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Structure and equilibria in triorganolead halide adduct formation

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Abstract

The Lewis acidities of triorganolead halides have been studied by ^{31}P and ^{207}Pb NMR spectroscopy and by calorimetry. The formation of 1:1 adducts with a variety of mono- and bidentate bases was demonstrated by the linearity of the plots of chemical shift *versus* (shift/conc.)$^{1/2}$. The ^{207}Pb chemical shifts of the bidentate adducts indicated only 5-coordinate lead and hence the absence of chelation. Towards triphenyllead chloride basicities varied in the order triethylphosphine oxide > tributylphosphine oxide ≥ triphenylphosphine oxide > DMSO > tributylphosphine ≥ pyridine. Triphenyllead chloride was found to be a stronger acid (larger K) than both triphenyllead bromide and triethyllead chloride. The effects of solvent on the equilibrium constants paralleled the trend reported previously for organotin halides.

The equilibrium constant for adduct formation with triphenylphosphine oxide (TPPO) is slightly larger for triphenyllead chloride relative to triphenyltin chloride, confirming the expected increase in acidities down Group 14. X-Ray diffraction studies of triphenylphosphine oxide adducts of triphenyllead bromide and triphenyltin chloride showed both structures to be trigonal bipyramidal with the phenyl groups on the equatorial positions. Additionally, the structures are crystallographically isomorphous.

Introduction

Although organolead adducts have been known since the report of pyridine complexes of diphenyllead dichloride, -dibromide, and -dinitrate in 1916 [1], very little is known about the structure of these adducts and the equilibria involved in their formation. Indeed, only two reports of equilibrium constant determinations for adduct formation could be found: one for the formation of a tetramethylene sulfoxide adduct of triethyllead chloride ($K = 0.196$) [2], the other for adduct formation with dimethylbis(oxinato)lead (for pyridine $K = 2.1$) [3].

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The present study completes our investigation of the relative acidities of organo Group 14 halides [4] through the determination, by NMR and calorimetry, of the equilibria involved in the formation of the adducts of several triorganolead halides with a variety of bases. The structures of an organo lead and organo tin adduct were also determined by X-ray diffraction.

Experimental

All organolead halides were obtained commercially and were dried *in vacuo*. The acids were checked for purity by ^{13}C NMR and were determined to be greater than 98% pure. All phosphines and phosphine oxides were obtained commercially and were dried *in vacuo*.

The bis oxides of bis(diphenylphosphino)methane, -propane, and -butane were prepared by adding 5 mL of 10% H_2O_2 to 6.5 mmol of the phosphine in 60 mL (90 mL for the butane) of acetone. After removal of most of the solvent, 50 mL of hexane was added and the resulting precipitate was filtered, recrystallized from hot acetone and vacuum desiccated. Melting points agreed with literature values [5] and the ^{31}P NMR showed complete conversion to the oxides.

Acetonitrile, *o*-dichlorobenzene, chlorobenzene, chloroform, carbon tetrachloride, and methylene chloride were refluxed over P_2O_5 and then distilled under argon. Toluene and benzene were dried over sodium. All solvents were stored over Linde Type 4A molecular sieves.

All glassware was oven-dried at 115°C for at least 1.5 h and all solutions were prepared in a glove bag (containing P_2O_5) or a HE-43-2 DriLab (Vacuum Atmospheres).

The methods used for the NMR and calorimetric analyses have been previously described [4]. ^{31}P spectra were obtained on a JEOL FX-90Q spectrometer operating at 36.19 MHz with gated decoupling (no NOE). ^{207}Pb spectra were obtained on a Varian Unity 300 spectrometer operating at 62.757 MHz with gated decoupling.

X-Ray data collection for $Ph_3SnCl(TPPO)$ (1) and $Ph_3PbBr(TPPO)$ (2)

Crystal, data collection and refinement parameters are contained in Table 1. Colorless crystals of **1** and **2** were each mounted on fine glass fibers with epoxy cement. The structures of **1** and **2** are isomorphous. The unit-cell parameters of **1** and **2** were each obtained from the least squares fit of 25 reflections ($20° \leq 2\theta \leq 25°$). Preliminary photographic characterization showed $2/m$ Laue symmetry. The systematic absences in the diffraction data uniquely established the space group as $P2_1/c$. An empirical absorption correction was applied to the data set of **2** (216 ψ-scan reflections, pseudoellipsoid model), but was not applied to the data set of **1** (low μ, $T_{max}/T_{min} = 1.106$).

