate in methanol–water solution. Thus, we suggest that the structures of **1** and **3** are linear similar to those of complexes **5** and **6**.

Beryllium and Magnesium Complexes. Di-μ-chloro-bis(methylenetriphenylarsorane)beryllium dichloride (**7**) and the corresponding magnesium dichloride (**8**) have been isolated from a reaction mixture of arsorane (**L**) with $BeCl_2$ or $MgCl_2$ in the mole ratio of 2:2. Complex **7** is somewhat soluble in nitric acid and hydrochloric acid, and very slightly soluble in chloroform and dichloromethane. Complex **8** is soluble in DMF, DMSO, methanol, and water, and very slightly soluble in chloroform and dichloromethane, though insoluble in acetic acid, benzene,

$$2(C_6H_5)_3As{=}CH_2 + 2MCl_2 \longrightarrow [(C_6H_5)_3AsCH_2\text{-}M(Cl)_2M\text{-}CH_2As(C_6H_5)_3]Cl_2$$

7, 8

7: M=Be, **8**: M=Mg

ether, ethyl acetate and THF. They are highly hygroscopic. Complexes **7** and **8** were slowly changed to the corresponding arsonium salt (**4**) in dichloromethane at −80°C, showing signals of both **4** and **7** or **8** in the NMR spectra. The NMR spectra of **7** and **8** showed a singlet signal for the CH_2As at δ 2.79 and 2.84, respectively and the multiplet signal for phenyl groups at δ 7.2—8.7 as is shown in Table 1. The chemical shifts of the methylene protons of **7** and **8** are at a lower field than that of **L**. The arsorane is bonded to the metal atom. Two structures for the beryllium compounds have been considered possible: either a tetrahedral (monomeric,[30] dimeric,[31] and polymeric[32,33]) or trigonal (monomeric,[34] dimeric,[35] and trimeric[36,37]) structure. Complexes **7** and **8** are not polymeric, because they are soluble in methanol. They have free anion from the equivalent conductance. Their solubility in chloroform and dichloromethane is different from that of **1**, **3**, **5**, and **6** (linear structure) and di-μ-chloro-bis[bis(methylenetriphenylphosphorane)-metal] tetrachloride,[7] $[\{(C_6H_5)_3PCH_2\}_2\text{-}M(Cl)_2M\text{-}\{CH_2P(C_6H_5)_3\}_2]Cl_4$ M:Al, Ga, In (**9**), and the corresponding arsorane complexes (**11**, **12**) (tetrahedral structure). Their elemental analyses agreed with that of $[LMCl_2]_2$. These properties resemble those of di-μ-chloro-bis(methylenetriphenylphosphorane)metal dichloride, $[(C_6H_5)_3PCH_2\text{-}M(Cl)_2M\text{-}CH_2P(C_6H_5)_3]Cl_2$ M:Be and Mg (**10**), of dimeric trigonal structure,[7] reported previously. Thus, we suggest that their structure can be neither linear nor tetrahedral but a dimeric trigonal structure bridged by chloride.

Table 1. ^1H NMR data of complexes

No	CH_2As δ	C_6H_5 δ	Temp °C	Solvent and Standard
1	2.88 s	7.1—8.7 m	−80	1)
3	2.90 s (4H)	7.1—9.2 m (30H)	−80	1)
7	2.79 s	7.3—8.1 m	−80	1)
8	2.84 s	7.2—8.7 m	−80	1)
11	2.90 s	7.3—8.3 m	−80	1)
12	2.88 s	7.3—8.3 m	−80	1)
13	2.32 s (2H)	6.9—8.0 m (30H)	r.t.	1)
14	2.45 s (2H)	6.8—8.0 m (30H)	r.t.	1)
L[a]	0.93 s (2H)	7.1—8.2 m (15H)	r.t.	2)
4	3.2 s (3H)	7.7 m (15H)	r.t.	1)

Solvents: 1) CD_2Cl_2, 2) $C_6D_5CD_3$. Standard: internal TMS (δ=0). a) Ref. 26.

