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Organoaluminum-phosphine chemistry. Synthesis and molecular structure of $[Ph_2P(O)_2 \cdot AlCl_2]_2$ and $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$

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Abstract

The crystalline complex $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I) was prepared by reaction of the disproportionation product of ethylaluminum sesquichloride, $EtAlCl_2$, or by direct reaction of $EtAlCl_2$ with diphenylphosphinic acid, $Ph_2P(O)OH$, in a heptane/toluene mixture. I crystallizes in the monoclinic space group $C2/c$ with unit cell parameters a 11.201(9), b 15.059(6), c 17.665(5) Å, β 105.4°(2), V 2979.7 Å^3 and D_{calc} 1.40 g cm^{-3} for $Z = 4$. Least-squares refinement based on 1656 observed reflections led to a final R factor of 0.047 ($R_w = 0.062$). I resides about a crystallographic center of symmetry. The core of the dimer consists of a planar eight-membered $(Al\text{–}O\text{–}P\text{–}O)_2$ ring. The mean Al–O bond distance is 1.73(1) Å while the independent Al–Cl bond distance is 2.085(2) Å. The organoaluminum crystalline product $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II) was prepared by reaction of diisobutylaluminum hydride with diphenylphosphine in heptane. II crystallizes in the orthorhombic space group $Pbca$ with unit cell parameters a 12.809(2), b 15.207(2), c 20.574(3) Å, V 4008.5 Å^3, and D_{calc} 1.08 g cm^{-3} for $Z = 4$. Least-squares refinement based on 2111 observed reflections converged at $R = 0.036$, $R_w = 0.049$. The dimeric organoaluminium compound possesses crystallographic inversion symmetry and contains a planar four-membered Al_2P_2 ring. The mean Al–P bond distance is 2.476(1) Å. The Al–P–Al bond angle in the Al_2P_2 ring is 93.8(1)° while the P–Al–P bond angle is determined to be 86.2(1)°. The Al · · · Al and P · · · P contact distances are 3.615(1) Å and 3.381(1) Å, respectively.

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Introduction

Although the interactions of aluminum species with various Lewis bases have traditionally been a mainstay of organoaluminum chemistry, the reaction of AlR_3 with various oxygen based ligands is currently experiencing considerable activity. This renewed interest in the chemistry of Al–O fragments may be traced to a number of factors: (a) A recent report of an oxygen based ligand assisting in the stabilization of a rare six-coordinate aluminum alkyl [1]; (b) Studies concerning the role of oxygen based crown ethers in organoaluminum based inclusion compounds [2–4]; and (c) The interactions of AlR_3 species with calixarenes [5]. The organoaluminum chemistry of oxygen based ligands notwithstanding, workers investigating the role of phosphines in sterically crowded aryloxide compounds of aluminum [6] and assessing the disproportionation of alkylaluminum halides in the presence of phosphines have contributed also to a resurgence of Al–P chemistry [7]. The study described herein was undertaken in an effort to further explore the organoaluminum chemistry of Al–O and Al–P fragments. Herein we report the synthesis and X-ray crystal structures of $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I) and $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II). I may be prepared from reaction of either the disproportionation product of ethylaluminum sesquichloride, $EtAlCl_2$, or directly from ethylaluminum dichloride with diphenylphosphinic acid, $Ph_2P(O)OH$. II was isolated from reaction of diphenylphosphine with diisobutylaluminum hydride.

Experimental

General comments

Owing to the extreme air and water sensitivity of both reactants and products, the exclusion of oxygen and moisture from the synthesis and subsequent manipulations was essential. To this end, standard Schlenk technique was employed in conjunction with an inert atmosphere glove box (Vacuum Atmospheres HE-43 Dri-Lab). Solvents were distilled under pre-purified argon prior to use. Diphenylphosphinic acid, diphenylphosphine, and ethylaluminum sesquichloride were purchased from Aldrich Chemical Co. Trimethylaluminum and ethylaluminum dichloride were generously donated by Ethyl Corporation.

