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Chandra S. Sharma^a; Brajesh K. Jha^a; Padam N. Nagar^a

^a Department of Chemistry, University of Rajasthan, Jaipur, India

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Reactions of Ammonium Alkylene Dithiophosphates with Phosphorus Trichloride and Thiophosphoryl Chloride*

Chandra S. Sharma

Brajesh K. Jha

Padam N. Nagar

Department of Chemistry, University of Rajasthan, Jaipur, India

Phosphorus trichloride and thiophosphoryl chloride derivatives of alkylene dithiophosphates of the type $PCl_{3-n}(S_2\dot{P}OGO)_n$ and $P(S)Cl_{3-n}(S_2\dot{P}OGO)_n$ (where $G = -CH_2CH_2CHMe-$, $-CMe_2CMe_2-$, $-CH_2CH_2CH_2CH_2-$ and $-CMe_2CH_2CHMe-$, $n = 1, 2, 3$) have been synthesized for the first time by reacting the ammonium salt of alkylene dithiophosphoric acid with phosphorus trichloride and thiophosphoryl chloride in different stoichiometric ratios under anhydrous reaction conditions. The newly synthesized derivatives are either colorless liquids or viscous semisolids, hygroscopic in nature and are soluble in common organic solvents. These are characterized by elemental analysis, molecular weight determinations and physicochemical studies IR, NMR (1H and ^{31}P). On the basis of above studies the formation of P-S-P and S=P-S-P(S) chemical linkages have been established. ^{231}P NMR studies provide convincing evidence regarding the chemical bonding mode in these derivatives.

Keywords Ammonium alkylene dithiophosphate; phosphorus trichloride; thiophosphoryl chloride

INTRODUCTION

O,O' Dialkyl and alkylene dithiophosphoric acids, the cyclic analogous of dialkyl dithiophosphate, have been described and their metal and organometal complexes have been studied extensively. In 1983, Chauhan et al.¹ first reported the synthesis of alkylene dithiophosphoric acids by reacting phosphorus pentasulfide with 1,2 and 1,3-glycols. Various interesting reactions, as well as spectroscopic characterization of metal and organometal derivatives of dithiophosphoric acids, have

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Address correspondence to Padam Nagar, Department of Chemistry, University of Rajasthan, Jaipur 302004, India. E-mail: brajeshchem05@rediffmail.com

been reported during last two decades.²⁻⁹ More recently, the work has been extended to synthesis, characterization and biological studies of organic derivatives of dithioesters.¹⁰⁻¹²

A number of single crystal X-ray structures had also been reported in the literature for the metal and organometal derivatives of dithio ligands.¹³⁻¹⁴ The derivatives of the type $M[S_2POGO]_n$, $M = As, Sb \& Bi$ have wide range of industrial applications such as analytical reagents, lubricants, and catalytic activities for regeneration of cracking catalysts.³ The importance of the work in this research area can be judged by the National Cancer Research Institute, USA, inviting samples of the type $M[S_2POGO]_n$; $M = Ni, Pd \& Pt$, for studying their anti-cancer and anti AIDS activities. Bhasin et al. recently reported the first approach towards synthesis, properties, catalytic and antimicrobial aspects of some Ni (II) macro cyclic complexes having tetraoxa, tetrathio, and tetraaza ligands in 14-20¹⁵ and 24-28¹⁶ membered rings.

In view of the interesting chemical bonding characteristics,¹⁷⁻¹⁹ as well as synthetic and biological applicability of the organophosphorus compounds,²⁰⁻²² a study has been undertaken on the reactions of ammonium alkylene dithiophosphates with phosphorus trichloride and thiophosphoryl chloride.

