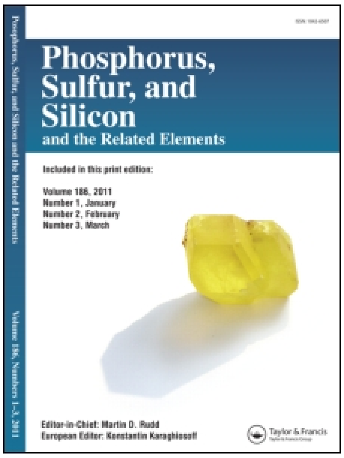

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Synthetic and Spectroscopic Studies on Some *Bis*(dialkyldithiocarbomato)-arsenic(III) O,O'-dialkyldithiophosphates

H. P. S. Chauhan[a]; Kavita Kori[a]; N. M. Shaik[a]; U. P. Singh[a]
[a] School of Chemical Sciences, Devi Ahilya University, Takshshila Campus, Khandwa Road, Indore, India

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Synthetic and Spectroscopic Studies on Some *Bis*(dialkyldithiocarbomato)-arsenic(III) O,O′-dialkyldithiophosphates

H. P. S. Chauhan
Kavita Kori
N. M. Shaik
U. P. Singh
School of Chemical Sciences, Devi Ahilya University, Takshshila
Campus, Khandwa Road, Indore, India

The reactions of bis*(dialkyldithiocarbamato)arsenic(III) chloride with sodium salts of dialkyldithiophosphates in 1:1 molar ratio give the corresponding mixed derivatives of the type* $[R_2NCS_2]_2AsS_2P(OR')_2$ *[where,* $R = CH_3$ *and* C_2H_5*;* $R' =$ *Et,* Pr^n*,* Pr^i*,* Bu^n *and* Bu^i*]. These derivatives are yellow crystalline solids or yellow viscous liquids. All these newly synthesized derivatives are soluble in common organic solvents such as benzene, chloroform, carbondisulphide, acetone, and dichloromethane. These derivatives have been characterized by melting points (only solid derivatives), elemental analysis (C, H, N, S, and As) as well as spectral IR, NMR [*^1H*,* ^{13}C*, and* ^{31}P*] studies. Based on these studies tentative structures for these derivatives have been proposed.*

Keywords 1,1-Dithiolates; ^1H, ^{13}C and ^{31}P NMR spectra; arsenic; dialkyldithiocarbamate; dialkyldithiophosphate; IR spectra

INTRODUCTION

The 1,1-dithiolato ligands, dialkyldithiocarbamates,[1–11] dialkyldithiophosphates[12–21] are versatile in nature and exhibit remarkable diversities in their bonding/coordination patterns with transition as well as main group metals.[1–9] Although a number of tris[22] as well as mixed halide dithiolates of arsenic(III) and organoarsenic(III) derivatives with these ligands are known and some of them are fully characterized by single crystal X-ray structural analysis,[18–29] the corresponding arsenic(III) derivatives with mixed 1,1-dithiolato ligands do not

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Address correspondence to H. P. S. Chauhan, School of Chemical Sciences, Devi Ahilya University, Takshshila Campus, Khandwa Road, Indore 452017, India. E-mail: hpsc@rediffmail.com

appear to have received much attention of chemists. In continuation of our interest in synthetic and structural aspects of mixed sulphur ligand complexes of group 15 elements,[7–11,30–32] we report herein some new mixed *bis*(dialkyldithiocarbamato)arsenic(III) complexes with dialkyldithiophosphates of the type $[R_2NCS_2]_2AsS_2P(OR')_2$ [where, R = CH_3 and C_2H_5; R' = Et, Pr^n, Pr^i, Bu^n, and Bu^i].

RESULTS AND DISCUSSION

Derivatives of *bis*(dialkyldithiocarbamato)arsenic(III) dialkyldithiophosphates have been prepared by the reaction of *bis*(dialkyldithiocarbamato)arsenic(III) chloride with sodium dialkyldithiophosphates in 1:1 molar ratio in refluxing benzene for ~4 hours.

$$[R_2NCS_2]_2AsCl + NaS_2P(OR')_2 \xrightarrow{\text{Benzene}} [R_2NCS_2]_2AsS_2P(OR')_2 + NaCl\downarrow$$

$$(\text{where } R = CH_3 \text{ and } C_2H_5;\ R' = \text{Et, Pr}^n\text{, Pr}^i\text{, Bu}^n\text{, and Bu}^i)$$

All these newly synthesized derivatives are either pale yellow solids or non-volatile yellow viscous liquids. All these derivatives are soluble in common organic solvents such as benzene, chloroform, acetone, and dichloromethane.