Synthesis of $[Ph_2P(O)_2 \cdot AlCl_2]_2$ *(I)*

A reaction vessel was charged with diphenylphosphinic acid, $Ph_2P(O)OH$, (4.6 mmol) and taken into the drybox where heptane and toluene were added (20 and 2 ml, respectively). Ethylaluminum sesquichloride ($Et_3Al_2Cl_3$, 9.20 mmol) was slowly added via syringe. Reaction was immediate, vigorous, and exothermic. The reaction vessel was removed from the dry box and heated in an oil bath (100 °C) for 12 h. Upon cooling to room temperature a multitude of colorless, air-sensitive crystals were obtained in quantitative yield. ^1H NMR ($CDCl_3$): δ 7.28 (s, 20H, Ph). The same product was obtained from reaction of ethylaluminum dichloride with diphenylphosphinic acid.

Synthesis of $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ *(II)*

Inside the drybox a reaction vessel was charged with heptane (20 ml) and diphenylphosphine, $Ph_2P–H$, (5.7 mmol). Diisobutylaluminum hydride, (i-

Bu)$_2$Al–H, 5.7 mmol was slowly added via syringe. The system was removed from the drybox and heated in an oil bath (80 °C) for 12 h. The reaction vessel was vented frequently. Upon cooling, a multitude of large, colorless, parallelepiped crystals were obtained in quantitative yield. ^{1}H NMR ($CDCl_3$): δ 7.16 (s, 20H, Ph), −0.44 to −0.51 (m, 36H, Al(i-Bu)).

X-ray structural solution and refinement

[$Ph_2P(O)_2 \cdot AlCl_2]_2$ (I). A colorless, rectangular crystal was mounted in a thin-walled glass capillary under an inert atmosphere of argon. Preliminary examination and data collection were performed on an Enraf–Nonius CAD4 diffractometer equipped with a graphite crystal incident beam monochromator. Cell parameters and an orientation matrix for data collection, obtained from least-squares refinement using the setting angles of 25 carefully centered reflections in the range $12.00 < 2\theta < 16.00$, corresponded to a monoclinic cell. The space group was determined to be $C2/c$. A total of 2764 reflections were collected. The structure was solved by direct methods. Hydrogen atoms were located by standard difference techniques. The phenyl hydrogen atoms were refined using isotropic thermal parameters. Relevant crystallographic data are given in Table 1 while final fractional coordinates and selected bond distances and angles are provided in Tables 2 and 3, respectively. Figure 1 provides the X-ray crystal structure of I.

[$Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II). A colorless parallelepiped crystal was mounted in a thin-walled glass capillary under an inert atmosphere. All measurements were made

Table 1

Summary of the crystallographic data for $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I) and $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II)

	I	II
Empirical formula	$C_{24}H_{20}Al_2Cl_4P_2O_4$	$C_{40}H_{56}Al_2P_2$
Color; habit	colorless; parallelepiped	
Space group	$C2/c$	$Pbca$
Unit cell Dimensions		
a, Å	11.201(9)	12.809(2)
b, Å	15.059(6)	15.207(2)
c, Å	17.665(5)	20.574(3)
β, deg	105.4(2)	
V, Å^3	2979.7	4008.5
Molecules/cell	4	4
Mol. wt.	630.15	652.86
D (calcd), g/cm^3	1.40	1.08
Diffractometer	Enraf-Nonius CAD4	Nicolet R3/m
	Mo-K_α (λ = 0.71073 Å)	
Temp, °C	23	21
2θ range, deg	0.00–50.0	3.50–45.0
Reflections collected	2764	2610
Reflections obsd	1656	2111
GOF	2.02	1.38
Data to parameter ratio	8.20 : 1	6.88 : 1
R	0.047	0.036
R_w	0.062	0.049

Table 2

Positional parameters and their estimated standard deviations for $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I) [a]

