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Reactions of Dithioxo-1,3,2λ^5,4λ^5-dithiadiphosphetanes with Antimony(III) Alkoxides

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ABSTRACT: *New antimony(III) derivatives of dithiophosphonic and trithiophosphoric acids* **3a–c** *and* **5** *were obtained by the reactions of Lawesson's reagent* **1a**, *its homologue* **1b**, *and the isobutyl homologue of Davy's reagent* **4** *with antimony(III) alkoxides* **2a–c**. © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 399–403, 1999

INTRODUCTION

Organic derivatives of main group V elements of tetracoordinated phosphorus thioacids with the P(S)SE (E = As, Sb, Bi) structural fragment appear to have been limited to dithiophosphato and dithiophosphinato moieties [1–15]. We deemed it to be necessary to synthesize new types of organothiophosphorus compounds with the P(S)SE structural fragment with widely varied substituents on the phosphorus atom in order to develop a series of fundamental studies, such as chemical behavior, complexation, influence of heteroatoms in the fragment P(S)SE, tautomerism, conjugation, stereochemistry, etc. Different modes of bonding of dithiophosphato and dithiophosphinato moieties (e.g., monodentate, bridging/chelating, bidentate) in the complex formation with antimony have been elucidated by physicochemical techniques [2,5–8,11–13,15]. Antimony(III) dithiophosphates and dithiophosphinates have been obtained by the reactions of dithiophosphoric and dithiophosphinic acids, or their salts, with antimony(III) oxides, halides, acetates, and triphenylantimony [1–14]. We have now developed a new approach to antimony(III) derivatives of pentavalent phosphorus thioacids having the P(S)SSb structural fragment by the use of 1,3,2,4-dithiadiphosphetane-2,4-disulfides **1a,b** and **4**.

RESULTS AND DISCUSSION

Over the past few years, we have been involved in developing new synthetic routes for organic derivatives of main group V elements of tetracoordinated phosphorus thioacids. We have recently developed convenient methods for the synthesis of S-organoarsenic(III) derivatives of tetrathiophosphoric, trithiophosphonic, and dithiophosphonic acids on the basis of the reactions of tetraphosphorus decasulfide and 1,3,2,4-dithiadiphosphetane-2,4-disulfides with alkylthioarsenites [16] and alkyl and phenyl ethers of arsinous acids, O,O-dialkyl(phenyl)arsonites, and triakylarsenites [17]. We have now extended this approach to antimony(III) derivatives having Sb–O bonds.

We have now found that 1,3,2,4-dithiadiphosphetane-2,4-disulfides **1a,b** react with antimony(III)

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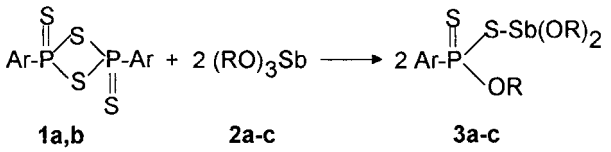
alkoxides **2a–c** at 20–70°C for 4–10 hours to give S-dialkoxyantimony(III) O-alkylaryldithiophosphonates **3a–c** (Reaction 1, Tables 1–5).



Ar = 4-$MeOC_6H_4$, R = Pr-i (**1a, 2a, 3a**);

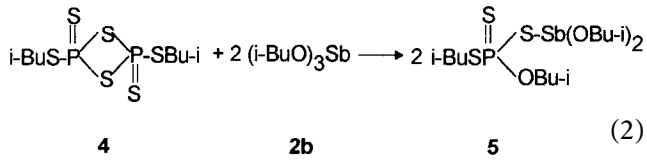
Ar = 4-$MeOC_6H_4$, R = Bu-i (**1a, 2b, 3b**);

Ar = HO-(C6H2)-, R = Bu-sec. (**1b, 2c, 3c**) (1)

Reaction 1 proceeds in anhydrous benzene or with no solvent at room temperature (in case of **2b** and **2c**) and at 60–70°C (for **2a**). Our approach allowed us to obtain new types of antimony(III) derivatives of pentavalent phosphorus thioacids containing the CP(S)(OC)SSb structural fragment. Products **3a,b** are yellow, oily liquids, whereas **3c** is a crystalline solid. All of these compounds are soluble in common organic solvents. The product **3b** was purified both by use of a falling-film distillation and by column chromatography (see Experimental).

