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**Supporting information for article:**

**Between MOFs and molecules: organolead(IV) compounds with chain structures**

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**Table S1.** Atomic coordinates ( $\cdot 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \cdot 10^3$ ) for  $[(\text{Ph}_3\text{Pb})_2\text{Cl}(\text{O}_2\text{AsMe}_2)]$  (**1**).  $U_{\text{(eq)}}$  is defined as one third of the trace of the orthogonalized  $U^{\text{ij}}$  tensor.

	x	y	z	U(eq)
Pb(1)	2153(1)	2534(1)	890(1)	22(1)
Pb(2)	2468(1)	3307(1)	3193(1)	21(1)
As	1958(1)	1232(1)	-372(1)	22(1)
Cl	2468(1)	2938(1)	1987(1)	27(1)
O(1)	2518(3)	1413(4)	-892(2)	29(1)
O(2)	1834(3)	2131(3)	-4(3)	27(1)
C(1)	3288(2)	2547(4)	685(2)	30(2)
C(2)	3801(3)	2420(4)	1087(2)	28(2)
C(3)	4507(2)	2448(4)	941(2)	39(2)
C(4)	4699(2)	2602(4)	394(3)	41(3)
C(5)	4186(3)	2730(4)	-8(2)	43(3)
C(6)	3481(3)	2702(4)	137(2)	35(2)
C(7)	1587(3)	1393(3)	1192(3)	24(2)
C(8)	1921(2)	757(3)	1500(3)	33(2)
C(9)	1570(3)	-3(3)	1626(3)	40(3)
C(10)	886(3)	-128(3)	1443(3)	45(3)
C(11)	552(3)	508(4)	1135(3)	47(3)
C(12)	902(3)	1269(3)	1009(3)	35(2)
C(13)	1551(3)	3738(3)	779(2)	22(2)
C(14)	1211(3)	3876(3)	277(2)	25(2)
C(15)	832(3)	4630(3)	192(2)	29(2)
C(16)	793(3)	5246(3)	610(2)	31(2)
C(17)	1133(3)	5108(3)	1112(2)	32(2)
C(18)	1512(3)	4354(3)	1197(2)	27(2)
C(19)	1776(3)	2159(3)	3269(2)	24(2)
C(20)	1786(3)	1660(4)	3748(2)	35(2)
C(21)	1357(3)	938(3)	3789(2)	37(2)
C(22)	919(3)	715(3)	3351(3)	36(2)
C(23)	909(3)	1214(4)	2872(2)	35(2)
C(24)	1338(3)	1936(3)	2831(2)	30(2)
C(25)	3624(2)	3285(4)	3184(2)	24(2)
C(26)	3991(3)	3391(4)	3678(2)	28(2)
C(27)	4719(3)	3355(4)	3680(2)	34(2)
C(28)	5082(2)	3213(4)	3188(2)	32(2)
C(29)	4716(3)	3107(4)	2694(2)	32(2)
C(30)	3987(3)	3143(4)	2692(2)	26(2)
C(31)	1862(3)	4447(3)	2943(3)	23(2)
C(32)	1144(3)	4337(3)	2871(3)	31(2)
C(33)	730(2)	5026(3)	2703(3)	31(2)

C(34)	1034(3)	5826(3)	2607(3)	34(2)
C(35)	1752(3)	5937(3)	2679(3)	35(2)
C(36)	2166(2)	5247(3)	2847(3)	31(2)
C(37)	2334(5)	304(6)	61(4)	38(2)
C(38)	1081(5)	862(7)	-660(4)	38(2)

