

Reactions of Dithioxo-1,3,2 λ^5 ,4 λ^5 -dithiadiphosphetanes with Triorganolead Derivatives

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ABSTRACT

The reactions of Lawesson's reagent 1a and its 4-ethoxy homologue 1b with triethyl- and triphenyl(alkoxy)plumbanes 2a,b and -(alkylthio)plumbanes 4a,b were studied. On the basis of these reactions, novel, advantageous methods of synthesizing S-triethyl and triphenylplumbyl derivatives of aryldithio- and trithiophosphonic acids 3a-d and 5a,b were developed. © 1997 John Wiley & Sons, Inc.

INTRODUCTION

The chemistry of organoelement derivatives of main group IV elements is quite well developed. However, the synthesis and properties of silicon, germanium, tin, and lead derivatives of tetracoordinated phosphorus thioacids have been studied only to various degrees. Organothiophosphorus derivatives of silicon mainly have been studied [1–8]. The chemistry of silyldithiophosphates has been studied in detail [1,4,6]. They are used as intermediates for the synthesis of organothiophosphorus compounds [1]. Some S-triorganotin dithio- and tetrathiophosphates are used as pesticides and additives for lubricants [9,10]. However, organogermyl dithiophosphates

had not been given due consideration [11–13]. Besides, organothiophosphorus derivatives of lead appear to be limited to dithiophosphato moieties [6,14,15]. Also, organolead dithio- and trithiophosphonates containing a P–C bond remain unknown.

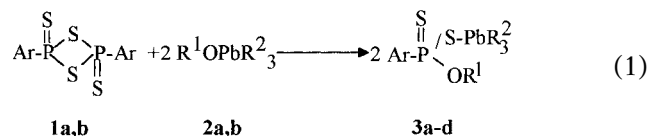
The common methods of synthesizing these compounds are usually based on the reactions of dithiophosphoric acids or other phosphorus(V) thioacids or their salts with silylating, germylating, stannylating and plumbylating agents [2–15]. These techniques, however, are multistep procedures. Alternatively, over the past few years, we have been involved in developing new synthetic routes for organoelement derivatives of phosphorus(V) thioacids. We have recently developed facile and efficient methods for the synthesis of S-triorganylsilyl-, germyl- and stannyl dithio-, trithio- and tetrathiophosphates, and also aryldithio- and trithiophosphonates on the basis of reactions involving phosphorus sulfide (P₄S₁₀) and 1,3,2,4-dithiadiphosphetane-2,4-disulfides [16–29]. We have tried to find the limits of these methods and have extended the reactions of 1,3,2,4-dithiadiphosphetane-2,4-disulfides to organolead derivatives. In this article, convenient methods are presented for the synthesis of S-triorganylplumbyl aryldithio- and trithiophosphonates.

RESULTS AND DISCUSSION

2,4-Bis(4-alkoxyphenyl)-2,4-dithioxo-1,3,2 λ^5 ,4 λ^5 -dithiadiphosphetanes 1a,b react with triethyl- and triphenyl(alkoxy)plumbanes 2a,b in anhydrous ben-

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zene at 20°C for 1.5–4.5 hours to give new triethyl- and triphenyllead(IV) S-(O-alkyl-4-methoxyphenyl) dithiophosphonates **3a–d** (Reaction 1, Tables 1 and 2).