RESULTS AND DISCUSSION

Reactions of phosphorus trichloride and thiophosphoryl chloride with the ammonium salt of alkylene dithiophosphates in different molar ratios yield the derivatives of the type $PCl_{3-n}[S_2POGO]_n$ and $P(S)Cl_{3-n}[S_2POGO]_n$:

1. (A) $[\overline{OGOP}(S)S]NH_4 + PCl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]PCl_2 + NH_4Cl \downarrow$
 (B) $2[\overline{OGOP}(S)S]NH_4 + PCl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]_2PCl + 2 NH_4Cl \downarrow$
 (C) $3[\overline{OGOP}(S)S]NH_4 + PCl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]_3P + 3 NH_4Cl \downarrow$; and
2. (A) $[\overline{OGOP}(S)S]NH_4 + P(S)Cl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]P(S)Cl_2 + NH_4Cl \downarrow$
 (B) $2[\overline{OGOP}(S)S]NH_4 + P(S)Cl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]_2P(S) + 2 NH_4Cl \downarrow$
 (C) $3[\overline{OGOP}(S)S]NH_4 + P(S)Cl_3 \xrightarrow{\text{Benzene}} [\overline{OGOP}(S)S]_3P(S) + 3 NH_4Cl \downarrow$

where $G = -CMe_2CMe_2-$, $-CH_2CH_2CHMe-$, $-CH_2CH_2CH_2CH_2-$, and $-CMe_2CH_2CHMe-$; and $n = 1, 2 \& 3$.

The products formed are generally colorless liquids, hygroscopic in nature and are soluble in common organic solvents. The above reactions

are exothermic; therefore, we preferred a slow dropwise addition of reactants. The tricovalent phosphorus derivatives are sensitive towards atmospheric moisture and to atmospheric oxygen on exposure for 48 h. These compounds show the tendency to convert into pentavalent phosphorus atom. These compounds also exhibit the tendency of polymerization on storage at ambient condition.

IR Spectra

IR spectra of phosphorus (III) and phosphorus (V) derivatives are comparable to those of the corresponding free alkylene dithiophosphoric acid and its various metal derivatives. All assignments have been made on the basis of literature.¹⁻³ The bands present in the region 1020–1125 cm⁻¹ and 915–790 cm⁻¹ have been assigned to $\nu(\text{P})\text{--O--C}$ and $\nu\text{P--O--(C)}$ modes, respectively. The absorption bands due to the ring vibrations of the dioxaphospholanes and phosphorinanes may be assigned in the region 990–945 cm⁻¹. The absorption (sharp) bands in the region 650–625 cm⁻¹ for compounds **1–9**, whereas for compounds **9–18** are in the region 652–625 cm⁻¹ may be attributed to $\nu\text{P}=\text{S}$.²³ It has been observed that in most of the cases, the $\nu_{\text{p}} = \text{s}$ band shows shifting towards higher wave numbers. However, for compounds **7**, **8**, **9**, the tris (alkylene dithiophosphate) phosphorus (III), shifting (20–10 cm⁻¹) towards lower wave number is observed.

Similar variations have also been observed in the IR spectra of mono, bis and tris (alkylene dithiophosphate) derivatives of the thiophosphoryl chloride. The prominent feature of IR spectra of above derivatives is the shifting in $\nu\text{P--Cl}$ vibrations. It is in 525–510 cm⁻¹ for mono (alkylene dithiophosphate) phosphorus (III) with a negative shift with respect to $\nu\text{P--Cl}$ vibration in the IR spectra of PCl_3 (~580 cm⁻¹), whereas for compounds **4–6**, it appeared in the range 520–500 cm⁻¹, and it completely disappeared for PL_3 (L = alkylene dithiophosphato) derivatives (Table I).

¹H NMR Spectra

The ¹H NMR spectra show the characteristic resonance signals in accordance with their structure. Signals due to protons present on the α -carbon of the P--O--C skeleton of the alkylene chain appeared as doublet or multiplets in some cases due to coupling with ³¹P nucleus. The coupling constant, ³J_{POCH}, has been observed in the range of 16–18 Hz for this type of coupling with magnetically active phosphorus atom.