**Table S2.** Anisotropic displacement parameters ( $\text{\AA}^2 \cdot 10^3$ ) for  $[(\text{Ph}_3\text{Pb})_2\text{Cl}(\text{O}_2\text{AsMe}_2)]$  (**1**). The anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U^{11} + \dots + 2hk a^* b^* U^{12}]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Pb(1)	26(1)	17(1)	22(1)	0(1)	1(1)	-1(1)
Pb(2)	22(1)	20(1)	22(1)	1(1)	-1(1)	0(1)
As	26(1)	19(1)	22(1)	-1(1)	0(1)	1(1)
Cl	31(1)	26(1)	24(1)	-1(1)	-1(1)	1(1)
O(1)	28(3)	36(3)	23(3)	-1(2)	2(3)	-1(3)
O(2)	39(3)	19(3)	24(3)	-5(2)	0(3)	3(3)
C(1)	38(5)	16(4)	36(5)	-9(4)	-3(4)	0(4)
C(2)	26(4)	27(4)	31(5)	-2(4)	1(4)	2(4)
C(3)	25(5)	36(5)	57(7)	-14(5)	-6(4)	0(4)
C(4)	31(5)	32(5)	60(8)	-21(5)	13(5)	-6(4)
C(5)	46(6)	40(6)	44(7)	-7(5)	12(5)	-13(5)
C(6)	37(5)	34(6)	34(6)	-1(4)	5(4)	-6(4)
C(7)	27(5)	20(4)	25(5)	3(3)	8(4)	-3(3)
C(8)	32(5)	21(4)	47(6)	1(4)	6(4)	2(4)
C(9)	53(7)	25(5)	43(7)	12(4)	5(5)	3(4)
C(10)	49(7)	31(5)	56(8)	-5(5)	5(6)	-19(5)
C(11)	36(6)	43(6)	63(8)	5(5)	-5(5)	-18(5)
C(12)	34(5)	31(5)	40(6)	7(4)	-6(5)	-3(4)
C(13)	23(4)	19(4)	24(5)	1(3)	4(4)	0(3)
C(14)	20(4)	24(4)	31(5)	-5(4)	-1(4)	-4(3)
C(15)	27(5)	32(5)	29(6)	-3(4)	0(4)	8(4)
C(16)	28(5)	28(5)	38(6)	0(4)	-6(4)	10(4)
C(17)	36(6)	27(5)	33(6)	-3(4)	2(5)	1(4)
C(18)	26(5)	31(5)	25(5)	-1(4)	6(4)	-2(4)
C(19)	20(4)	23(4)	30(6)	8(4)	1(4)	-2(3)
C(20)	48(6)	33(5)	24(5)	-3(4)	-1(4)	-7(5)
C(21)	59(7)	24(5)	28(6)	3(4)	9(5)	-15(4)

C(22)	36(5)	24(5)	47(7)	0(4)	12(5)	-9(4)
C(23)	27(5)	31(5)	46(7)	-8(4)	-8(4)	-11(4)
C(24)	28(5)	30(5)	32(6)	5(4)	-3(4)	0(4)
C(25)	35(5)	16(4)	22(5)	3(3)	-4(4)	-3(3)
C(26)	27(5)	29(5)	27(5)	-1(4)	-1(4)	1(4)
C(27)	29(5)	39(5)	34(6)	4(4)	-9(4)	-2(4)
C(28)	22(4)	34(5)	40(6)	5(4)	-1(4)	0(4)
C(29)	31(5)	33(5)	32(6)	-1(4)	7(4)	-8(4)
C(30)	27(5)	22(4)	27(5)	-1(4)	1(4)	-3(3)
C(31)	26(5)	19(4)	23(5)	-1(3)	-4(4)	5(3)
C(32)	32(5)	21(5)	38(6)	1(4)	0(4)	6(4)
C(33)	28(5)	31(5)	34(6)	2(4)	1(4)	0(4)
C(34)	36(5)	22(4)	42(6)	-1(4)	-8(4)	9(4)
C(35)	34(5)	17(4)	54(7)	5(4)	-3(5)	-3(4)
C(36)	24(5)	32(5)	38(6)	4(4)	-3(4)	1(4)
C(37)	40(6)	41(5)	33(6)	12(4)	4(4)	8(4)
C(38)	36(6)	43(6)	35(6)	-11(5)	4(4)	4(5)