Ar = 4-MeOC₆H₄ (**1a**, **3a**), R¹ = i-Bu, R² = Et (**2a**, **3a**)

Ar = 4-EtOC₆H₄ (**1b**, **3b**), R¹ = i-Bu, R² = Et (**2a**, **3b**)

Ar = 4-MeOC₆H₄ (**1a**, **3c**), R¹ = Me, R² = Ph (**2b**, **3c**)

Ar = 4-EtOC₆H₄ (**1b**, **3d**), R¹ = Me, R² = Ph (**2b**, **3d**)

This method is characterized by a minimum of steps, simple operation, mild conditions, high yields of products (61–98%, Table 2) and formation of no by-products. Before our work in this field, compounds with O₂P(S)SPb structural fragments were known only among organolead derivatives of phosphorus(V) thioacids [6,14,15]. Our approach allowed

us to obtain new types of organothiophosphorus derivatives of lead **3** containing the CP(S)(OC)SPb structural fragment. This reaction served as a preparative method for obtaining the products **3**. They were yellow pastes (**3a,c,d**) or red, oily liquids (**3b**). All these compounds were soluble in common organic solvents. However, they showed a tendency to decompose at elevated temperature. Thus, we have not managed to isolate **3** in pure form by high vacuum distillation, as these compounds decomposed even when a falling-film distillation apparatus was used. The product **3b** was purified by column chromatography (see Experimental). However, **3a** and **3c,d** decomposed even when column chromatography was used. Therefore, **3a** and **3c,d** were purified by filtration and subsequent evaporation under reduced pressure. These compounds were identified only in crude forms.

In continuation of our approach, we have managed to involve lead alkylmercaptides in the reaction with 1,3,2,4-dithiadiphosphetane-2,4-disulfides. Thus, we have shown that the reactions of **1a,b** with

TABLE 1 Experimental Data and Yields of the Products Obtained

	Initial Compounds Quantity, g (mmol)	Reaction Conditions Temp. (°C) Time (h)	Product Yield, g(%)
1a	10.4 (25.7)/ 2a 18.9 (51.4)	20/1.5 10 mL C ₆ H ₆	3a 19.0 (65) ^a
1b	7.9 (18.3)/ 2a 13.4 (36.5)	20/2 10 mL C ₆ H ₆	3b 13.5 (63) ^a ; 0.3 ^b
1a	2.4 (5.9)/ 2b 5.6 (11.9)	20/4.5 ^c 10 mL C ₆ H ₆	3c 4.9 (61) ^a
1b	2.7 (6.3)/ 2b 5.9 (12.5)	20/4.5 ^c 5 mL C ₆ H ₆	3d 8.5 (98) ^a
1b	10.3 (23.8)/ 4a 18.3 (47.8)	20/2 100 mL C ₆ H ₆	5a 7.0 (24) ^a
1a	2.2 (5.4)/ 4b 5.7 (11.1)	20/2 100 mL C ₆ H ₆	5b 6.0 (77) ^a

^aYield of crude product.

^bYield of isolated product.

^cThe reaction mixtures were stored for 4.5 months at 20°C.

TABLE 2 Physical, Analytical, and ³¹P NMR Data of the Products Obtained

Prod.	R _f Value (C ₆ H ₆) Silufof	n _D ²⁰	Molecular Formula (Mol. mass)	Found/ Calculated, % P	³¹ P NMR, δ (C ₆ H ₆)
3a	0.88		C ₁₇ H ₃₁ O ₂ PPbS ₂ (569.4)	4.94 5.44	100.5
3b	0.92	1.5465	C ₁₈ H ₃₅ O ₂ PPbS ₂ (585.5)	5.19 5.29	100.2
3c	0.75		C ₂₆ H ₂₅ O ₂ PPbS ₂ (671.4)	4.38 ^a 4.61	101.2
3d	0.83		C ₂₇ H ₂₇ O ₂ PPbS ₂ (685.4)	4.92 4.52	102.9
5a	0.94	1.6028	C ₁₈ H ₃₃ OPPbS ₃ (599.4)	5.49 5.17	82.1
5b	0.77	1.6919	C ₂₈ H ₂₉ OPPbS ₃ (715.4)	4.60 4.33	76.7

^aFound, %: C, 46.93; H, 3.90; S, 9.39. Calculated. %: C, 46.76; H, 3.76; S, 9.52.