Structure solution and refinement

Compound **2** was solved by heavy-atom methods which located the Pb atom. The remaining non-hydrogen atoms were located through subsequent difference Fourier and least-squares syntheses. Compound **1** was solved using the coordinates of the Pb, Br, P, and O atoms of **2** for the Sn, Cl, P, and O atoms of **1**. All hydrogen atoms for **1** and **2** were included as idealized isotropic contributions (d CH = 0.960 Å, $U = 1.2\ U$ for attached C). All non-hydrogen atoms were anisotrop-

Table 1

Crystallographic data for **1** and **2**

(a) *Crystal parameters*		
Formula	$C_{36}H_{30}OPClSn$	$C_{36}H_{30}OPBrPb$
Formula weight	663.75	796.71
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a (Å)	9.4221(24)	9.468(3)
b (Å)	18.200(5)	18.431(6)
c (Å)	18.198(5)	18.326(5)
β (deg)	93.979(21)	93.090(26)
V (Å^3)	3111.8(13)	3193.4(18)
Z	4	4
Crystal dimensions (mm)	0.30×0.41×0.52	0.38×0.40×0.46
Crystal color	Colorless	Colorless
D(calc.) (g cm^3)	1.416	1.657
μ(Mo-K_α) (cm^{-1})	9.86	65.18
Temperature (K)	297	296
T(max)/T(min)	1.106	2.273
(b) *Data collection*		
Diffractometer	Nicolet R3m	
Monochromator	Graphite	
Radiation	Mo-K_α (λ = 0.71073 Å)	
2θ scan range (deg)	4–50	4–50
Data collected (h,k,l)	±12, +22, +22	±12, +22, +22
Reflections collected	5878	6026
Independent reflections	5492	5633
Independent observed reflections $F_o \geq n\sigma(F_o)$ ($n = 5$)	3698	3258
Standard/reflections	3/197	3/197
Variation in standards	<1	~2
(c) *Refinement*		
$R(F)$ (%)	5.44	5.84
$R(wF)$ (%)	5.70	5.81
Δ/σ(max)	0.025	0.006
$\Delta(\rho)$ (e Å^{-3})	1.002	1.905
N_o/N_v	9.6	11.8
GOF	1.381	1.271

ically refined. For **1**, the [21–26] and [31–36] phenyl rings are 50/50 disordered, presumably in unison. The disorder is manifested as a rotation of the phenyl-ring plane about ipso/para pivot points, which are at full occupancy. The remaining eight C-atom sites were fixed at half occupancy. For **2**, only the disorder in the [31–36] ring was resolved. The disordering in the other ring contained several peaks too closely spaced to refine. Tables 2 and 3 contain positional parameters for **1** and **2**, respectively, and Table 4 contains relevant bond distances and angles for **1** and **2**.

All computer programs and the sources of the scattering factors are contained in the SHELXTL program library (5.1) (G.M. Sheldrick; Nicolet (Siemens), Madison, WI).

Table 2

Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($Å^2 \times 10^3$) for $Ph_3Sn(OPPh_3)Cl$