TABLE I IR Spectral Data of the Compounds (in cm⁻¹)

Compound no.	Compound formula	$\nu(\text{P})-\text{O}-\text{C}$	$\nu\text{P}-\text{O}-(\text{C})$	Ring	$\nu\text{P}=\text{S}$	$\nu\text{P}-\text{S}$	$\nu\text{P}-\text{Cl}$	$\nu\text{P}-\text{S}$ (Thio)
1	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl ₂	1125	890	—	645	590	520	—
2	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl ₂	1080	820	990	640	585	517	—
3	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl ₂	1020	840	970	650	600	515	—
4	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl	1040s	860m	980	640s	560	515w	—
5	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl	1110m	835m	—	650m	580w	525w	—
6	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] PCl	1090m	805m	940	635m	565w	510w	—
7	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P	1110s	865	985	635	555	—	—
8	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P	1040	790	945	640	565	—	—
9	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P	1030	910	955	625s	570s	—	—
10	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl ₂	1035	805	945s	640	585	585	680w
11	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl ₂	1130	765	—	625	595	590	675
12	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl ₂	1105	880	995	630	590	590	680
13	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl	1055	835m	—	650m	575s	670	575m
14	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl	1125	910	985	655	565	665	580
15	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)Cl	1025	895	970	645	600	660	580
16	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)	1055	805	985	650	575	—	650
17	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)	1125	910	975	652	560	—	640
18	[OCMe ₂ CH ₂ CH ₂ CH ₂ OP(S)S] P(S)	1110	915	950	648	565	—	645

^{31}P NMR Spectra

^{31}P NMR spectra were recorded in benzene and are tabulated in Table II. The two signals for each compound (**1–9**) indicate the presence of two types of chemically non-equivalent phosphorus atoms. The chemical shift range between δ 75–98 is assigned for the phosphorus atom of the pentavalent dithiophosphate moiety. Gradual shielding has also been recorded for trivalent phosphorus atom.²⁴ A comparison may be setup for mono, bis and tris (alkylene dithiophosphate) phosphorus compounds **1–9** that the respective shifts are in the range of δ 190–198, δ 181–187 and δ 167.5–169.5. Shielding is observed to the extent of about δ 40–50 from its position in phosphorus trichloride ($^{31}\text{P} = \delta$ 221). On the basis of the above discussion a phosphate-phosphite type structure has been proposed for P(III) derivatives of dithiophosphates.

TABLE II ^1H and ^{31}P NMR Spectral Data for the Compounds

Compound no.	^1H (δ ppm)	^{31}P (δ ppm) P=S (P)
1	1.40,s,12 H (CH_3)	95.8 (194)
2	1.35, d, 3H (CH_3); 1.65–1.80, m, 2H (CH_2); 4.30–4.70, m, 3H (OCH_2)	75.6 (197.8)
3	1.80–2.05, m, 4H (CH_2); 3.95–4.15, m, 4H (OCH_2)	83.5 (190.4)
4	1.36, d, 6H(CH_3); 1.48–1.54, m, 4H(CH_2); 4.28–4.50, m, 6H (OCH_2)	79.2 (182.5)
5	1.52, m, 8H(CH_2); 3.88–4.12, m, 8H(OCH_2)	85.6 (181.4)
6	1.34–2.15, m 18H(CH_3); 4.78, s(broad) 2H (CHO)	88.25 (186.6)
7	1.35, d, 9H(CH_3); 1.65–1.80, m, 6H(CH_2); 4.30–4.70, 9H(OCH_2)	81.5 (169.5)
8	1.56, m, 12H(CH_2); 3.80–4.25, m 12 (OCH_2)	89.0 (168.7)
9	1.42–2.24, m, 27H(CH_3); 4.88, s, 3H (CHO)	91.5 (167.5)
10	1.35, s, 12H (CH_3)	104 (47.58)
11	1.35, d,3H (CH_3); 1.58–1.75, m, 2H(CH_2); 4.30–4.75, m, 3H (OCH_2)	86.68 (47.00)
12	1.43, m, 4H (CH_2); 3.75–4.15, m, 4H(CH_2)	83.00 (48.50)
13	1.25, d, 6H (CH_3); 1.45–1.62, m, 4H(CH_2); 4.21–4.36, m 6H (OCH_2)	85.56 (54.55)
14	1.52, m, 8H (CH_2); 3.78–4.05, m, 8H (OCH_2)	82.00 (58.61)
15	1.25–1.95, s, 12H(CH_3); 4.80, m, 2H(CH_2O); 1.15,d,6H (CH_3)	95.00 (56.26)
16	1.28, d, 9H(CH_3); 1.65–1.80, m, 6H(CH_2); 4.30–4.75m, 9H(OCH_2)	83.50 (55.95)
17	1.58, m, 12H(CH_2); 3.68–4.18, m, 12H(OCH_2)	80.23 (63.59)
18	4.75, m, 3H(CHO); 1.25, d, 9H(CH_3)	91.78 (61.25)