TABLE 3 IR Data of the Products Obtained

Prod.	ν , cm^{-1}
3a	3085, 3070, 3050 ν (Ar); 2960, 2920, 2890, 2045 ν (CH ₃ as, s; CH ₂ as, s); 1602, 1510, 1470 ν (C=C, Ar); 1399, 1375 δ (Me ₂ C gem s); 695, 666, 645, 625 ν (P=S, PS ₂ as); 560, 485 ν (Pb-C, P-S, PS ₂ s); 460 ν (Pb-S).
3b	3080, 3040 ν (=C-H, Ar), 2980, 2940, 2880 ν (CH ₃ as, s; CH ₂ as, s); 1600, 1505, 1480 ν (C=C, Ar); 1375, 1398 δ (Me ₂ C gem s); 686, 647, 625 ν (P=S, PS ₂ as); 512 ν (Pb-C, P-S, PS ₂ s); 465 ν (Pb-S).
3c	3090, 3070, 3050 ν (=C-H, Ar); 2940, 2840 ν (CH ₃ as, s; CH ₂ as, s); 1600, 1505, 1440 ν (C=C, Ar); 685, 625 ν (P=S, PS ₂ as); 490 ν (Pb-C, P-S, PS ₂ s); 450 ν (Pb-S).
3d	3070 cp, 3056 cp ν (=C-H, Ar); 2990 cp, 2945 cp, 2900 III, 2850 cp ν (CH ₃ as, s; CH ₂ as, s); 1600 c, 1505 cp, 1445 cp ν (C=C, Ar); 695 cp, 685 cp, 645 cp, 625 cp ν (P=S, PS ₂ as); 548 c.III ν (Pb-C, P-S, PS ₂ s); 450 cp ν (Pb-S).
5a	3066 ν (=C-H, Ar); 2960, 2930, 2890, 2870 ν (CH ₃ as, s; CH ₂ as, s); 1590, 1495, 1460 ν (C=C, Ar); 1395, 1370 δ (Me ₂ C gem s); 698, 677, 665, 626 ν (P=S, PS ₂ as); 465, 445 ν (Pb-C, P-S, PS ₂ s); 465 ν (Pb-S).
5b	3065, 3050 ν (=C-H, Ar); 2970, 2930, 2870, 2840 ν (CH ₃ as, s; CH ₂ as, s); 1590, 1500, 1430 ν (C=C, Ar); 695, 684, 622 ν (P=S, PS ₂ as); 547, 515 ν (Pb-C, P-S, PS ₂ s); 450 ν (Pb-S).