It should be emphasized that, formally, **3a–c** are the products of the insertion of the monomeric unit, ZPS_2 [Z = 4-$MeOC_6H_4$, 3,5-$(C_4H_9)_2$-4-HO-C_6H_2], of **1a,b** into the Sb–O bond of **2a–c**. It is noteworthy that only one alkoxy group of **2a–c** takes part in the reaction with **1a,b** under mild conditions. Other alkoxy groups remain attached to the antimony atom in **3a–c**.

In continuation of our approach, we have managed to involve other 1,3,2,4-dithiadiphosphetane-2,4-disulfides in the reactions with antimony(III) compounds containing the Sb–O bond. Thus, we have shown that the reaction of the isobutyl homologue of Davy's reagent **4** with **2b** at 20°C for 3 hours yielded S-diisobutoxyantimony(III) O-isobutyl-S-isobutyltrithiophosphate **5** (Reaction 2, Tables 1–5).



(2)

We believe that compound **5** is a new type of antimony(III) derivative of pentavalent phosphorus thioacids containing the SP(S)(OC)SSb structural fragment. The product **5** as well as **3b** was obtained as a yellow liquid and isolated both by use of a falling-film distillation and by column chromatography (see Experimental).

The structures of **3** and **5** were confirmed by IR (Table 3), ^{1}H (Table 4), and ^{31}P NMR (Table 2) and mass spectroscopy (Table 5), as well as by elemental analyses (Table 2). The ^{31}P NMR spectra of **3a–c** and **5** (Table 2) reveal signals in the region δ = 83.0–85.3. These resonances are shifted toward high field in comparison with the ^{31}P NMR data of $(RO)_2PS_2SbPh_2$ (δ = 91.4–99.4) [12], whereas the ^{31}P NMR spectrum of $Et_2PS_2Sb(C_6H_4$-Me-4$)_2$ is known to show a signal at δ = 76.2 [12]. Thus, the ^{31}P chemical shift values of antimony(III) derivatives of pentavalent phosphorus thioacids are shifted to low field when passing from the compounds with the $C_2P(S)SSb$ structural fragment to ones with the O(C)P(S)SSb and $O_2P(S)SSb$ structural fragments. This is due to the increase of the chelating nature of the ligands as the amounts of the P–O bonds are increased. Moreover, the ^{31}P NMR spectra of **3a–c** in benzene solutions show a low field shift of δ of about 4.9–5.6 with respect to the corresponding free (O-alkyl)aryldithiophosphonic acids **6a–c** that were obtained by treatment of **1a,b** with the corresponding alcohols [19,20].

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 6.7 (16.6)	/**2a** 9.9 (33.1)	60–70/4	**3a** 6.0 (36)[a]
1a 2.7 (6.7)	/**2b** 4.5 (13.2)	20–4	**3b** 4.8 (66)[a]/1.8 (25)[b]
1a 3.7 (9.2)	/**2b** 6.2 (18.2)	20/4 10 mL C_6H_6	**3b** 7.8 (79)[a]/0.5[c]
1b 13.0 (21.7)	/**2c** 14.8 (43.4)	20/4	**3c** 25.1 (90)[d]
4 3.0 (8.1)	/**2b** 5.5 (16.1)	20/3	**5** 4.5 (52)[a]/1.1 (13)[b]
4 2.4 (6.5)	/**2b** 4.5 (13.2)	20/3	**5** 3.9 (45)[a]/0.8[c]

[a]Yield of crude product.
[b]Yield of product isolated by a falling-film distillation.
[c]Yield of product isolated by column chromatography.
[d]Yield of solid product.