^{31}P NMR studies of $\text{P}(\text{S})\text{Cl}_3$ derivatives (**10–18**) indicate remarkable changes in chemical shift values for both the phosphorus atoms bearing sulfur atoms. ^{31}P signals for $\text{P}(\text{S})\text{S}$ phosphorus atom show downfield shift (δ 15–30) in comparison to ammonium dithiophosphates, while $\text{S}=\text{PCl}_{3-n}$ phosphorus atom show down field shift (δ 35) in comparison to its position in thiophosphoryl chloride ($\delta^{31}\text{P} = 29$). All the ^{31}P spectra recorded are completely proton or phosphorus decoupled so couplings between these nuclei have not been recorded. The presence of two ^{31}P signals indicates presence of two different types of nonequivalent phosphorus atoms (δ 47–63.59). Although both the phosphorus atoms bear the sulfur atom in thiophosphoryl ($\text{P}=\text{S}$) form, there is a remarkable difference in the chemical shift values of both the phosphorus atoms.

On the basis of the above a thiophosphoryl-thiophosphate ester type structure $(\text{S})\text{P}-\text{S}-\text{P}(\text{S})\text{X}_{3-n}$ has been proposed for the above derivatives.

EXPERIMENTAL

Stringent precautions were taken to exclude moisture during experimental work. *O,O'*-Alkylene dithiophosphoric acids have been prepared by the method reported in the literature and have been purified by vacuum distillation. Sulfur was estimated gravimetrically as barium sulphate,²⁴ while chlorine was estimated by Vohlard's method.²⁵ Infrared spectra were recorded on a Shimadzu FTIR8400 spectrophotometer and ^1H and ^{31}P NMR spectra on a JEOL FX300MHz FTNMR spectrometer using TMS as internal reference (for ^1H) and H_3PO_4 (for ^{31}P) as external reference.

Reaction between Ammonium Alkylene Dithiophosphate and Phosphorus Trichloride in Different Molar Ratios

A dropwise addition of a benzene solution of phosphorus trichloride to the ammonium salt of alkylene dithiophosphate taken in benzene was carried out with simultaneous stirring of the reaction mixture. After complete addition of phosphorus trichloride solution, the reaction mixture had been refluxed for 4–6 h. The ammonium chloride thus precipitated during the course of reaction was filtered off and finally the filtrate was evaporated under reduced pressure. Relevant data are tabulated in Table III.

Reaction between Ammonium Alkylene Dithiophosphate and Thiophosphoryl Chloride in Different Molar Ratios

A benzene solution of thiophosphoryl chloride was added dropwise to a suspension of ammonium alkylene dithiophosphate in benzene.

TABLE III Synthesis and Analytical Data for the Compounds

S. No.	Reactant g(mmole)		Product (g)	Yield (%)	Analysis %	
	PCl ₃ (P(S)Cl ₃)	OGOP(S)/SNH ₄ G=			S Found (calcd.)	Cl Found (calcd.)
1	0.68 (4.94)	—C(Me) ₂ C(Me) ₂ 1.13 (4.94)	[OC(Me) ₂ C (Me) ₂ OP(S)SPCl ₂ 1.26	68.35	20.05 (20.44)	21.23 (22.68)
2	0.69 (5.01)	—CH ₂ CH ₂ CHMe— 1.01 (5.01)	[OCH ₂ CH ₂ CH MeOP(S)SPCl ₂ 1.112	77.62	21.95 (22.45)	24.15 (24.91)
3	1.03 (7.52)	—CH ₂ CH ₂ CH ₂ CH ₂ — 1.51 (7.52)	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S]P(S)Cl ₂ 1.485	69.39	20.14 (22.45)	22.86 (24.91)
4	0.68 (4.94)	—CH ₂ CH ₂ CHMe— 1.80 (9.88)	[OCH ₂ CH ₂ CH MeOP(S)S] ₂ PCl 1.80	84.50	29.15 (29.59)	7.86 (8.20)
5	0.78 (5.67)	—CH ₂ CH ₂ CH ₂ CH ₂ — 1.44 (11.34)	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] ₂ PCl 1.89	77.14	29.45 (29.59)	8.11 (8.20)
6	0.95 (6.90)	—C(Me) ₂ CH ₂ CHMe— 3.16 (13.8)	[OC(Me) ₂ CH ₂ CHMeOP(S)S] ₂ PCl 3.08	91.56	25.70 (26.20)	7.04 (7.26)
7	0.88 (6.4)	—CH ₂ CH ₂ CHMe— 3.85 (19.2)	[OCH ₂ CH ₂ CHMeOP(S)S] ₃ P 3.32	89.6	32.89 (33.10)	—
8	1.10 (8.2)	—CH ₂ CH ₂ CH ₂ CH ₂ — 4.82 (24.6)	[OCH ₂ CH ₂ CH ₂ CH ₂ OP(S)S] ₃ P 4.64	98.5	28.16 (33.10)	—
9	0.70 (5.09)	—C(Me) ₂ CH ₂ CHMe— 3.49 (15.27)	[OC(Me) ₂ CH ₂ CHMeOP(S)S] ₃ P 3.07	91.09	28.16 (28.91)	—