TABLE 4 ¹H NMR Data of the Products Obtained

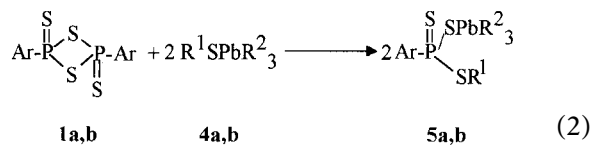
Prod.	δ , J[Hz]
3a	0.77 (t, 9H, CH ₃ CH ₂ Pb, ³ J _{HH} 7.2); 0.78 (d, 6H, CH ₃ CHCH ₂ O, ³ J _{HH} 6.0); 1.55–2.91 (m, CH ₃ CH ₂ Pb, 6H, CH ₃ CHCH ₂ O, 1H); 3.20 (s, 3H, CH ₃ OC ₆ H ₄); 3.98 (d.d, 2H, CH ₃ CHCH ₂ OP, ³ J _{HH} 7.2, ³ J _{PH} 7.2); 6.57 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.0); 7.98 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 14.0).
3b	0.8 (t, 9H, CH ₃ CH ₂ Pb, ³ J _{HH} 7.0); 0.9 (d, 6H, CH ₃ CHCH ₂ O, ³ J _{HH} 6.0); 1.00 (t, 3H, CH ₃ CH ₂ O, ³ J _{HH} 7.0); 1.62–3.13 (m, CH ₃ CH ₂ O, 2H, CH ₃ CHCH ₂ O, 1H); 3.46 (q, 6H, CH ₃ CH ₂ Pb, ³ J _{HH} 7.0); 4.00 (d.d, 2H, CH ₃ CHCH ₂ OP, ³ J _{HH} 6.0, ³ J _{PH} 8.0); 6.65 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.0); 8.07 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 14.0).
3c	3.24 (d, 3H, CH ₃ OP, ³ J _{PH} 16.0); 3.57 (s, 3H, CH ₃ OC ₆ H ₄); 6.53 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.0); 6.83–7.67 (m, 15H, C ₆ H ₅); 7.90 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 15.0).
3d	1.30 (t, 3H, CH ₃ CH ₂ O, ³ J _{HH} 7.0); 3.33 (d, 3H, CH ₃ OP, ³ J _{PH} 16.0); 3.87 (q, 2H, CH ₃ CH ₂ O, ³ J _{HH} 7.0); 6.57 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.0); 6.87–7.62 (m, 15H, C ₆ H ₅); 7.90 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 15.0).
5a	0.85–1.02 (m, 9H, CH ₃ CH ₂ Pb; 6H, CH ₃ CHCH ₂ S; 3H, CH ₃ CH ₂ O); 1.52–2.05 (m, 1H, CH ₃ CHCH ₂ SP); 2.52 (q, 6H, CH ₃ CH ₂ Pb, ³ J _{HH} 7.0); 2.85 (d.d, 2H, CH ₃ CHCH ₂ SP, ³ J _{HH} 7.5, ³ J _{PH} 14.0); 4.00 (q, 2H, CH ₃ CH ₂ O, ³ J _{HH} 7.0); 6.81 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.5); 7.84 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 14.5).
5b	0.57 (t, 3H, CH ₃ CH ₂ CH ₂ S, ³ J _{HH} 7.2), 1.00–1.65 (m, 2H, CH ₃ CH ₂ CH ₂ SP); 2.67 (d.t, 2H, CH ₃ CH ₂ CH ₂ SP, ³ J _{HH} 7.2, ³ J _{PH} 14.0); 3.12 (s, 3H, CH ₃ OC ₆ H ₄); 6.48 81 (d.d, 2H, 3,5-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ⁴ J _{PH} 3.0); 6.79–7.70 (m, 15H, C ₆ H ₅); 7.96 (d.d, 2H, 2,6-H ₂ C ₆ H ₂ , ³ J _{HH} 9.0, ³ J _{PH} 15.0).