Aluminium and Indium Complexes. Di-μ-chloro-bis[bis(methylenetriphenylarsorane)aluminium] tetrachloride (**11**) and the corresponding indium tetrachloride (**12**) have been isolated from a reaction mixture of arsorane (**L**) and $AlCl_3$ or $InCl_3$ in the mole ratio of 4:2. Complexes **11** and **12** are soluble in chloroform, dichloromethane, DMF, DMSO, methanol and water, though insoluble in acetone, benzene, acetic acid, and THF. They are highly hygroscopic. The ^1H NMR

$$4(C_6H_5)_3As{=}CH_2 + 2MCl_3 \longrightarrow \left[\begin{matrix}(C_6H_5)_3AsCH_2 & Cl & Cl & CH_2As(C_6H_5)_3\\ & M & M & \\ (C_6H_5)_3AsCH_2 & Cl & Cl & CH_2As(C_6H_5)_3\end{matrix}\right]Cl_4$$

11, 12

11: M=Al, **12**: M=In

spectra of complexes **11** and **12** showed a singlet signal for the CH_2As at about δ 2.9 and a multiplet signal for the phenyl groups at δ 7.3—8.3, though both complexes were slowly changed to salt **4** in dichloromethane at −80°C. Complexes **11** and **12** are not polymeric, because they are soluble in dichloromethane and methanol. The elemental analyses of **11** and **12** agreed with that of $[L_2MCl_3]_2$. Their colors are white. They have free anions from the equivalent conductance. Their properties resemble those of complex **9**.[7] The structure of $AlCl_3$, $InCl_3$ and related compounds are of a dimeric, tetrahedral structure bridged by chloride.[38—42] Then, we suggest that their structure is dimeric bridged by chloride and tetrahedral.

Tin and Lead Complexes. (Methylenetriphenylarsorane)triphenyltin chloride (**13**) and the corresponding lead chloride (**14**) have been isolated from a reaction mixture of the arsorane (**L**) and triphenyltin chloride or triphenyllead chloride in the mole ratio of 1:1. Complexes **13** and **14** are soluble in alcohols,

$$(C_6H_5)_3As{=}CH_2 + (C_6H_5)_3MCl \longrightarrow [(C_6H_5)_3AsCH_2\text{-}M(C_6H_5)_3]Cl$$

13, 14

13: M=Sn, **14**: M=Pb

chloroform, dichloromethane, DMF, DMSO, and THF, but insoluble in benzene, ether, and water. They are hygroscopic. Complex **14** is soluble in chloroform and dichloromethane, though complex **13** is slightly soluble in both solvents. The ^1H NMR spectra of **13** and **14** showed a singlet signal for the methylene group

(CH_2As) at δ 2.32 for **13** (δ 2.45 for **14**) and a multiplet signal for the phenyl group at δ 6.9—8.0 in a ratio of 2:30 in dichloromethane at room temperature as is shown in Table 1. The chemical shifts of their methylene protons are at a lower field than that of **L**. Complexes **13** and **14** have free anion from the equivalent conductance, and the chloride is precipitated by silver nitrate in ethanol–water solution. Thus, their structure is monomeric tetrahedral. The chemical shift of methylene protons of **14** is different from that (δ 2.68)[5] of the corresponding phosphorane lead complex, $[(C_6H_5)_3PCH_2Pb(C_6H_5)_3]Cl$ (**15**), and complex **13** is stable in dichloromethane at room temperature, though the corresponding phosphorane tin complex[5] (**16**) is unstable at −80 °C. This can be ascribed to a property of the As or P in the complexes. The configuration (sp^3-sp^3) of the CH_2–M bonds of **13** and **14** is similar to those of **11** and **12** but complexes **11** and **12** are not so stable as complexes **13** and **14** in dichloromethane.