Infra-Red Spectra

IR spectra of all these newly synthesised compounds have been recorded as KBr disc in the range 4000–200 cm^{-1} and tentative assignments of the important characteristic bands have been made with the help of earlier publications.[7–11,15,21,24–27,30–32]

The bands of medium to weak intensity in the regions 960–990 cm^{-1} and 820–890 cm^{-1} are assigned to [(P)—O—C] and [P—O—(C)] stretching modes, respectively. A strong band due to (P=S) stretching vibrations present in the spectra of sodium salts of dithiophosphoric acids in the region 660–690 cm^{-1} is shifted towards lower frequencies in the spectra of these arsenic(III) dithiophosphate derivatives and is present at 615–650 cm^{-1}. This shifting indicates a probable bidentate chelation of dithiophosphate moieties with arsenic. Bands of weak to medium intensities present in the region 510–545 and 320–325 cm^{-1} are due to (P—S) and (As—S) stretching vibrations, respectively. These derivatives also show medium to strong intensity absorption bands in the region 1425–1520 and 1020–1040 cm^{-1} owing to (C=N) and (C=S) stretching vibrations, respectively and thus indicating the biconnective behavior of the dithiocarbamate moieties in these complexes.

^{1}H NMR

The ^{1}H NMR spectra of *bis*(dialkyldithiocarbamato)arsenic(III) complexes with dialkyldithiophosphates have been recorded in $CDCl_3$ solutions using TMS as an internal standard. In the corresponding dimethyldithiocarbamate derivatives, the methyl protons appear as a singlet at 3.45–3.46 δ ppm thus suggesting the magnetic equivalence of these protons, while the diethyldithiocarbamate derivatives exhibit a triplet at 1.22–1.42 δ ppm and a quartet at 3.07–3.81 δ ppm due to CH_3 and CH_2 proton resonances, respectively (Table I). In addition, these derivatives also exhibit the expected signals due to resonances of the corresponding ethyl, *n*-propyl, *i*-propyl, *n*-butyl, and *i*-butyl protons of dialkyldithiophosphate moieties.[12–21] The signals, due to the coupling of α-CH_2O protons (attached to the carbon atom nearest to the phosphorous atom) with ^{31}P nuclei, of the corresponding *n*-propoxy (compound No. 2, 7 in Table I), *n*-butoxy (compound No. 9 in Table I) and *i*-butoxy (compound No. 5, 10 in Table I) split as doublet of triplet and doublet of doublet, respectively.

^{13}C NMR

The proton decoupled ^{13}C NMR spectra (Table I) of these compounds have been recorded in $CDCl_3$ solutions using TMS as an internal standard. The ^{13}C NMR spectra of the dimethyldithiocarbamate complexes shows a signal in the region 42.7–43.1 δ ppm due to magnetic equivalent methyl carbon. The complexes of diethyldithiocarbamate show two signals one at 11.18–12.1 δ ppm and other at 42.13–47.8 δ ppm due to CH_3 and CH_2 carbons, respectively. All these compounds show a weak signal at 192.8–198.0 δ ppm due to NCS_2 carbon resonances. In addition, these derivatives also exhibit the expected signals due to alkyl (ethyl, *n*-propyl, *i*-propyl, *n*-butyl, and *i*-butyl) carbons of dialkyldithiophosphate moieties (Table I). The ^{13}C NMR spectral data are comparable with the data reported earlier of corresponding arsenic(III) dialkyldithiophosphate complexes.[24–27]

^{31}P NMR Spectra

On the basis of the analysis of proton decoupled ^{31}P NMR chemical shift values of a number of metal dialkyldithiophosphates, Glidewell[33] concluded that complexes showing their NMR signal in the range 82–101 δ ppm exhibit bidentate mode of attachment of the dialkyldithiophosphate ligands toward metals. Proton decoupled ^{31}P NMR spectral data for these compounds (Table I) exhibit only one ^{31}P