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**Table S3.** Selected bond lengths (Å) and angles (°) for [(Ph<sub>3</sub>Pb)<sub>2</sub>Cl(O<sub>2</sub>AsMe<sub>2</sub>)] (1)

Pb(1)-C(7)	2.199(4)	C(31)-Pb(2)-O(1) <sup>#1</sup>	97.8(2)
Pb(1)-C(13)	2.210(4)	C(25)-Pb(2)-O(1) <sup>#1</sup>	88.3(2)
Pb(1)-C(1)	2.219(4)	C(19)-Pb(2)-O(1) <sup>#1</sup>	95.7(2)
Pb(1)-O(2)	2.323(6)	C(31)-Pb(2)-Cl	83.49(18)
Pb(1)-Cl	2.782(2)	C(25)-Pb(2)-Cl	89.22(14)
Pb(2)-C(31)	2.198(4)	C(19)-Pb(2)-Cl	85.75(15)
Pb(2)-C(25)	2.205(4)	O(1)#1-Pb(2)-Cl	177.54(17)
Pb(2)-C(19)	2.224(4)	O(1)-As-O(2)	110.4(3)
Pb(2)-O(1) <sup>#1</sup>	2.248(6)	O(1)-As-C(38)	109.9(4)
Pb(2)-Cl	2.963(2)	O(2)-As-C(38)	108.9(4)
As-O(1)	1.670(6)	O(1)-As-C(37)	107.2(4)
As-O(2)	1.672(5)	O(2)-As-C(37)	113.0(4)
As-C(38)	1.898(10)	C(38)-As-C(37)	107.5(5)
As-C(37)	1.918(9)	Pb(1)-Cl-Pb(2)	167.35(9)
O(1)-Pb(2) <sup>#2</sup>	2.248(6)	As-O(1)-Pb(2) <sup>#2</sup>	137.8(4)
C(7)-Pb(1)-C(13)	117.8(2)	As-O(2)-Pb(1)	132.9(3)
C(7)-Pb(1)-C(1)	124.0(2)	C(2)-C(1)-C(6)	120.0
C(13)-Pb(1)-C(1)	118.2(2)	C(2)-C(1)-Pb(1)	121.9(3)
C(7)-Pb(1)-O(2)	87.8(2)	C(6)-C(1)-Pb(1)	118.1(3)
C(13)-Pb(1)-O(2)	88.86(19)	C(8)-C(7)-Pb(1)	121.6(3)
C(1)-Pb(1)-O(2)	92.9(2)	C(12)-C(7)-Pb(1)	117.9(3)
C(7)-Pb(1)-Cl	88.44(17)	C(14)-C(13)-Pb(1)	118.5(3)
C(13)-Pb(1)-Cl	92.05(14)	C(18)-C(13)-Pb(1)	121.5(3)
C(1)-Pb(1)-Cl	89.98(15)	C(20)-C(19)-Pb(2)	120.4(3)
O(2)-Pb(1)-Cl	176.11(16)	C(24)-C(19)-Pb(2)	119.6(3)
		C(26)-C(25)-Pb(2)	119.4(3)
C(31)-Pb(2)-C(25)	122.3(2)	C(30)-C(25)-Pb(2)	120.6(3)
C(31)-Pb(2)-C(19)	110.9(2)	C(32)-C(31)-Pb(2)	116.9(3)
C(25)-Pb(2)-C(19)	125.5(2)	C(36)-C(31)-Pb(2)	123.1(3)

Symmetry transformations used for equivalent atoms: #1 x,-y+1/2,z+1/2 #2 x,-y+1/2,z-1/2

**Table S4.** Atomic coordinates ( $\cdot 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \cdot 10^3$ ) for Ph<sub>2</sub>ClPbO<sub>2</sub>AsMe<sub>2</sub> (2).  $U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U^{\text{ij}}$  tensor.