	x	*y*	*z*	*U* [a]
Sn	1980.4(5)	6397.4(3)	8508.5(2)	41.5(1)
Cl	3515(2)	5514(1)	7862(1)	54(1)
P	423(2)	7715(1)	9794(1)	46(1)
O	596(6)	7268(4)	9117(3)	78(2)
C(1)	−1255(5)	6431(3)	8043(4)	112(5)
C(2)	−2525	6197	7677	151(7)
C(3)	−2523	5605	7190	85(4)
C(4)	−1250	5248	7068	114(5)
C(5)	20	5483	7433	130(6)
C(6)	17	6074	7921	44(2)
C(11)	2407(5)	7885(3)	7768(3)	61(3)
C(12)	3147	8506	7558	90(4)
C(13)	4603	8568	7747	123(6)
C(14)	5319	8009	8145	136(6)
C(15)	4579	7388	8354	95(4)
C(16)	2123	7326	8166	49(2)
C(21)	1784(14)	5419(7)	9747(7)	47(4)
C(22)	2170(25)	5074(12)	10455(10)	62(8)
C(23)	3256(11)	5238(6)	10922(4)	81(4)
C(24)	3924(16)	6017(10)	10694(8)	68(6)
C(25)	3583(13)	6324(7)	10022(7)	50(5)
C(26)	2550(7)	5925(4)	9564(4)	50(2)
C(21′)	1552(21)	5599(11)	10068(10)	46(6)
C(22′)	1945(17)	5266(8)	10733(7)	51(5)
C(24′)	4405(16)	5359(8)	10482(8)	49(5)
C(25′)	3988(13)	5725(8)	9782(7)	43(5)
C(31)	296(15)	6719(8)	10873(7)	44(5)
C(32)	−464(18)	6241(7)	11331(8)	53(6)
C(33)	−2117(12)	6395(7)	11383(8)	116(6)
C(34)	−2702(19)	6775(9)	10826(11)	80(8)
C(35)	−1899(15)	7268(9)	10412(10)	66(7)
C(36)	−542(7)	7223(4)	10459(4)	44(2)
C(31′)	−604(16)	7457(8)	11233(7)	57(5)
C(32′)	−1419(16)	7097(9)	11731(9)	66(6)
C(34′)	−2332(16)	6275(9)	10836(11)	62(6)
C(35′)	−1477(16)	6628(9)	10276(9)	62(6)
C(41)	−1028(7)	8993(3)	10121(3)	83(4)
C(42)	−1844	9612	9931	116(6)
C(43)	−2240	9761	9193	115(6)
C(44)	−1819	9289	8644	106(5)
C(45)	−1003	8669	8833	73(3)
C(46)	−607	8521	9572	53(3)
C(51)	2611(6)	7838(4)	10933(3)	182(9)
C(52)	3978	8046	11196	168(8)
C(53)	4847	8435	10742	76(4)
C(54)	4349	8615	10025	117(5)
C(55)	2982	8407	9761	88(4)
C(56)	2113	8019	10215	47(2)

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

Results and discussion

The equilibrium constants determined by monitoring ^{31}P and ^{207}Pb chemical shifts as a function of concentration [4] are given in Table 5. The linearity of most

Table 3

Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($Å^2 \times 10^3$) for $Ph_3Pb(OPPh_3)Br$

	x	*y*	*z*	*U*
Pb	1972.2(6)	6364.7(3)	8494.9(3)	45.5(2) [a]
Br	3717(2)	5435(1)	7786(1)	58(1) [a]
P	316(4)	7709(2)	9820(2)	54(1) [a]
O	465(12)	7275(7)	9162(6)	76(5) [a]
C(1)	−1270(11)	6399(6)	8008(7)	94(9) [a]
C(2)	−2509	6174	7627	106(10) [a]
C(3)	−2473	5596	7137	81(8) [a]
C(4)	−1198	5244	7026	84(8) [a]
C(5)	41	5469	7407	96(9) [a]
C(6)	5	6046	7898	44(5) [a]
C(11)	2321(11)	7886(7)	7769(6)	64(7) [a]
C(12)	3038	8502	7547	98(10) [a]
C(13)	4491	8566	7702	129(13) [a]
C(14)	5227	8015	8081	135(14) [a]
C(15)	4510	7399	8303	92(9) [a]
c(16)	3057	7334	8147	53(6) [a]
C(21)	3777(13)	6105(8)	9939(8)	241(22) [a]
C(22)	4137	5784	10613	213(16)
C(23)	3238	5274	10905	99(10) [a]
C(24)	1978	5084	10523	154(14) [a]
C(25)	1618	5404	9849	148(13) [a]
C(26)	2517	5915	9557	58(6) [a]
C(31)	128(13)	6699(9)	10895(9)	50(8)
C(32)	−598	6264	11372	68(9)
C(33)	−2042	6370	11448	136(9)
C(34)	−2759	6911	11046	158(22)
C(35)	−2033	7347	10569	107(15)
C(36)	−589	7241	10493	58(6) [a]
C(41)	−1034(15)	9012(7)	10151(5)	110(10) [a]
C(42)	−1830	9626	9956	118(12) [a]
C(43)	−2282	9740	9228	94(10) [a]
C(44)	−1937	9240	8694	91(9) [a]
C(45)	−1141	8626	8889	75(7) [a]
C(46)	−689	8512	9617	53(6) [a]
C(51)	2450(13)	7864(9)	10937(6)	180(18) [a]
C(52)	3801	8078	11191	158(15) [a]
C(53)	4690	8432	10724	99(10) [a]
C(54)	4226	8572	10002	111(11) [a]
C(55)	2875	8359	9748	95(9) [a]
C(56)	1986	8004	10216	58(6) [a]
C(31′)	−1585(37)	6629(20)	10330(20)	77(11)
C(32′)	−2369(43)	6332(25)	10845(25)	97(13)
C(34′)	−1564(44)	7129(26)	11720(24)	101(14)
C(35′)	−738(37)	7470(21)	11220(19)	73(10)