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

Prod.	*Bp, °C (mm Hg)[a] or mp*	n_D^{20}	*Molecular Formula (Mol. mass)*	*Found/Calc. %* Sb	P	*^{31}P NMR δ (C_6H_6)*
3a		1.5959	$C_{16}H_{28}O_4PS_2Sb$ (500.9)	24.62 24.30	6.58 6.18	83.0
3b[b]	135 (0.04)	1.5350	$C_{19}H_{34}O_4PS_2Sb$ (543.0)	22.03 22.42	5.70 5.70	85.3
3c	59–60		$C_{26}H_{48}O_4PS_2Sb$ (641.1)	19.12 18.99	4.64 4.83	85.0[c]
5[d]	120 (0.06)	1.4974	$C_{16}H_{36}O_3PS_3Sb$ (525.0)	22.86 23.19	5.51 5.99	84.7

[a]Temperature of thermal element of a falling-film distillation apparatus.
[b]R_f 0.86 (CH_2Cl_2).
[c]Broad signal.
[d]R_f 0.94 (Et_2O).

TABLE 3 IR Data of the Products Obtained

Prod.	*ν, cm^{-1}*
3a	3073 ν (:CH, Ar); 2980, 2910, 2840, ν (CH_3, CH); 1597, 1570, 1501, 1440 ν (Ar); 1462 δ^{as} (CH_3); 1380, 1373 δ^s (CH_3, gem); 1297, 1260 ν (Ar-O-C); 1184, 1110 ν (*i*-PrO, P-Ar); 1031, 980 ν (PO-C, SbO-C); 840 γ (:CH); 820 ν (P-OC); 678 ν (P=S); 619 ν (Sb-OC); 532 ν^{as} (P-S-Sb), 386 ν^s (P-S-Sb).
3b	3080, 3015 ν (:CH, Ar); 2970, 2945, 2915, 2885, 2847 ν (CH_3, CH_2, CH); 1602, 1579, 1510, 1446 ν (Ar); 1472 δ (CH_3^{as}, CH_2); 1399, 1374 δ^s (CH_3, gem); 1302, 1266 ν (Ar-O-C); 1188, 1123 ν (*i*-BuO, P-Ar); 1030/1020/1010 ν (PO-C, SbO-C); 839 γ (:CH, Ar); 863, 811 ν (PO-C); 687 ν (P=S); 624 ν (Sb-OC); 540 ν^{as} (P-S-Sb).
3c[a]	3620 ν (OH); 3090, 3070, 3040 ν (:C-H, Ar); 2970, 2940, 2915, 2880 ν (CH_3, CH_2, CH); 1580, 1490, 1430 ν (Ar); 1460 δ (CH_3^{as}, CH_2); 1392, 1380, 1364 δ^s (CH_3); 1325 δ (OH), 1242 ν (Ar-OH); 1150, 1125 (*i*-BuO, P-Ar); 1030, 980/968 ν (PO-C, SbO-C); 826 ν (P-OC); 680 ν (P=S); 656 ν^s (*t*-Bu); 620, 600 ν (Sb-O); 510 ν^{as} (P-S-Sb).
5	2970, 2945, 2930, 2880 ν (CH_3, CH_2, CH); 1470 δ (CH_3^{as}, CH_2); 1395, 1370 δ^s (CH_3, gem); 1070/1040/970 ν (PO-C, SbO-C); 878, 800 ν (PO-C), ρ (CH_2); 673 ν (P=S); 580, 555, 535 ν^{as} (P-S-Sb), δ (CCC), δ (CCO).

[a]In vaseline oil.