The monomeric complexes have a linear and tetrahedral structure. The structure of the dimeric complexes is trigonal and tetrahedral. All complexes are thermally stable. The Ag, Au, Hg, Sn, and Pb arsorane complexes are stable but the Cu, Be, Mg, Zn, Al, and In complexes are unstable. In the other hand, the Cu, Ag, Au, Mg, Zn, Cd, Al, Ga, In, and Pb phosphorane complexes are stable in dichloromethane, but the Be, Hg, and Sn complexes are not. The stable and unstable metal complexes with arsorane are the unstable and stable metal complexes of phosphorane, respectively, except for Ag, Au, Pb, and Be complexes. This difference is ascribed to both ylides, *i.e.*, the carbanionic carbon of the arsorane can be tetrahedral[26] and that of the phosphorane is trigonal.[43,44]

Experimental

Measurements. The NMR spectra were recorded with an R-40 spectrometer (Hitachi), Varian XL-200 spectrometer for ^1H NMR. The electric conductivities of solutions were determined by the use of a conductometric meter, CM-30 (Shimadzu) in dichloromethane at −80 °C for **1**, **3**, **7**, **8**, **11**, **12** and room temperature for **13**, **14**.

Starting Materials. Methylenetriphenylarsorane (**L**) was prepared from a reaction mixture of methyltriphenylarsonium bromide[45] and sodium amide in dry THF by the method of the previous paper.[26,28]

Ylide-metal Complexes. Bis(methylenetriphenylarsorane)-zinc Dichloride (1): Zinc dichloride (0.15 g, 1.10 mmol) was added to a dry THF solution (15 cm^3) of arsorane (0.71 g, 2.22 mmol). The mixture was stirred for 1 d at room temperature. The color of the ylide changed to colorless from yellow. The white complex which formed was filtered, washed with pentane and dried. Yield: 0.54 g (62.3%). Found: C, 58.78; H, 4.50%. Calcd for $C_{38}H_{34}As_2ZnCl_2$ (MW 776.82) C, 58.76; H, 4.41%. Mp 117—120 °C.

Bis(methylenetriphenylarsorane)cadmium Dichloride (2): Cadmium dichloride (0.22 g, 1.20 mmol) was added to a dry THF solution (10 cm^3) of arsorane (0.78 g, 2.44 mmol). The mixture was stirred for 1—24 h at room temperature. Complex **2** was not isolated from the reaction mixture, because the precipitate was a gum.

Bis(methylenetriphenylarsorane)mercury Dichloride (3): Mercury(II) chloride (0.33 g, 1.22 mmol) was added to a dry THF solution (11 cm^3) of arsorane (0.78 g, 2.44 mmol). The mixture was stirred for 12 h at room temperature. The color of the ylide changed to colorless from yellow. The white complex precipitate was filtered, washed with pentane and dried. Yield: 0.7 g (62.9%). Found: C, 49.81; H, 4.03%. Calcd for $C_{38}H_{34}As_2HgCl_2$ (MW 912.03) C, 50.04; H, 3.76%. Mp 196—198 °C.

Di-μ-chloro-bis[(methylenetriphenylarsorane)beryllium] Dichloride (7): The arsorane **L** (0.44 g, 1.37 mmol) was added to dry THF (14 cm^3), and beryllium dichloride (0.11 g, 1.38 mmol) was added to the solution. The mixture was stirred for 20 h at room temperature, the white complex which precipitated was filtered, washed with pentane and dried. Yield: 0.28 g (51.1%). Found: C, 57.28; H, 4.62%. Calcd for $C_{38}H_{34}As_2Be_2Cl_4$ (MW 800.37) C, 57.03; H, 4.28%. Mp 133—135 °C.

Di-μ-chloro-bis[(methylenetriphenylarsorane)magnesium] Dichloride (8): To a dry THF (10 cm^3) solution of arsorane (0.35 g, 1.09 mmol), magnesium dichloride (0.1 g, 1.05 mmol) was added and the mixture was stirred for 1 d at room temperature. The white complex which formed was filtered, washed with pentane and dried. Yield: 0.4 g (88.3%). Found: C, 54.85; H, 4.50; Cl, 16.14%. Calcd for $C_{38}H_{34}As_2Mg_2Cl_4$ (MW 830.96) C, 54.93; H, 4.12; Cl, 17.06%. Dp 190 °C.