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

Table 4

Relevant bond distances (Å) and bond angles (deg) for **1** and **2**

1		**2**	
Bond distances			
Sn–Cl	2.510(2)	Pb–Br	2.754(2)
Sn–O	2.374(6)	Pb–O	2.556(12)
Sn–C(6)	2.153(5)	Pb–C(6)	2.189(11)
Sn–C(16)	2.120(5)	Pb–C(16)	2.174(12)
Sn–C(26)	2.138(7)	Pb–C(26)	2.153(14)
O–P	1.494(6)	O–P	1.461(12)
Bond angles			
Cl–Sn–O	177.8	Br–Pb–O	176.9(3)
Sn–O–P	147.4(3)	Pb–O–P	146.7(7)
Cl–Sn–C(6)	95.5(2)	Br–Pb–C(6)	96.5(3)
Cl–Sn–C(16)	93.0(1)	Br–Pb–C(16)	93.9(3)
Cl–Sn–C(26)	92.7(2)	Br–Pb–C(26)	93.8(4)
C(6)–Sn–C(16)	120.5(2)	C(6)–Pb–C(16)	118.5(4)
C(6)–Sn–C(26)	119.3(2)	C(6)–Pb–C(26)	120.3(5)
C(6)–Sn–O	86.2(2)	C(6)–Pb–O	86.1(4)
C(16)–Sn–C(26)	118.9(2)	C(16)–Pb–C(26)	119.2(5)
C(16)–Sn–O	85.0(2)	C(16)–Pb–O	83.4(4)
C(26)–Sn–O	87.8(2)	C(26)–Pb–O	86.2(5)

of the shift *versus* (shift./conc.)$^{1/2}$ plots indicates predominant formation of the 1:1 adduct.

$\mathbf{A} + \mathbf{B} \rightarrow \mathbf{A}\text{–}\mathbf{B}$

In most cases the correlation coefficients for the plots were greater than 0.98. For those systems where K is given as 0, the shift of the base (or acid) in a 1:1 mixture with the acid (or base) was within experimental error of the shift of the free base (or acid). For four systems, the deviations from linearity were sufficiently large to indicate the presence of an equilibrium in addition to the formation of the **AB** adduct. This equilibrium or equilibria could be formation of the 1:2 adduct ($\mathbf{AB}_2$), formation of associated **AB** adduct (($\mathbf{AB})_2$), formation of associated acid ($\mathbf{A}_2$), formation of associated base ($\mathbf{B}_2$), or solvent interaction with acid, base, or adduct. For one system, $Ph_3PbBr(Et_3PO)$, with a distinctly curved plot and a correlation coefficient of 0.92, a total of 14 mixtures containing varying amounts of acid and base were examined and the data analyzed by schematic mapping [4]. The results of this analysis showed that none of the systems of simultaneous equilibria, for example, $\mathbf{AB} + \mathbf{AB}_2$, $\mathbf{AB} + \mathbf{A}_2$, $\mathbf{AB} + \mathbf{B}_2$, and so on, gave a better fit to the data than formation of just the **AB** adduct. This is in agreement with our previous observations that in all triorgano- and diorganotin halide adduct systems, the **AB** adduct predominates in solution [4]. Hence, although we chose not to report a 1:1 equilibrium constant for the four systems labelled *curv* in Table 5, formation of the **AB** adduct is very likely the main process occurring in solution for even these systems.

From Table 5 a variety of trends, many previously observed in organotin halide adduct formation, can be postulated:

(1) Bases whose basic site is not an oxygen are significantly weaker toward

Table 5

Equilibrium constants for formation of 1 : 1 adducts with triorganolead halides [a]