TABLE 4 ^{1}H NMR Data of Products Obtained

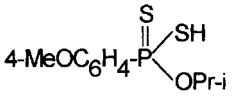
Prod.	*δ, J [Hz]*
3a[a]	0.99 (d, 6H, $\underline{CH_3}CHOP$, $^3J_{HH}$ 6.5); 1.18 (d, 12H, $\underline{CH_3}CHOSb$, $^3J_{HH}$ 6.5); 3.21 (s, 3H, $\underline{CH_3}OC_6H_4$); 6.51 (d. d, 2H, 3,5-$H_2C_6\underline{H_2}$, $^3J_{HH}$ 9.0, $^4J_{PH}$ 3.0); 7.75 (d. d, 2H, 2,6-$\underline{H_2}C_6H_2$, $^3J_{HH}$ 9.0, $^3J_{PH}$ 14.5).
3b[a]	0.72 (d, 6H, $\underline{CH_3}CHCH_2OP$, $^3J_{HH}$ 6.0); 0.73 (d, 12H, $\underline{CH_3}CHCH_2OSb$, $^3J_{HH}$ 6.0); 1.33–1.77 (m, 2H, $CH_3\underline{CH}CH_2OSb$; 1H, $CH_3\underline{CH}CH_2OP$); 3.12 (d, 4H, $CH_3CH\underline{CH_2}OSb$, $^3J_{HH}$ 6.0); 3.15 (s, 3H, $\underline{CH_3}OC_6H_4$; 3.63–3.99 (m, 2H, $CH_3CH\underline{CH_2}OP$); 6.50 (d. d, 2H, 3,5-$\underline{H_2}C_6\underline{H_2}$, $^3J_{HH}$ 9.0, $^4J_{PH}$ 3.0); 7.77 (d. d, 2H, 2,6-$H_2C_6\underline{H_2}$, $^3J_{HH}$ 9.0, $^3J_{PH}$ 15.0).
3c[b,c]	0.86–1.46 (m, 15H, $\underline{CH_3}$); δ_1 1.49 and δ_2 1.50 (two s, 18H, $(\underline{CH_3})_3C$); 1.63–1.86 (m, 6H, $\underline{CH_2}$); 4.72–4.83 (m, 2H, $CH_2\underline{CH}OSb$); 4.83–5.05 (m, 1H, $CH_2\underline{CH}OP$); 5.69 (m, 1H, $\underline{HO}C_6H_2$; 7.86 (d, 2H, 2,6-$C_6\underline{H_2}$, $^3J_{PH}$ 16.2).
5[a]	0.68 (d, 6H, $\underline{CH_3}CHCH_2SP$, $^3J_{HH}$ 6.0); 0.71 (d, 12H, $\underline{CH_3}CHCH_2OSb$, $^3J_{HH}$ 6.0); 0.74 (d, 6H, $\underline{CH_3}CHCH_2OP$, $^3J_{HH}$ 6.0); 1.71–1.85 (m, 4H, $\underline{CH}$); 2.49 (d, 4H, $CH_3CH\underline{CH_2}OSb$, $^3J_{HH}$ 6.0); 2.80 (d. d, 2H, $CH_3CH\underline{CH_2}SP$, $^3J_{HH}$ 6.0, $^3J_{PH}$ 16.0); 3.80 (d. d, 4H, $CH_3\underline{CH}CH_2OP$, $^3J_{HH}$ 6.0 $^3J_{PH}$ 9.0).

[a]In C_6D_6.
[b]In $CDCl_3$.
[c]The mixture of diastereoisomers.

TABLE 5 Mass Spectral Data of the Products Obtained

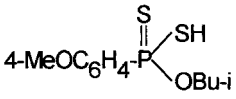
Prod.	*i-CH_4H_{10}, m/e (I_{rel}, %)*
3a[a]	262 [M + H − Sb(OPr−*i*)$_2$]$^+$ (100); 187 [M + M − H − S − Sb(OPr−*i*)$_2$ − Pr−i]$^+$ (100).
3a[b]	410 [M − OPr−*i* − S]$^+$ (12); 367 [M − OPr−*i* − S − Pr−i]$^+$ (10); 186 [M − S − Sb(OPr−*i*)$_2$ − Pr−*i*]$^+$ (100).
3b[a]	276 [M + H − Sb(OBu−*i*)$_2$]$^+$ (80).
3b[b]	486 [M − Bu−*i*]$^+$ (10); 413 [M − Bu−*i* − OBu−*i*]$^+$ (10).
3c[a]	374 [M + H − Sb(OBu−*i*)$_2$]$^+$ (7); 359 [M + H − Me − Sb(OBu−*i*)$_2$]$^+$ (2).
5[b]	452 [M − OBu−*i*]$^+$ (12); 436 [M − Bu−*i* − S]$^+$ (12); 389 [M − SBu−*i* − S − Me]$^+$ (16); 363 [M − 2Bu−*i* − S − O]$^+$ (5); 331 [M − SBu−*i* − S − OBu−*i*]$^+$ (100); 243 [M − SBu−*i* − S − Me − 2*i*−BuO]$^+$ (30).