Atoms	x	y	z	B (Å^2)
Al(1)	0.9266(1)	0.33350(7)	0.04273(7)	3.26(2)
P(1)	0.8255(1)	0.15546(6)	0.09281(7)	3.83(2)
O(1)	0.9102(2)	0.2310(2)	0.0844(2)	3.89(6)
O(2)	0.7876(3)	0.3519(2)	−0.0240(2)	5.05(8)
Cl(1)	0.9548(1)	0.43246(8)	0.12873(8)	6.26(3)
Cl(2)	1.0713(1)	0.33018(9)	−0.01095(8)	6.58(3)
C(1)	0.4742(6)	0.6075(4)	−0.1141(4)	8.7(2)
C(2)	0.4197(6)	0.5365(4)	−0.1541(5)	10.8(2)
C(3)	0.4793(5)	0.4547(4)	−0.1465(4)	8.2(2)
C(4)	0.9055(4)	0.0532(3)	0.0991(3)	4.4(1)
C(5)	0.6480(6)	0.5194(3)	−0.0589(4)	8.4(2)
C(6)	0.5872(6)	0.5998(4)	−0.0660(5)	9.6(2)
C(7)	0.7018(4)	0.1116(4)	0.2008(3)	6.8(1)
C(8)	0.6629(5)	0.1249(5)	0.2657(4)	9.1(2)
C(9)	0.6967(6)	0.1998(6)	0.3099(3)	9.2(2)
C(10)	0.7704(5)	0.2630(4)	0.2899(3)	7.3(2)
C(11)	0.8115(4)	0.2487(3)	0.2238(3)	5.2(1)
C(12)	0.7764(4)	0.1743(3)	0.1783(3)	4.4(1)

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

on a Nicolet R3m/V diffractometer. Cell parameters and an orientation matrix for data collection, obtained from a least-squares refinement of the setting angles of 40 carefully centered reflections in the range $26.15 < 2\theta < 37.66°$ corresponded to an

Table 3

Selected bond distances (Å) and angles (deg) for $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I)

Al(1)–O(1)	1.741(3)	C(2)–C(3)	1.391(9)
Al(1)–O(2)	1.716(3)	C(5)–C(6)	1.378(8)
Al(1)–Cl(1)	2.091(2)	C(1)–C(2)	1.335(9)
Al(1)–Cl(2)	2.085(2)	C(1)–C(6)	1.329(8)
P(1)–O(1)	1.514(3)	C(7)–C(8)	1.346(9)
P(1)–C(12)	1.762(5)	C(8)–C(9)	1.37(1)
P(1)–C(4)	1.770(4)	C(9)–C(10)	1.37(1)
C(7)–C(12)	1.388(7)	C(10)–C(11)	1.382(8)
C(11)–C(12)	1.374(6)		
O(1)–Al(1)–O(2)	104.7(1)	P(1)–C(12)–C(11)	121.8(4)
O(1)–Al(1)–Cl(1)	109.7(1)	C(7)–C(12)–C(11)	119.2(5)
O(1)–Al(1)–Cl(2)	110.7(1)	C(3)–C(2)–C(1)	121.2(6)
O(2)–Al(1)–Cl(1)	108.5(1)	C(5)–C(6)–C(1)	119.9(5)
O(2)–Al(1)–Cl(2)	111.8(1)	C(12)–C(7)–C(8)	120.0(5)
Cl(1)–Al(1)–Cl(2)	111.2(8)	C(7)–C(8)–C(9)	120.4(6)
O(1)–P(1)–C(12)	108.0(2)	C(8)–C(9)–C(10)	121.5(6)
O(1)–P(1)–C(4)	109.8(2)	C(9)–C(10)–C(11)	118.0(6)
C(12)–P(1)–C(4)	110.8(2)	C(12)–C(11)–C(10)	120.9(5)
Al(1)–O(1)–P(1)	147.9(2)	C(5)–C(1)–C(6)	119.5(5)
P(1)–C(12)–C(7)	119.0(4)		