Acid	Base	Solvent	*K*
Ph_3PbCl	Ph_3PO	CH_2Cl_2	34
Ph_3PbCl	Et_3PO	CH_2Cl_2	1.2×10^2
Ph_3PbCl	Bu_3PO	CH_2Cl_2	34
Ph_3PbCl	Bu_3P	CH_2Cl_2	0
Ph_3PbCl	$Ph_2P(O)(CH_2)_4P(O)Ph_2$	CH_2Cl_2	1.9×10^2
Ph_3PbCl	$Ph_2P(O)(CH_2)_3P(O)Ph_2$	CH_2Cl_2	58
Ph_3PbCl	$Ph_2P(O)(CH_2)_2P(O)Ph_2$	CH_2Cl_2	60
Ph_3PbCl	$Ph_2P(O)CH_2P(O)Ph_2$	CH_2Cl_2	2.6×10^2
Ph_3PbCl	$Ph_2PCH_2PPh_2$	CH_2Cl_2	0
Ph_3PbCl	Et_3PS	CH_2Cl_2	0
Ph_3PbCl	Et_3PO	THF	1.2×10^2
Ph_3PbCl	Et_3PO	CH_3CN	*curv* [b]
Ph_3PbCl	Ph_3PO	THF	*curv*
Ph_3PbCl	DMSO	CH_2Cl_2	13
Ph_3PbCl	C_5H_5N	CH_2Cl_2	0
Ph_3PbCl	Ph_3PO	C_6H_6	79
Ph_3PbCl	Ph_3PO	$CH_3C_6H_5$	1.2×10^2
Ph_3PbCl	Ph_3PO	CCl_4	1.0×10^2
Ph_3PbCl	Ph_3PO	$CHCl_3$	*curv*
Ph_3PbCl	Ph_3PO	$o\text{-}C_6H_5Cl_2$	*curv*
Ph_3PbCl	Ph_3PO	C_6H_5Cl	72
Ph_3SnCl	Ph_3PO	C_6H_5Cl	51
Ph_3PbBr	Ph_3PO	CH_2Cl_2	18
Ph_3PbBr	Et_3PO	CH_2Cl_2	34
Et_3PbCl	Ph_3PO	CH_2Cl_2	27
Et_3PbCl	Et_3PO	CH_2Cl_2	19

[a] Error $\leq 10\%$. [b] *curv* indicates significant curvature in the shift *versus* $(\text{shift./conc.})^{1/2}$ plots.

Ph_3PbCl than bases with an oxygen. For example, very low equilibrium constants were obtained for Bu_3P, $Ph_2PCH_2PPh_2$, Et_3PS, and C_5H_5N.

(2) Basicity toward Ph_3PbCl in CH_2Cl_2 varies $Et_3PO > Bu_3PO \geq Ph_3PO$, consistent with the electron-withdrawing ability of the phenyl group and the greater steric hindrance of the butyl group.

(3) Ph_3PbCl is a stronger acid than Et_3PbCl toward Et_3PO, while Ph_3PbCl is stronger than Ph_3PbBr (at least as measured by the equilibrium constant [6]). Both observations parallel those made in earlier studies on the tin homologs [4,6].

(4) For the Ph_3PbCl–TPPO system, the equilibrium constant is strongly solvent dependent and varies from 116 for toluene to 0 for $CHCl_3$. Except for the lower value in chlorobenzene, this order also duplicates that observed in an extensive study of solvent effects on Et_2SnCl_2 [7].

(5) The bidentate bases with two and three membered bridges have equilibrium constants between those of TPPO and triethylphosphine oxide, consistent with the one less electron-withdrawing phenyl group in their structures. Somewhat unexpected is the significantly higher equilibrium constant found for $Ph_2P(O)(CH_2)_4P(O)Ph_2$. Although the plot for this compound has more curvature than the plots for the other bidentate bases, it is unlikely that any of the adducts are chelated with six-coordinate lead. If the solutions of these adducts contained

significant amounts of six-coordinate lead, the equilibrium constants would be expected to be inconsistent with those for the monodentate phosphine oxides. Moreover, since it is well known that lead chemical shifts decrease by 100–200 ppm in going from four to five and five to six coordination [8], a significant amount of 6-coordinate lead should result in an observed ^{207}Pb chemical shift at a much lower frequency than observed for triphenyllead chloride. In fact, at 0.025 *M* concentrations of triphenyllead chloride and triphenylphosphine oxide, the ^{207}Pb shift is 82 ppm lower in frequency than the free acid. Because the plot of shift *versus* (shift./conc.)$^{1/2}$ is linear ($r = 0.999$), this system can be assumed to contain only 5-coordinate lead. For a 0.02 *M* mixture of triphenyllead chloride and $Ph_2P(O)(CH_2)_4P(O)Ph_2$, the observed shift is 40 ppm lower in frequency than the free acid and an increase in base concentration to an 8:1 ratio produces a decrease of only an additional 20 ppm. Thus, the presence of a sizeable percentage of 6-coordinate lead in the solutions with bidentate phosphine oxides is unlikely.