[a]Chemical ionization, 100 eV.
[b]Electron impact, 70 eV.



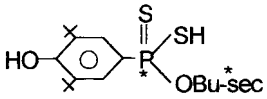
6a

δ = 78.1



6b

δ = 80.1



6c

δ = 79.7 and 79.4

S-Antimony(III) aryldithiophosphonate **3c** was formed as a mixture of diastereoisomers, there being four chiral centers (three number 2-carbon atoms of three sec-butyl groups and the phosphorus atom). The ^{1}H NMR spectrum of **3c** (Table 4) shows two singlets at δ = 1.49 and 1.50 due to the methyl protons of $(CH_3)_3C$ groups. The compound **3c** reveals a broad signal at δ = 85.0 in its ^{31}P NMR spectrum in benzene solution. It is of interest that the corresponding O-sec-butyl-3,5-ditert-butyl-4-hydroxyphenyldithiophosphonic acids **6c** was also formed as a 1:1 mixture of diastereoisomers, there being two chiral centers.

Bands of small intensity present in the region ν 386 cm^{-1} in the IR spectra of **3a** (Table 3) are mainly due to the P-S-Sb valence vibrations, such as those found for antimony(III) dithiophosphates [5,6,8]. Bands in the region ν 619–624 cm^{-1} are assigned to the Sb–OC valence vibrations of two alkoxy groups that remain attached to the antimony atom of **3a,b** [18]. Bands in the region ν 687–673 cm^{-1} are assigned to the P=S valence vibration. The position of the P=S bond seems to be an index of hydrolytic stability of **3** and **5**. The partial hydrolysis of **3c** brought about by exposure to the open air during one day resulted in the formation of dithiophosphonic acids **6c**. Consequently, we have simultaneously observed two bands at ν 680 and 650 cm^{-1} due to the various P=S valence vibrations of **3c** and **6c**, respectively. The symmetrical deformation vibrations of geminal methyl groups $(CH_3)_2C$ appear as a pair of bands of medium intensities at ν 1395 and 1370 cm^{-1} in the IR spectrum of **5**. Bands of strong intensities present in the region ν 1070–1010 and 980–968 cm^{-1} are due to the PO–C and SbO–C valence vibrations. Interpretation of key bands is given in Table 3. This is confirmed by attachment of the alkoxy group to the phosphorus atom.

The ready rupture of the S–Sb bond of **3** is confirmed by mass spectral data (Table 5). The chemical ionization mass spectra of **3a** and **3b** exhibit the mass peaks m/e 262 and 276 that may be attributed to the ions [M + H − Sb(OPr−*i*)$_2$]$^+$ (100%) and [M + H − Sb(OBu−*i*)$_2$]$^+$ (80%), respectively. The mass peaks m/e 410 and 486 observed in the electron impact mass spectra of **3a** and **3b**, respectively, are due to the ions [M − OPr−*i* − S]$^+$ and [M − Bu−*i*]$^+$. The mass peak m/e 559 was observed in the electron impact mass spectrum of crude **3b** that may be attributed to the ion [M + O] of an oxidation product of **3b**. The largest mass peak m/e 452 in the electron impact mass spectrum of **5** was assigned to the ion [M − OBu−*i*]$^+$ (Table 5). It is remarkable that the electron impact mass spectrum of the crude reaction mixture of **4** with **2b** also indicates the mass peak m/e 589 that is considered to be a product of the addition of two sulfur atoms to the molecule of **5** [M + 2S]$^+$. The initial **4** and some products of partial destruction of **4** are likely to serve as sulfur donors.