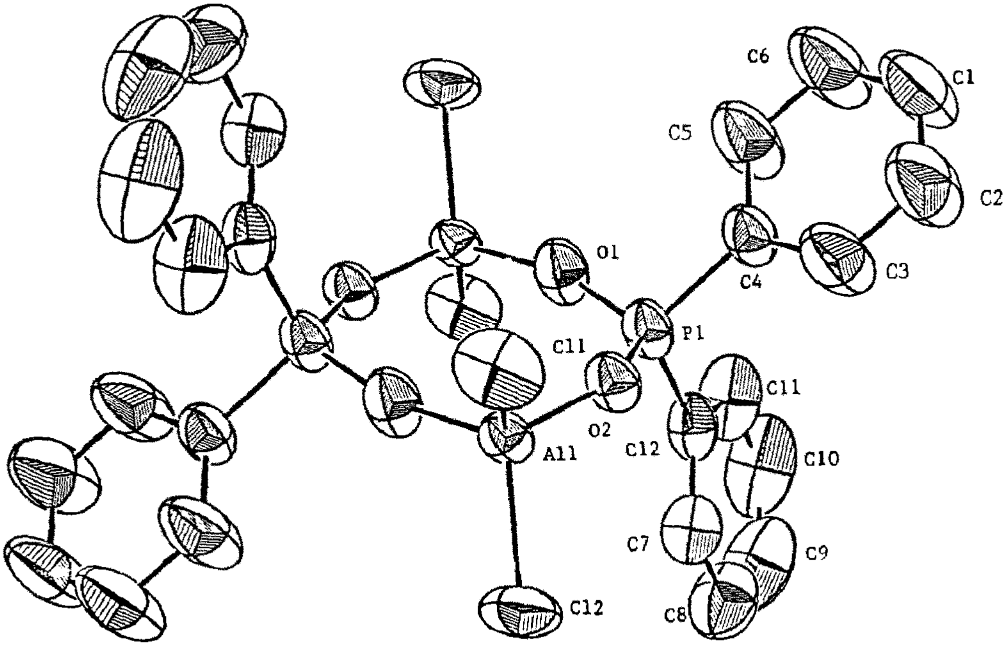

Fig. 1. A view of the $[Ph_2P(O)_2 \cdot AlCl_2]_2$ (I) molecule showing the atom labeling scheme. Thermal ellipsoids show 50% probability levels; hydrogen atoms have been omitted.