In order to compare the acidity of lead as the central atom to that of tin, the TPPO adducts of both triphenyltin chloride and triphenyllead chloride were studied in chlorobenzene by ^{31}P NMR and calorimetry. Both form 1:1 adducts and the equilibrium constants and enthalpy changes are: $Ph_3PbCl(TPPO)$: $K(NMR) = 72 \pm 10$, $K_{cal} = 61 \pm 1$, $\Delta H = -7.9 \pm 0.1$ kcal mol^{-1}; $Ph_3SnCl(TPPO)$: $K(NMR) = 51 \pm 6$, $K_{cal} = 37 \pm 9$, $\Delta H = -8 \pm 1$ kcal mol^{-1}. It is clear from the equilibrium constants that the lead derivative is the slightly stronger acid. This is in agreement with the previously observed trend of increasing acidities down Group 14 [9]. Unfortunately, the error in ΔH for the tin derivative precludes a determination of the origin (ΔH or ΔS) of the difference in equilibrium constant.

The structures of two adducts, $Ph_3SnCl(TPPO)$ (**1**) and $Ph_3PbBr(TPPO)$ (**2**), were determined by X-ray diffraction and the relevant bond angles and distances are reported in Table 4. The ORTEP diagrams of the isomorphous compounds (Figs.

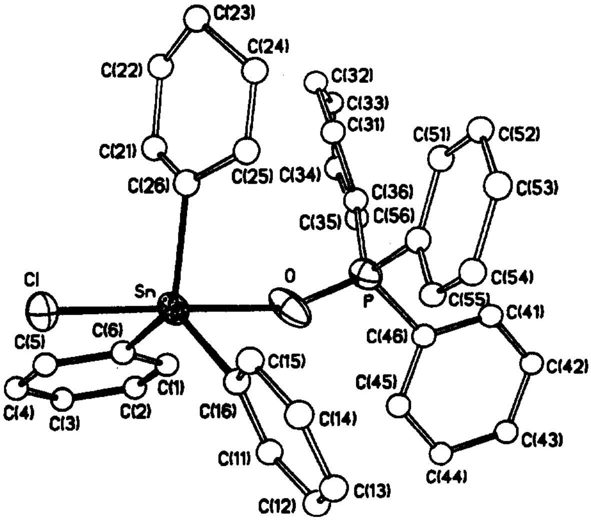

Fig. 1. ORTEP drawing and labeling scheme for **1**. Ellipsoids are drawn at 35% probability. Hydrogen atoms are omitted and carbon atoms are set at a fixed radius for clarity.

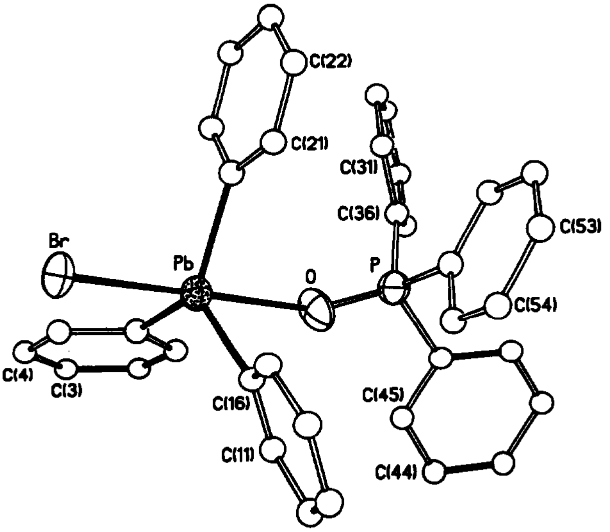

Fig. 2. ORTEP drawing and labeling scheme for 2. Ellipsoids are drawn at 35% probability. Hydrogen atoms are omitted and carbon atoms are set at a fixed radius for clarity.

1 and 2) show that the adducts have a trigonal bipyramidal structure with the phenyl groups occupying the equatorial positions. The phenyl groups are staggered, presumably to minimize steric interactions. The angle at oxygen in both adducts is 147°, indicating considerable widening to accommodate the triphenylphosphino group in the adduct. The Pb–O bond length of 2.56 Å can be compared to a length of 2.01 Å in four-coordinate $Ph_3PbOSiPh_3$ [10], and 2.36 Å and 2.56 Å in associated, 5-coordinate triphenyllead acetate [11].

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