	x	y	z	U(eq)
Pb	7294(1)	189(1)	3920(1)	21(1)
Cl	6838(2)	669(1)	1426(2)	28(1)
As	8043(1)	-239(1)	7182(1)	21(1)
O(1)	9474(6)	-265(3)	6067(5)	23(1)
O(2)	6000(6)	-103(3)	6226(5)	25(1)
C(1)	7357(9)	1241(3)	4576(7)	20(2)
C(2)	6534(9)	1432(4)	5607(8)	26(2)
C(3)	6607(10)	2101(4)	5982(9)	30(2)
C(4)	7498(10)	2567(4)	5307(8)	30(2)

C(5)	8295(10)	2366(4)	4244(9)	29(2)
C(6)	8233(10)	1696(4)	3858(8)	24(2)
C(7)	7202(9)	-825(4)	3123(8)	22(2)
C(8)	6264(9)	-917(4)	1841(8)	23(2)
C(9)	6262(10)	-1551(4)	1269(9)	29(2)
C(10)	7132(10)	-2074(4)	1978(9)	32(2)
C(11)	8025(10)	-1982(4)	3244(10)	34(2)
C(12)	8073(10)	-1359(4)	3841(9)	27(2)
C(13)	8766(10)	463(4)	8386(9)	32(2)
C(14)	8019(10)	-1056(5)	8132(9)	33(2)

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**Table S5.** Anisotropic displacement parameters ( $\text{\AA}^2 \cdot 10^3$ ) for  $\text{Ph}_2\text{ClPbO}_2\text{AsMe}_2$  (**2**). The anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U^{11} + \dots + 2hk a^* b^* U^{12}]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Pb	16(1)	22(1)	26(1)	-1(1)	2(1)	0(1)
Cl	29(1)	27(1)	25(1)	0(1)	2(1)	-2(1)
As	14(1)	26(1)	22(1)	1(1)	2(1)	0(1)
O(1)	13(2)	31(3)	25(3)	-5(3)	4(2)	-2(2)
O(2)	9(2)	31(3)	34(3)	-3(3)	-2(2)	1(2)
C(1)	22(3)	14(3)	22(4)	-10(3)	0(3)	1(3)
C(2)	18(3)	23(4)	35(5)	-9(4)	4(3)	4(3)
C(3)	21(4)	35(5)	35(5)	-5(4)	2(3)	3(3)
C(4)	25(4)	23(4)	37(5)	-7(4)	-4(3)	3(3)
C(5)	27(4)	28(4)	33(5)	3(4)	3(3)	0(3)
C(6)	25(4)	23(4)	25(4)	-3(3)	1(3)	-3(3)
C(7)	17(3)	15(4)	32(5)	0(3)	-2(3)	-2(3)
C(8)	24(3)	25(4)	22(4)	3(3)	6(3)	-5(3)
C(9)	25(4)	30(4)	31(5)	-2(4)	3(3)	-5(3)
C(10)	25(4)	25(4)	44(5)	-11(4)	0(4)	1(3)
C(11)	24(4)	24(4)	51(6)	-5(4)	0(4)	4(3)
C(12)	19(3)	31(4)	29(5)	-1(4)	-3(3)	2(3)
C(13)	18(3)	38(5)	41(5)	-1(4)	6(3)	7(3)
C(14)	23(4)	38(5)	37(5)	2(4)	7(3)	0(3)