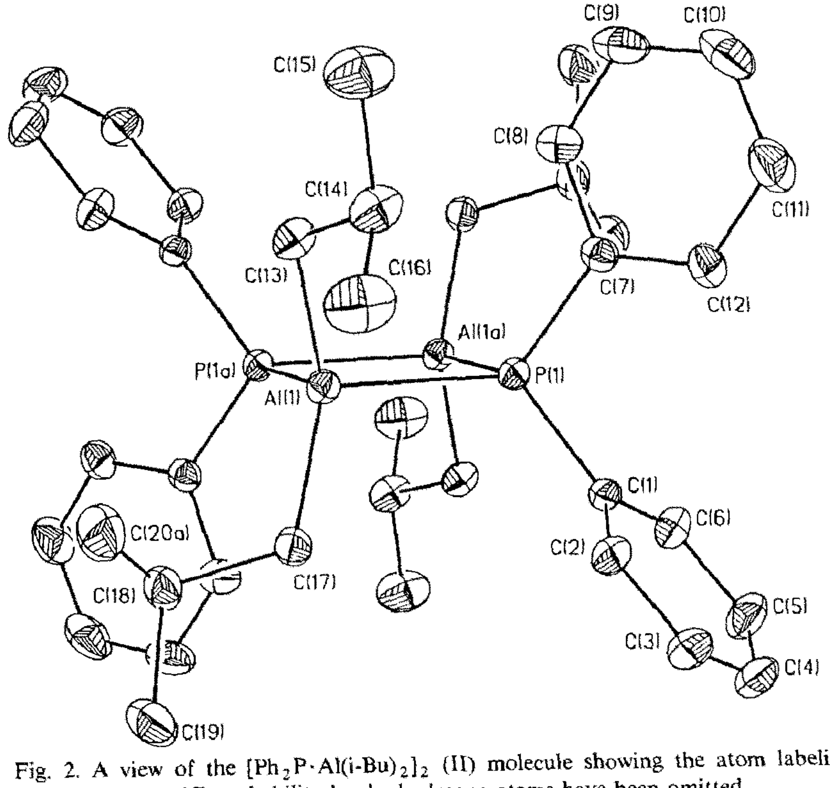

Fig. 2. A view of the $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II) molecule showing the atom labeling scheme. Thermal ellipsoids show 20% probability levels; hydrogen atoms have been omitted.

Table 4

Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($Å^2 \times 10^3$) for $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II)

	x	y	z	U_{eq} [a]
P(1)	9696(1)	478(1)	4263(1)	38(1)
Al(1)	9192(1)	822(1)	5392(1)	39(1)
C(1)	10107(2)	1621(2)	3838(1)	42(1)
C(2)	9703(2)	2582(2)	3917(1)	55(1)
C(3)	9968(3)	3440(3)	3561(2)	72(1)
C(4)	10644(3)	3354(3)	3131(2)	79(1)
C(5)	11059(2)	2422(3)	3046(2)	77(1)
C(6)	10797(2)	1546(3)	3401(1)	59(1)
C(7)	8807(2)	−9(2)	3751(1)	44(1)
C(8)	8401(2)	−938(2)	3904(2)	64(1)
C(9)	7700(2)	−1301(3)	3541(2)	82(1)
C(10)	7398(3)	−737(4)	3025(2)	94(2)
C(11)	7793(2)	176(4)	2861(2)	89(2)
C(12)	8502(2)	541(3)	3215(1)	63(1)
C(13)	7998(2)	232(2)	5483(2)	52(1)
C(14)	7274(2)	714(2)	5060(2)	65(1)
C(15)	6426(2)	106(4)	5096(3)	99(2)
C(16)	7112(3)	1844(3)	5218(3)	98(2)
C(17)	9565(2)	2234(2)	5658(1)	51(1)
C(18)	9629(2)	2466(2)	6389(1)	66(1)
C(19)	10035(4)	3528(3)	6518(2)	99(2)
C(20A)	8676(19)	2566(13)	6662(11)	97(6)
C(20B)	8873(24)	2195(20)	6769(13)	97(10)

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

Table 5

Selected bond distances (Å) and angles (deg) for $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II)

P(1)–Al(1)	2.476(1)	P(1)–C(1)	1.821(2)
P(1)–C(7)	1.830(2)	P(1)–Al(1A)	2.475(1)
Al(1)–C(13)	1.975(3)	Al(1)–C(17)	1.974(3)
C(13)–C(14)	1.533(4)	C(14)–C(15)	1.510(5)
C(14)–C(16)	1.504(5)	C(17)–C(18)	1.537(4)
C(18)–C(19)	1.518(5)	C(18)–C(20A)	1.56(3)
C(18)–C(20B)	1.43(3)		
Al(1)–P(1)–C(1)	114.6(1)	Al(1)–P(1)–C(7)	112.0(1)
C(1)–P(1)–C(7)	104.2(1)	Al(1)–P(1)–Al(1A)	93.8(1)
C(1)–P(1)–Al(1A)	116.4(1)	C(7)–P(1)–Al(1A)	116.2(1)
P(1)–Al(1)–C(13)	107.8(1)	P(1)–Al(1)–C(17)	109.5(1)
C(13)–Al(1)–C(17)	126.0(1)	P(1)–Al(1)–P(1A)	86.2(1)
C(13)–Al(1)–P(1A)	110.1(1)	C(17)–Al(1)–P(1A)	110.0(1)
Al(1)–C(13)–C(14)	116.8(2)	C(13)–C(14)–C(15)	112.2(3)
C(13)–C(14)–C(16)	112.5(3)	C(15)–C(14)–C(16)	110.2(3)
Al(1)–C(17)–C(18)	117.8(2)	C(17)–C(18)–C(19)	111.7(3)
C(17)–C(18)–C(20A)	108.0(9)	C(19)–C(18)–C(20A)	104.0(7)
C(17)–C(18)–C(20B)	116(1)	C(19)–C(18)–C(20B)	117(1)