It is noteworthy to compare the reactivity of reagents in Reactions 1 and 2. The reactivity of alkyl homologues of Davy's reagent **4a,b** is higher than that of Lawesson's reagent **1a** toward **2b**. Reaction 2 is exothermic and occurs during 3 hours.

EXPERIMENTAL

General Data

The ^{31}P NMR spectra in C_6H_6 solution were recorded with a Bruker MSL 400 (162 MHz) instrument. The

^{1}H NMR spectra were taken on a Bruker MSL-400 (400 MHz) spectrometer and a Varian T-60 (60 MHz) spectrometer in C_6D_6 or $CDCl_3$. The IR spectra were obtained with a Bruker IFS 113v and a UR-20 infrared spectrophotometers. Mass spectra (EI, 70 eV; CI, 100 eV) were determined on an M 80 B Hitachi chromatomass spectrometer.

S-Diisobutoxyantimony(III) O-Isobutyl-4-methoxyphenyldithiophosphonate **3b** *a*. Compound **1a** [2.7 g (6.7 mmol)] was added portionwise under dry argon with stirring at 20°C to 4.5 g (13.2 mmol) of **2b**, and stirring was continued for 4 hours at 20°C. The mixture was centrifuged (3000 turns/min) for 0.5 hour. The liquid layer was evaporated at reduced pressure (0.1 and 0.05 mm Hg) at 50°C for 2 hours to give 4.8 g (66%) of crude **3b**. Product **3b** (1.8 g, 25%) was isolated from the residue by means of a falling-film distillation (see Tables 1–5).

b. Similarly, compounds **1a** [3.7 g (9.2 mmol)] and **2b** [6.2 g (18.2 mmol)] (reaction conditions: 20°C, 4 hours, 10 mL of anhydrous benzene) gave 7.8 g (79%) of crude **3b**. Part (2.1 g) of crude **3b** was chromatographed on a silica-gel column with CH_2Cl_2 as eluant to yield 0.5 g of pure **3b** (see Tables 1–5).

The crude product **3a** was obtained similarly (see Tables 1–5).

S-Disec-butoxyantimony(III) O-sec-butyl-3,5-ditert.-butyl-4-hydroxyphenyldithiophosphonate **3c**

Compound **1b** 13.0 g (21.7 mmol)] was added portionwise under dry argon with stirring at 20°C to 14.8 g (43.4 mmol) of **2c**, and stirring was continued for 4 hours at 20°C. The liquid layer was decanted and kept for a month at ~20°C. The crystalline precipitate of **3c** (25.1 g, 90%) that had formed was filtered off, washed with anhydrous benzene, and dried under vacuum (0.05 mm Hg) (see Tables 1–5).

S-Diisobutoxyantimony(III) O-Isobutyl-S-isobutyltrithiophosphate **5** *a*. Compound **4** [3.0 g (8.1 mmol)] was added portionwise under dry argon with stirring to 5.5 g (16.1 mmol) of **2b**. After an exothermic period of the reaction had been completed, the stirring of the reaction mixture was continued for 3 hours at 20°C. The mixture was centrifuged (3000 turns/min) for 0.5 hour. The liquid layer was decanted and evaporated at reduced pressure (0.1 and 0.02 mm Hg) at 50°C for 2 hours to give 4.5 g (52%) of crude **5**, Product **5** (1.1 g, 13%) was isolated from the residue by means of a falling-film distillation (see Tables 1–5).

b. Similarly, compounds **4** [2.4 g (6.5 mmol)] and **2b** [4.5 g (13.2 mmol)] (reaction conditions: 20°C, 3 hours) gave 3.9 g (45%) of the crude **5**. Part (1.1 g) of crude **5** was chromatographed on a silica-gel column with anhydrous Et_2O as eluant to yield 0.8 g of pure **5** (see Tables 1–5).

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