**Table S6.** Selected bond lengths (Å) and angles (°) for Ph<sub>2</sub>CIPbO<sub>2</sub>AsMe<sub>2</sub> (**2**).

Pb-C(7)	2.180(7)	O(2) <sup>#2</sup> -Pb-O(1)	119.20(16)
Pb-C(1)	2.202(7)	C(7)-Pb-Cl	88.9(2)
Pb-O(1) <sup>#1</sup>	2.389(4)	C(1)-Pb-Cl	86.9(2)
Pb-O(2) <sup>#2</sup>	2.416(4)	O(1) <sup>#1</sup> -Pb-Cl	86.92(13)
Pb-O(1)	2.662(5)	O(2) <sup>#2</sup> -Pb-Cl	90.25(13)
Pb-Cl	2.706(2)	O(1)-Pb-Cl	150.20(10)
As-O(2)	1.678(4)	O(2)-As-O(1)	102.1(2)
As-O(1)	1.687(5)	O(2)-As-C(13)	113.1(3)
As-C(13)	1.885(9)	O(1)-As-C(13)	109.3(3)
As-C(14)	1.902(9)	O(2)-As-C(14)	110.7(3)
O(1)-Pb <sup>#1</sup>	2.389(4)	O(1)-As-C(14)	112.6(3)
O(2)-Pb <sup>#2</sup>	2.416(4)	C(13)-As-C(14)	109.0(4)
C(1)-C(2)	1.363(11)	As-O(1)-Pb <sup>#1</sup>	137.2(3)
C(1)-C(6)	1.396(11)	As-O(1)-Pb	101.16(19)
C(2)-C(3)	1.389(11)	Pb <sup>#1</sup> -O(1)-Pb	116.49(19)
C(3)-C(4)	1.390(12)	As-O(2)-Pb <sup>#2</sup>	145.1(3)
C(5)-C(6)	1.392(11)	C(2)-C(1)-C(6)	122.5(7)
C(7)-C(12)	1.390(11)	C(2)-C(1)-Pb	121.0(6)
C(7)-C(8)	1.394(11)	C(6)-C(1)-Pb	116.5(5)
C(8)-C(9)	1.394(11)	C(1)-C(2)-C(3)	118.9(8)
C(9)-C(10)	1.370(12)	C(2)-C(3)-C(4)	120.2(8)
C(10)-C(11)	1.368(12)	C(5)-C(4)-C(3)	120.1(8)
C(11)-C(12)	1.385(11)	C(4)-C(5)-C(6)	120.3(8)
		C(5)-C(6)-C(1)	118.0(7)
C(7)-Pb-C(1)	175.8(3)	C(12)-C(7)-C(8)	120.9(7)
C(7)-Pb-O(1) <sup>#1</sup>	91.6(2)	C(12)-C(7)-Pb	122.0(6)
C(1)-Pb-O(1) <sup>#1</sup>	88.2(2)	C(8)-C(7)-Pb	117.2(5)
C(7)-Pb-O(2) <sup>#2</sup>	86.8(2)	C(7)-C(8)-C(9)	118.9(7)
C(1)-Pb-O(2) <sup>#2</sup>	93.2(2)	C(10)-C(9)-C(8)	120.0(8)
O(1) <sup>#1</sup> -Pb-O(2) <sup>#2</sup>	176.80(18)	C(11)-C(10)-C(9)	120.7(8)
C(7)-Pb-O(1)	88.4(2)	C(10)-C(11)-C(12)	121.0(8)
C(1)-Pb-O(1)	95.3(2)	C(11)-C(12)-C(7)	118.5(8)
O(1) <sup>#1</sup> -Pb-O(1)	63.51(19)		

Symmetry transformations used to generate equivalent atoms: #1 -x+2,-y,-z+1 #2 -x+1,-y,-z+1

**Table S7.** Atomic coordinates ( $\cdot 10^4$ ) and equivalent isotropic displacement parameters ( $\text{Å}^2 \cdot 10^3$ ) for Ph<sub>3</sub>PbO<sub>2</sub>PPh<sub>2</sub> (**3**). U<sub>(eq)</sub> is defined as one third of the trace of the orthogonalized U<sup>ij</sup> tensor.