TABLE 5 Mass Spectral Data of the Products Obtained

Prod.	$i\text{-C}_4\text{H}_{10}^+$ m/e (I_{rel} %)
3a ^a	555 [M + H - Me] ⁺ (25); 483 [M + H - 3Et] ⁺ (13); 334 [M + H - EtPb] ⁺ (10); 306 [M + 2H - Et ₂ Pb] ⁺ (100); 276 [M + 2H - Et ₃ Pb] ⁺ (34).
3a ^b	522 [M - Me - S] ⁺ (50).
3b ^a	529 [M + H - 2Et] ⁺ (15).
3b ^b	586 [M] ⁺ (40); 557 [M - Et] ⁺ (100); 321 [M - Et ₂ Pb] ⁺ (100); 292 [M - Et ₃ Pb] ⁺ (15); 265 [M - Et - MeOC ₆ H ₄ P(S)S(OBu)] ⁺ (100); 232 [M - OBu- <i>i</i> - Et ₃ Pb] ⁺ (73).
3c ^a	439 [M + H - MeOC ₆ H ₄ PS ₂ - OMe] ⁺ (15); 285 [M + H - MeOC ₆ H ₄ PS ₂ - OMe - 2Ph] ⁺ (15); 139 [M + H - S - Ph ₃ Pb - OMe] ⁺ (48).
3c ^b	202 [M - Ph ₃ Pb - OMe] ⁺ (100); 107 [M - 2S - Ph ₃ Pb - OMe] ⁺ (14).
5a ^a	543 [M + H - Bu- <i>i</i>] ⁺ (24); 528 [M + H - Me - Bu- <i>i</i>] ⁺ (10); 499 [M + H - Me - Et - Bu- <i>i</i>] ⁺ (10); 455 [M + H - 3Et - Bu- <i>i</i>] ⁺ (10); 399 [M + H - 3Et - Bu- <i>i</i> - 2S] ⁺ (10).
5a ^b	469 [M - Me - 2Et - Bu- <i>i</i>] ⁺ (10); 334 [M - Et ₂ Pb] ⁺ (100); 305 [M - Et ₃ Pb] ⁺ (10); 290 [M - Me - Et ₃ Pb] ⁺ (55).
5b ^a	395 [M + H - 3Ph - Pr - Me - S] ⁺ (10); 108 [M + H - P(S)SPbPh ₃ (SPr)] ⁺ (100).
5b ^b	438 [M - MeOC ₆ H ₄ PS ₂ - SPr] ⁺ (20); 245 [M - Ph ₃ Pb - S] ⁺ (25); 230 [M - Me - Ph ₃ Pb - S] ⁺ (10); 170 [M - Ph ₃ Pb - Pr - S] ⁺ (10).

^aChemical ionization, 100 eV.^bElectron impact, 70 eV.

triethyl- and triphenyl(alkylthio)plumbanes **4a,b** give triethyl- and triphenyllead(IV) S-(S'-alkyl-4-methoxyphenyltrithiophosphonates) **5a,b** (Reaction 2, Tables 1 and 2).



Ar = 4-EtOC₆H₄ (**1b**, **5a**), R¹ = *i*-Bu, R² = Et (**4a**, **5a**)

Ar = 4-MeOC₆H₄ (**1a**, **5b**), R¹ = Pr, R² = Ph (**4b**, **5b**)

Reaction 2 is exothermic and occurs at room temperature in anhydrous benzene in a great dilution (2.2–48 mmol of reagents in 100 mL of solvent), resulting in the formation of compounds **5** in 24–77% yields (Tables 1 and 2). We believe that compounds **5** are new types of organothiophosphorus derivatives of lead containing the CP(S)(SC)SPb structural fragment, as also found in compounds **3**. Compounds **5** are yellow, oily, viscous liquids. They are nonvolatile even under reduced pressure, stable at room temperature, but tend to decompose at high temperatures and even when column chromatography is used in attempted purification. Thus, we purified **5** by filtration, centrifugation, and with subsequent evaporation under high vacuum. These compounds are characterized only in crude forms (Tables 1 and 2).

Lead trithiophosphonates **5a,b** are more highly moisture sensitive than lead dithiophosphonates **3a,b**. The thermal stabilities of **5a,b** are lower than those of **3a,b**. In general, according to our earlier experimental data [24,26], the thermal stabilities of silicon, germanium, tin, and lead aryltrithiophosphonates decrease in this order.

The structures of **3** and **5** were confirmed by IR (Table 3), ¹H (Table 4), ³¹P NMR (Table 2), and mass spectra (Table 5), as well as by elemental analyses (Table 2). Bands of medium intensities present in the region ν 465–450 cm⁻¹ in the IR spectra (Table 3) are due to Pb–S valence vibrations. Bands in the region ν 695–685 and 560, 490–485 cm⁻¹ are assigned to P=S, PS₂ as and Pb–C, P–S, PS₂ s valence vibrations. The ¹H NMR spectra of **3** (Table 4) show the characteristic resonances due to the presence of alkoxy groups attached to the phosphorus atom. The CH₃OP protons appear as a doublet in the region of δ 3.24 with ³J_{PH} 16.0 Hz for **3c**. A doublet of doublets observed in the ¹H NMR spectrum of **5a** in the range of δ = 2.85 has been assigned to the methylene protons of the thioisobutyl group (CH₃)₂CHCH₂SP with