Di-μ-chloro-bis[bis(methylenetriphenylarsorane)aluminium] Tetrachloride (11): Aluminium trichloride (0.18 g, 1.35 mmol) was added to a dry THF (10 cm^3) solution of arsorane (0.87 g, 2.72 mmol) and the mixture stirred for 2 h at room temperature. The white complex precipitate was filtered and washed with pentane and dried. Yield: 0.56 g (53.2%). Found: C, 58.98; H, 4.40; Cl, 13.48%. Calcd for $C_{76}H_{68}As_4Al_2Cl_6$ (MW 1547.74) C, 58.98; H, 4.43; Cl, 13.74%. Mp 157—160 °C.

Di-μ-chloro-bis[bis(methylenetriphenylarsorane)indium] Tetrachloride (12): Indium trichloride (0.20 g, 0.904 mmol) was added to a dry THF (10 cm^3) solution of arsorane (0.58 g, 1.81 mmol) and the mixture stirred for 3 h at room temperature. The white complex which formed was filtered, washed with pentane and dried. Yield: 0.55 g (70.5%). Found: C, 52.61; H, 4.11; Cl, 11.89%. Calcd for $C_{76}H_{68}As_4In_2Cl_6$ (MW 1723.42) C, 52.97; H, 3.98; Cl, 12.34%. Mp 122—125 °C.

Methylenetriphenylarsorane(triphenyl)tin Chloride (13): The arsorane **L** (0.67 g, 2.09 mmol) was added to dry THF (12 cm^3) under nitrogen, and triphenyltin chloride (0.80 g, 2.08 mmol) was added to the solution. The mixture was stirred for 2 h at −40 °C. The color of the ylide changed from yellow to colorless. Dry ether was then added to the solution at −40 °C. The white complex precipitate was filtered at −40 °C, washed with dry ether and dried under vacuum. Yield: 0.56 g (38.0%). Found: C, 62.80; H, 4.45%. Calcd for $C_{37}H_{32}AsSnCl$ (MW 705.73) C, 62.97; H, 4.57%. Dp 120 °C.

Methylenetriphenylarsorane(triphenyl)lead Chloride (14): The arsorane **L** (0.74 g, 2.31 mmol) was added to dry THF (12 cm^3) under nitrogen, and triphenyllead chloride (1.1 g, 2.32 mmol) was added to the solution. The mixture was stirred 1 h at room temperature. The color of the ylide changed to colorless from yellow. Dry pentane was added to the solution, and the white complex precipitate was filtered, washed with pentane and dried under vacuum. Yield: 0.51 g (27.8%). Found: C, 55.69; H, 4.04%. Calcd for $C_{37}H_{32}AsPbCl$ (MW 794.16) C, 55.96; H, 4.06%. Mp 157 °C.

References

1) Part VIII: Y. Yamamoto, *Bull. Chem. Soc. Jpn.*, **57**, 43 (1984).

2) Y. Yamamoto and H. Schmidbaur, *J. Organomet. Chem.*, **96**, 133 (1975).

3) Y. Yamamoto and Z. Kanda, *Bull. Chem. Soc. Jpn.*, **52**, 2560 (1979).

4) Y. Yamamoto and H. Sugimoto, *Bull. Chem. Soc. Jpn.*, **53**, 3176 (1980).