	x	y	z	U(eq)
Pb	2267(1)	8437(1)	1955(1)	35(1)
P	1595(2)	6062(2)	3176(2)	34(1)
O(1)	1699(4)	6709(4)	2329(4)	39(1)
O(2)	2323(4)	5195(4)	3428(4)	40(1)
C(1)	2631(4)	7809(4)	635(3)	38(2)
C(2)	2790(4)	8476(3)	-93(3)	36(2)



C(3)	3081(4)	8058(4)	-901(3)	43(2)
C(4)	3213(4)	6972(4)	-980(3)	49(2)
C(5)	3054(4)	6304(3)	-252(4)	50(2)
C(6)	2763(4)	6722(4)	556(3)	42(2)
C(7)	3428(4)	8381(4)	3127(3)	36(2)
C(8)	4179(5)	7649(5)	3124(4)	60(3)
C(9)	4932(4)	7617(5)	3863(5)	75(4)
C(10)	4935(4)	8316(6)	4605(4)	62(3)
C(11)	4185(4)	9047(5)	4609(3)	52(3)
C(12)	3431(4)	9080(4)	3870(4)	39(2)
C(13A)	824(5)	9098(10)	2076(9)	39(2)
C(14A)	753(5)	9735(10)	2848(8)	44(3)
C(15A)	-118(7)	10250(9)	2937(7)	44(3)
C(16A)	-919(5)	10127(9)	2255(8)	41(3)
C(17A)	-849(6)	9490(9)	1484(7)	44(3)
C(18A)	22(7)	8975(9)	1394(7)	47(3)
C(13B)	852(5)	9165(10)	1989(9)	39(2)
C(14B)	516(7)	9464(10)	2814(7)	44(3)
C(15B)	-374(7)	9983(9)	2786(6)	44(3)
C(16B)	-927(5)	10204(9)	1934(8)	41(3)
C(17B)	-591(7)	9906(9)	1109(6)	44(3)
C(18B)	299(7)	9387(9)	1137(7)	47(3)
C(19)	383(3)	5496(4)	3019(4)	34(2)
C(20)	121(4)	4842(4)	3718(3)	39(2)
C(21)	-776(4)	4328(4)	3597(4)	50(2)
C(22)	-1411(3)	4467(5)	2776(4)	51(2)
C(23)	-1150(4)	5120(5)	2077(3)	52(3)
C(24)	-253(4)	5635(4)	2199(3)	43(2)
C(25)	1655(4)	6902(4)	4197(3)	36(2)
C(26)	2498(3)	6920(4)	4838(4)	43(2)
C(27)	2547(3)	7561(5)	5624(3)	43(2)
C(28)	1752(4)	8184(4)	5768(3)	47(2)
C(29)	908(4)	8165(4)	5127(4)	66(3)
C(30)	860(3)	7524(4)	4341(4)	56(3)

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**Table S8.** Anisotropic displacement parameters ( $\text{\AA}^2 \cdot 10^3$ ) for  $\text{Ph}_3\text{PbO}_2\text{PPh}_2$  (**3**). The anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U^{11} + \dots + 2hk a^* b^* U^{12}]$