(Continued on next page)

TABLE III Synthesis and Analytical Data for the Compounds (Continued)

S. No.	Reactant g(mmole)		Product (g)	Yield (%)	Analysis %	
	PCl ₃ (P(S)Cl ₃)	GOP(S)SNH ₄ G=			S Found (calcd.)	Cl Found (calcd.)
10	1.08 (6.37)	—C(Me) ₂ C(Me) ₂ — 1.46 (6.37)	[OC(Me) ₂ C(Me) ₂ OP(S)SP(S)Cl ₂ 2.02	92.58	26.51 (27.82)	19.74 (20.57)
11	0.85 (5.01)	—CH ₂ CH ₂ CHMe— 1.01 (5.01)	[OCH ₂ CH ₂ CHMeOP(S)SP(S)P(S)Cl ₂ 1.41	89.32	29.95 (30.28)	21.95 (22.59)
12	0.91 (5.37)	—CH ₂ CH ₂ CH ₂ CH ₂ — 1.08 (5.37)	[OCH ₂ CH ₂ CH ₂ OP(S)P(S)Cl ₂ 1.33	78.32	29.14 (30.28)	22.39 (23.89)
13	0.72 (4.25)	—CH ₂ CH ₂ CHMe— 1.71 (8.5)	[OCH ₂ CH ₂ CH ₂ OP(S)P(S)Cl ₂ 1.80	91.63	26.95 (27.55)	7.09 (7.64)
14	1.06 (6.25)	—CH ₂ CH ₂ CH ₂ CH ₂ — 2.51 (12.5)	[OCH ₂ CH ₂ CH ₂ OP(S)P(S)Cl ₂ 2.38	82.68	26.14 (27.55)	6.19 (7.64)
15	1.08 (6.37)	—C(Me) ₂ CH ₂ CHMe— 2.92 (12.74)	[OC(Me) ₂ CH ₂ CHMeOP(S)S ₂ P(S)Cl 2.62	79.26	22.68 (24.59)	5.24 (6.82)
16	0.81 (4.78)	—CH ₂ CH ₂ CHMe— 2.87 (14.34)	[OCH ₂ CH ₂ CHMeOP(S)S ₃ P(S) 2.60	89.23	29.56 (31.37)	—
17	0.84 (4.95)	—CH ₂ CH ₂ CH ₂ CH ₂ — 2.98 (14.85)	[OCH ₂ CH ₂ CH ₂ OP(S)S ₃ P(S) 2.81	93.04	29.85 (31.37)	—
18	0.90 (5.30)	—C(Me) ₂ CH ₂ CHMe— 3.64 (15.90)	[OC(Me) ₂ CH ₂ CHMeOP(S)S ₃ P(S) 3.26	88.58	26.54 (27.58)	—

After complete addition (1–2 h) of the benzene solution of thiophosphoryl chloride, the contents were refluxed (8–12 h). The precipitated ammonium chloride was filtered off; stripping off the solvent under vacuum has given the products. Relevant data are tabulated in Table III.

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