orthorhombic cell. The space group was determined to be *Pbca*. The structure was solved by direct methods. The non-hydrogen atoms were refined using anisotropic thermal parameters. Hydrogen atoms were located by standard difference Fourier techniques; positional parameters were refined for all of the hydrogen atoms. The phenyl hydrogen atoms were refined using isotropic thermal parameters, while a single group isotropic thermal parameter was utilized for the isobutyl groups. The carbon atom C(20) is disordered, occupying positions on either side of the plane defined by atoms C(17), C(18), and C(19). Refinement of the occupancy factors for these two groups led to values of 0.60 for the major component, C(20a), and 0.40 for the minor component C(20b). Hydrogen atoms H(26) and H(27) correspond to atoms which overlap for the two sites while H(28a) and H(28b) are associated with the major and minor components, respectively. Although the thermal motion of all of the carbon atoms of the isobutyl groups may be regarded as considerable, the refinement of the associated hydrogen atoms to reasonable bonding positions gives no indication of further disorder. The final cycle of full-matrix least-squares refinement was based on 2111 observed reflections ($I > 3\sigma(I)$). Structural solution, refinement and calculation of derived results were performed using the SHELXTL [8] package of computer programs. The X-ray crystal structure of II is given in Fig. 2. Table 1 provides relevant crystallographic data while Tables 4 and 5 provide final atomic coordinates and selected bond distances and angles, respectively.

Results and discussion

The fact that aluminum alkyls will react with virtually any compound having acidic hydrogen atoms is a fundamental cornerstone of organoaluminum chemistry. Principally due to the seminal work of Smith [9] and Cucinella [10], the interactions of aluminum species with monodentate amines is perhaps the most investigated area in this regard. We have recently discovered a somewhat similar, and quite intriguing, organoaluminum chemistry concerning phosphorus ligands.

Our first examination of the organoaluminum chemistry of phosphorus ligands concerned the reaction of trimethylaluminum with bis(diphenylphosphine)methane, $Ph_2PCH_2PPh_2$ [11]. The central methylene hydrogen atoms of $Ph_2PCH_2PPh_2$ proved to be unreactive with respect to $AlMe_3$ as only the 2/1 $[AlMe_3]_2[Ph_2PCH_2PPh_2]$ adduct complex was isolated. In stark contrast, recent reports from this laboratory have described cases where the central methylene hydrogen atoms of both oxygen and sulfur derivatives of $Ph_2PCH_2PPh_2$ based ligands have been induced to react with organoaluminum moieties. Reaction of $AlMe_3$ with bis(diphenylphosphinoyl)methane resulted in the crystalline product $[AlMe][Ph_2P(O)CP(O)Ph_2]_2[AlMe_2]_2$ [12]. This product was interesting in that both central methylene hydrogen atoms were observed to have reacted with a trimethylaluminum unit, eliminating methane, and resulting in dimeric product where two of the bidentate phosphine ligands were bridged by three organoaluminum fragments. A similar organoaluminum product, also involving the cleavage of C–H and Al–R fragments, was isolated from reaction of the sulfur derivative of $Ph_2PCH_2PPh_2$, $Ph_2P(S)CH_2P(S)Ph_2$, with diisobutylaluminum hydride [13]. Given our recent studies concerning the organoaluminum chemistry of bidentate phosphine ligands, we endeavoured to examine the organoaluminum chemistry of diphenylphosphinic acid, $Ph_2P(O)OH$, and diphenylphosphine.