³J_{PH} = 14.0 Hz. Similarly, the indicated methylene protons of the thiopropyl group CH₃CH₂CH₂SP of **5b** appear as a doublet of triplets in the range of δ = 2.67 with the same spin coupling constant. The electron impact mass spectra of **3a** (Table 5) exhibit the mass peaks *m/e* 586, 557, 321, and 292 that may be attributed to the ions [M]⁺, [M – Et]⁺, [M – Et₂Pb]⁺, and [M – Et₃Pb]⁺, respectively, of **3b**.

The ³¹P NMR resonances (Table 2) of compounds **3** show appreciable low field shifts (of about 15–25 ppm) with respect to the corresponding free aryldithiophosphonic acids [30]. We have shown that the ³¹P NMR resonances of trimethylsilyl, triphenylgermyl, trimethylstannyl, and triethylplumbyl O-alkyl-4-methoxyphenyl dithiophosphonates (δ = 87.6, 89.6, 98, and 100.5, respectively) are shifted toward low field when passing from silicon to lead dithiophosphonates. Similarly, the ³¹P resonances of tri-thiophosphonates of triorganylsilicon, germanium, tin, and lead (**5a,b**) show a tendency to shift to low field (δ = 69–71, 67.4–71.2, 74.5, and 76.7–82.1, respectively) [16–29].

It is noteworthy to compare the reactivity of reagents in Reactions 1 and 2. Reaction 1 proceeds rather slowly. Lawesson's reagent **1a** has proved to be more reactive than its 4-ethoxy homologue **1b** toward lead alkoxides **2**. The reactivity of triethyl (isobutoxy)plumbane **2a** is higher than that of triphenyl-(methoxy)plumbane **2b** in Reaction 1. It is remarkable that the reaction of lead alkoxides **2** with **1** proceeds more easily than that of lead alkylmercaptides **4**.

Reactions 1 and 2 are of interest from the point of view of preparative organophosphorus chemistry. Besides, they have a fundamental significance as they lead to new types of organoelement derivatives of phosphorus(V) thioacids containing the structural fragment P(S)SPb.

EXPERIMENTAL

General Data

The ³¹P NMR spectra were recorded with a Bruker MSL 400 (162 MHz), WM 250 (101.3 MHz), and CXP-100 (36.5 MHz) instruments in C₆H₆. The ¹H NMR spectra were taken on a Varian T-60 (60 MHz) spectrometer in C₆D₆ with CH₂Cl₂ or (Me₃Si)₂O as internal references. The IR spectra were obtained in KBr pellets with a UR-20 infrared spectrophotometer. Mass spectra (EI, 70 eV; CI, 100 eV) were determined on an M 80 B Hitachi chromatomass spectrometer.

Triethyllead S-(O-Isobutyl)-4-ethoxyphenyldithiophosphonate **3b**. *Typical Procedure*

Compound **1b** [7.9 g (18.3 mmol)] was added portionwise with stirring at 20°C to 13.4 g (36.5 mmol) of **2a** in 10 mL of anhydrous benzene, and stirring was continued for 2 hours at 20°C. The mixture was filtered and the filtrate evaporated at reduced pressure (0.02 mm Hg) at 40°C for 2 hours to give 13.5 g (63%) of crude **3b**. Part (2.8 g) of the crude **3b** was chromatographed on an Al₂O₃ column with benzene as an eluant to yield 0.3 g of pure **3b** (see Tables 1–5).

The products **3a**, **3c,d**, and **5a,b** were obtained similarly (see Tables 1–5) but without isolation by column chromatography. The reaction mixtures of **1a,b** with **2b** could be stored for up to 4.5 months in sealed tubes at 20°C. The products **5a,b** were purified by centrifugation.

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