	U11	U22	U33	U23	U13	U12
Pb	46(1)	23(1)	37(1)	0(1)	2(1)	3(1)
P	37(1)	24(1)	41(1)	3(1)	3(1)	-1(1)
O(1)	49(3)	24(3)	47(3)	7(3)	17(3)	2(3)
O(2)	43(3)	20(3)	56(4)	0(3)	2(3)	2(2)
C(1)	42(5)	25(4)	45(5)	2(4)	-4(4)	-2(4)
C(2)	39(4)	28(4)	40(4)	-5(4)	-2(3)	5(4)
C(3)	46(5)	42(5)	41(5)	10(4)	5(4)	4(4)
C(4)	46(5)	51(5)	48(5)	-14(5)	0(4)	-3(5)
C(5)	55(6)	33(5)	63(6)	-10(4)	12(5)	-7(4)
C(6)	58(5)	31(5)	38(4)	1(4)	12(4)	-3(4)
C(7)	40(4)	30(4)	35(4)	4(4)	-5(3)	-3(4)
C(8)	63(7)	62(6)	55(6)	0(5)	10(5)	34(5)
C(9)	64(7)	91(9)	66(7)	19(7)	-3(6)	40(7)
C(10)	47(6)	83(8)	52(6)	21(6)	-7(5)	-9(6)
C(11)	48(6)	63(6)	41(5)	5(5)	-6(4)	-8(5)
C(12)	33(5)	39(5)	42(5)	-10(4)	-2(4)	4(4)
C(19)	39(5)	28(4)	36(4)	-1(3)	4(4)	-3(4)
C(20)	42(5)	32(4)	44(5)	-2(4)	6(4)	-4(4)
C(21)	58(6)	34(5)	56(6)	-4(4)	5(5)	-8(4)
C(22)	38(5)	48(6)	67(6)	-18(5)	8(5)	-9(4)
C(23)	44(6)	62(6)	50(6)	-12(5)	-1(4)	3(5)
C(24)	41(5)	44(5)	43(5)	-1(4)	6(4)	6(4)
C(25)	40(5)	28(4)	38(4)	5(3)	-7(4)	-4(3)
C(26)	41(5)	50(5)	38(5)	7(4)	2(4)	2(4)
C(27)	43(5)	49(5)	35(5)	-5(4)	-2(4)	-6(4)
C(28)	55(6)	36(5)	46(5)	-10(4)	-3(4)	2(4)
C(29)	60(6)	48(6)	85(8)	-23(5)	-14(6)	20(5)
C(30)	56(6)	27(4)	78(7)	-15(5)	-11(5)	6(4)

**Table S9.** Selected bond lengths (Å) and angles (°) for Ph<sub>3</sub>PbO<sub>2</sub>PPh<sub>2</sub> (**3**).

Pb-C(7)	2.175(4)	C(13A)-Pb-O(1)	90.0(4)
Pb-C(13B)	2.168(4)	C(1)-Pb-O(1)	89.31(18)
Pb-C(13A)	2.192(4)	O(2) <sup>#1</sup> -Pb-O(1)	174.82(18)
Pb-C(1)	2.189(4)	O(1)-P-O(2)	117.6(4)
Pb-O(2) <sup>#1</sup>	2.375(5)	O(1)-P-C(19)	107.6(3)
Pb-O(1)	2.404(5)	O(2)-P-C(19)	108.8(3)
P-O(1)	1.496(6)	O(1)-P-C(25)	110.5(3)
P-O(2)	1.499(5)	O(2)-P-C(25)	105.9(3)
P-C(19)	1.809(4)	C(19)-P-C(25)	105.9(3)
P-C(25)	1.811(4)	P-O(1)-Pb	138.6(3)
O(2)-Pb <sup>#2</sup>	2.375(5)	P-O(2)-Pb <sup>#2</sup>	143.1(3)
		C(2)-C(1)-Pb	121.4(3)
C(7)-Pb-C(13B)	124.7(4)	C(6)-C(1)-Pb	118.5(3)
C(7)-Pb-C(13A)	122.3(4)	C(8)-C(7)-Pb	119.5(3)
C(7)-Pb-C(1)	115.9(2)	C(12)-C(7)-Pb	120.5(3)
C(13B)-Pb-C(1)	119.2(4)	C(14A)-C(13A)-Pb	116.6(6)
C(13A)-Pb-C(1)	121.8(4)	C(18A)-C(13A)-Pb	123.2(6)
C(7)-Pb-O(2) <sup>#1</sup>	92.2(2)	C(14B)-C(13B)-Pb	122.8(6)
C(13B)-Pb-O(2) <sup>#1</sup>	81.6(4)	C(18B)-C(13B)-Pb	117.1(6)
C(13A)-Pb-O(2) <sup>#1</sup>	84.9(4)	C(20)-C(19)-P	118.1(3)
C(1)-Pb-O(2) <sup>#1</sup>	92.54(19)	C(24)-C(19)-P	121.8(3)
C(7)-Pb-O(1)	91.4(2)	C(26)-C(25)-P	119.8(3)
C(13B)-Pb-O(1)	93.3(4)	C(30)-C(25)-P	120.2(3)