I results from reaction of the organoaluminum moiety with the acidic proton of the acid (eq. 1).

$$Et_3Al_2Cl_3 + Ph_2P(O)OH \rightarrow \tfrac{1}{2}[\underset{(I)}{Ph_2P(O)_2 \cdot AlCl_2}]_2 + Et_2AlCl + Et{-}H \qquad (1)$$

The fact that ethylaluminum sesquichloride reacted as ethylaluminum dichloride may be accounted for by considering the disproportionation of alkylaluminum halides in the presence of Lewis bases [7,14]. I may be considered a dimeric product containing a planar $(Al{-}O{-}P{-}O)_2$ eight-membered ring at the core. Interestingly, the independent Al–O bond distance of 1.716(3) Å must be considered among the shortest Al–O interactions reported [15]. Indeed, the Al(1)–O(2) bond distance is shorter than the value of 2.02(2) Å found in the bis(trimethylaluminum)dioxane adduct, $[Al(CH_3)_3]_2[C_4H_8O_2]$ [16]. The O(1)–Al(1)–O(2) bond angle is determined to be 104.7(1)°. The Al–Cl bond distances of 2.085(2) and 2.091(2) Å are within the expected range. While the reaction of aluminum, gallium and indium alkyls with phosphinic acids derivatives has been previously investigated [17,18], I represents, to the best of our knowledge, the first report of an aluminum-phosphinic acid complex characterized by single crystal X-ray diffraction. Indeed, Coates and Mukherjee [19] previously investigated the reaction of Me_3M (M = Al, Ga) with phosphinic acid. These workers predicted, from cryoscopic measurements, the reaction products to be dimeric with the phosphinic group serving as a three atom bridge (as is observed in I).

Although the interactions of organoaluminum species with primary and secondary amines resulting in alkane elimination and the formation of cage products containing characteristic Al_2N_2 four-membered rings are well documented in the literature, the corresponding organoaluminum chemistry concerning primary and secondary phosphines is comparatively less well developed. The reaction leading to the formation of $[Ph_2P \cdot Al(i\text{-}Bu)_2]_2$ (II) is given in eq. 2.

$$(i\text{-}Bu)_2AlH + Ph_2PH \rightarrow \tfrac{1}{2}[\underset{(II)}{Ph_2P \cdot Al(i\text{-}Bu)_2}]_2 + H_2 \qquad (2)$$

The organoaluminum dimer contains a planar Al_2P_2 four-membered ring at its core. The Al–P–Al bond angle in the Al_2P_2 ring is 93.8(1)° while the P–Al–P bond angle is determined to be 86.2(1)°. The Al · · · Al and P · · · P contacts of 3.615(1) and 3.381(1) Å, respectively, do not suggest significant interaction. The two independent Al–P bond distances in II, 2.476(1) and 2.475(1) Å for Al(1)–P(1) and Al(1)–P(1a), respectively, are comparable to the Al–P distance of 2.458(3) observed in $[Al(i\text{-}Bu)]_2[Ph_2P(S)CP(S)_2Ph_2][Al(i\text{-}Bu)_2]_2$ [13]. Further, all of the bond distances and angles in the Al_2P_2 ring in II are comparable to Al_2P_2 rings in some recently reported phosphinosilylalanes [20]. Indeed, given the considerable steric bulk of isobutyl groups relative to methyl groups, it is interesting that the Al–P bond distances in II are shorter than the values of 2.585(2) and 2.585(2) Å observed for the adduct products $[AlMe_3]_2[Ph_2PCH_2PPh_2]$ [11].

The interactions of aluminum species with phosphorus based ligands have proven to be a particularly fruitful area of organoaluminum chemistry. Future contributions from this laboratory will address other aspects of this interesting area of chemistry.

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