5) Y. Yamamoto, *Bull. Chem. Soc. Jpn.*, **55**, 3025 (1982).
6) D. Seyferth and S. O. Grim, *J. Am. Chem. Soc.*, **83**, 1610 (1961).
7) Y. Yamamoto, *Bull. Chem. Soc. Jpn.*, **56**, 1772 (1983).
8) H. Schmidbaur, J. Adlkofer, and M. Heimann, *Chem. Ber.*, **107**, 3697 (1974).
9) H. Schmidbaur, J. Adlkofer, and W. Buchner, *Angew. Chem. Int. Ed. Engl.*, **12**, 415 (1973).
10) H. Schmidbaur, J. Adlkofer, and W. Buchner, *Angew. Chem.*, **85**, 448 (1973).
11) H. Schmidbaur, J. R. Mandl, A. Frank, and G. Huttner, *Chem. Ber.*, **109**, 466 (1976).
12) H. Schmidbaur and R. Franke, *Inorg. Chim. Acta.*, **13**, 85 (1975).
13) H. Schmidbaur and R. Franke, *Angew. Chem.*, **85**, 449 (1973).
14) H. Schmidbaur and J. Eberlein, *Z. Anorg. Allg. Chem.*, **434**, 145 (1977).
15) H. Schmidbaur and H.-J. Füller, *Chem. Ber.*, **107**, 3674 (1974).
16) D. J. Brauer, C. Krüger, P. J. Roberts, and Y.-H. Tsay, *Chem. Ber.*, **107**, 3706 (1974).
17) H. H. Karsch and H. Schmidbaur, *Chem. Ber.*, **107**, 3684 (1974).
18) H. H. Karsch, H.-F. Klein, and H. Schmidbaur, *Chem. Ber.*, **107**, 93 (1974).
19) H. H. Karsch and H. Schmidbaur, *Angew. Chem.*, **85**, 910 (1973).
20) E. Karras, U. Rosenthal, H. Mennenga, and G. Oehme, *Angew. Chem.*, **85**, 913 (1973).
21) H. Schmidbaur and W. Wolfsberger, *Angew. Chem. Int. Ed. Engl.*, **6**, 448 (1967).
22) P. S. Kendurkar and R. S. Tewari, *J. Organomet. Chem.*, **102**, 141 (1975).
23) D. Seyferth and H. M. Cohen, *J. Inorg. Nucl. Chem.*, **20**, 73 (1961).
24) M. C. Henry and G. Wittig, *J. Am. Chem. Soc.*, **82**, 563 (1960).
25) S. O. Grim and D. Seyferth, *Chem. and Ind. Rev.*, 849 (1959).
26) Y. Yamamoto and H. Schmidbaur, *J. Chem. Soc., Chem. Commun.*, **1975**, 668.
27) S. Schmidbaur and W. Richter, *Chem. Ber.*, **108**, 2656 (1975).
28) W. Richter, Y. Yamamoto, and H. Schmidbaur, *Chem. Ber.*, **110**, 1312 (1977).
29) H. Schmidbaur, J. E. Mandl, W. Richter, V. Bejenke, A. Frank, and G. Huttner, *Chem. Ber.*, **110**, 2236 (1977).
30) V. Amirthalingham, V. M. Padmanabhan, and J. Shanker, *Acta Crystallogr., Sec. B*, **16**, 201 (1960).
31) O. Rahlfs and W. Fischer, *Z. Anorg. Chem.*, **211**, 349 (1933).
32) R. E. Rundle and P. H. Lewis, *J. Chem. Phys.*, **20**, 132 (1952).
33) H. Steinfink and G. D. Brunton, *Acta Crystallogr., Sec. B*, **24**, 807 (1968).
34) L. G. Brisseau, M. G. B. Drew, and J. E. Goulter, *J. Chem. Soc., Chem. Commun.*, **1972**, 916.
35) G. E. Coates and F. Glockling, *J. Chem. Soc.*, **1954**, 22.
36) J. L. Atwood and G. D. Stucky, *J. Am. Chem. Soc.*, **91**, 4426 (1969).
37) J. L. Atwood and G. D. Stucky, *J. Chem. Soc., Chem. Commun.*, **1967**, 1169.
38) H. Kato, K. Yamaguchi, and T. Yonezawa, *Bull. Chem. Soc. Jpn.*, **39**, 1377 (1966).
39) J. D. Forrester, A. Zalkin, and D. H. Templeton, *Inorg. Chem.*, **3**, 63 (1964).
40) M. P. Groenewege, J. Smidt, and H. De Vries, *J. Am. Chem. Soc.*, **82**, 4425 (1960).
41) L. O. Brockway and N. R. Davidson, *J. Am. Chem. Soc.*, **63**, 3287 (1941).
42) K. J. Palmer and N. Elliott, *J. Am. Chem. Soc.*, **60**, 1852 (1938).
43) J. C. J. Bart, *J. Chem. Soc. (B)*, **1969**, 350.
44) J. C. J. Bart, *Angew. Chem. Int. Ed. Engl.*, **7**, 730 (1968).
45) A. Michaelis, *Liebigs Ann. Chem.*, **321**, 141 (1902).