Symmetry transformations used to generate equivalent atoms: #1  $-x+1/2, y+1/2, -z+1/2$ ;

#2  $-x+1/2, y-1/2, -z+1/2$

**Table S10.** Selected IR and Raman frequencies ( $\text{cm}^{-1}$ ) of **1**, **2**, and **3**

[(Ph <sub>3</sub> Pb) <sub>2</sub> Cl(O <sub>2</sub> AsMe <sub>2</sub> )] ( <b>1</b> )		[Ph <sub>2</sub> ClPb(O <sub>2</sub> AsMe <sub>2</sub> )] ( <b>2</b> )		[Ph <sub>3</sub> Pb(O <sub>2</sub> PPh <sub>2</sub> )] ( <b>3</b> )		Assignment
IR	Raman	IR	Raman	IR	Raman	
				1131vs	1131wm	v <sub>as</sub> (PO <sub>2</sub> )
				1034s	1040w	v <sub>s</sub> (PO <sub>2</sub> )
995s	1001vs	992s	998vs	996s	1003vs	Ph ring breathing
870s-vs		880m-s	872vw			v <sub>as</sub> (AsO <sub>2</sub> )
841vs		847m-s				v <sub>s</sub> (AsO <sub>2</sub> )
632m		638m				v <sub>as</sub> (AsC <sub>2</sub> )
601vw	605w	602w	607m-s			v <sub>s</sub> (AsC <sub>2</sub> )
224s	230m		230m	230m-s	325w	v <sub>as</sub> (PbPh)
203s	200s-vs	214m	206s-vs		205s	v <sub>s</sub> (PbPh)
180w?		173s	177s			v (PbCl)

**Table S11.** Characteristic ions formed in the 70 eV mass spectra of **1**, **2**, and **3**.

	[(Ph <sub>3</sub> Pb) <sub>2</sub> ClO <sub>2</sub> AsMe <sub>2</sub> ] ( <b>1</b> )	[Ph <sub>2</sub> ClPbO <sub>2</sub> AsMe <sub>2</sub> ] ( <b>2</b> )	[Ph <sub>3</sub> PbO <sub>2</sub> PPh <sub>2</sub> ] ( <b>3</b> )
Ph <sub>2</sub> PbO <sub>2</sub> PPh <sub>2</sub> <sup>+</sup>			579(13) 502?
Ph <sub>2</sub> PbO <sub>2</sub> AsMe <sub>2</sub> <sup>+</sup>	499(6)		
Ph <sub>3</sub> Pb <sup>+</sup>	439(11)	439(10)	439(60)
PbO <sub>2</sub> PPh <sub>2</sub> <sup>+</sup>			425(10)
Ph <sub>2</sub> ClPb <sup>+</sup>	397(1)?	397(9)	
PbO <sub>2</sub> AsMe <sub>2</sub> <sup>+</sup>	345(6) 315(3)		
PhPb <sup>+</sup>	285(11)	285(18)	285(42)
PbCl <sup>+</sup> , O <sub>3</sub> As <sub>2</sub> Me <sub>3</sub> <sup>+</sup>		243(6)	
	230(4)		
O <sub>2</sub> As <sub>2</sub> Me <sub>3</sub> <sup>+</sup>		227(4)	
Pb <sup>+</sup>	208(9)	208(17)	208(30)
Ph <sub>2</sub> <sup>+</sup>	154(100)	154(100)	154(100) 126 or 127(4)
AsO <sup>+</sup>	91(5)	91(5)	
PhH <sup>+</sup>	78(69)	78(90)	78(62)