TABLE I **^{1}H, ^{13}C and ^{31}P NMR Spectral Data (δ ppm) of *bis*(dialkyldithiocarbamato)arsenic(III) Complexes with Dialkyldithiophosphates**

S. no.	Compound	H^1 NMR chemical shift (δ ppm)	^{13}C NMR chemical shift (δ ppm)	^{31}P NMR chemical shift (δ ppm)
1.	$[(CH_3)_2NCS_2]_2AsS_2P(OC_2H_5)_2$	1.36, t, 6H (CH_3 of dtp) J = 6.00 Hz 3.46, s, 12H (NCH_3 of dtc) 4.17,q, 4H (OCH_2 of dtp) J = 6.00 Hz	15.5 (CH_3 of dtp) 42.7 (CH_3 of dtc) 63.2 (OCH_2 of dtp) 192.8 (NCS_2 of dtc)	95.26
2.	$[(CH_3)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_3)_2$	0.95, t, 6H (CH_3 of dtp) J = 6.00 Hz 1.58–1.70, m, 4H, (CH_2 of dtp) 3.46, s, 12H, (NCH_3 of dtc) 3.95, dt, 4H (OCH_2 of dtp) J(OCH_2CH_2) = 6.00 Hz J($POCH_2$) = 9.00 Hz	10.3 (CH_3 of dtp) 23.5 (CH_2 of dtp) 43.1 (CH_3 of dtc) 68.0 (OCH_2 of dtp) 192.8 (NCS_2 of dtc)	99.50
3.	$[(CH_3)_2NCS_2]_2AsS_2P[(OCH(CH_3)_2]_2$	1.36, d, 12H[$(CH_3)_2$ of dtp] J = 6.00 Hz 3.46, s, 12H (NCH_3 of dtc) 4.83, sep, 2H (OCH of dtp) J = 6.00 Hz	23.7 [$(CH_3)_2$ of dtp] 43.1 (CH_3 of dtc) 69.9 (OCH of dtp) 193.4 (NCS_2 of dtc)	95.52
4.	$[(CH_3)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_2CH_3)_2$	0.93, t, 6H (CH_3 of dtp) J = 6.00 Hz 1.36–1.48, m, 4H (CH_2CH_3 of dtp) 1.65–1.74, m, 4H (CH_2CH_2O of dtp) 3.45, s, 12H, (NCH_3 of dtc) 4.11, t, 4H, (OCH_2 of dtp) J = 7.5 Hz	13.6 (CH_3 of dtp) 18.7 (CH_2 CH_3 of dtp) 32.0 (CH_2CH_2O of dtp) 43.0 (CH_3 of dtc) 67.1 (OCH_2 of dtp) 192.8 (NCS_2 of dtc)	97.05
5.	$[(CH_3)_2NCS_2]_2AsS_2P[(OCH_2CH(CH_3)_2]_2$	0.96, d, 12H [$(CH_3)_2$ of dtp] J = 6.00 Hz 1.96–2.036, m, 2H(CH of dtp) 3.45, s, 12H (NCH_3 of dtc) 3.89, dd, 4H (OCH_2 of dtp) J(OCH_2CH) = 6.00 Hz J($POCH_2$) = 9.00 Hz	18.9 [$(CH_3)_2$ of dtp] 28.9 (CH of dtp) 43.1 (CH_3 of dtc) 70.3 (OCH_2 of dtp) 195.2 (NCS_2 of dtc)	96.76

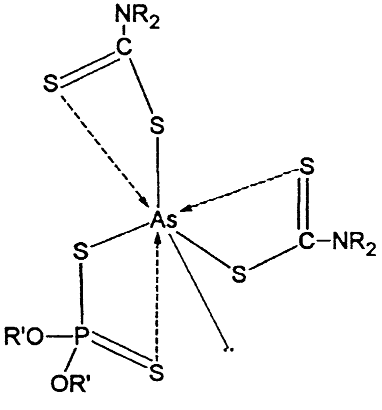
6.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OC_2H_5)_2$	1.25, t, 6H (CH_3 of dtp) J = 7.0 Hz 1.38, t, 12H (CH_3 of dtc) J = 7.0 Hz 3.10, q, 8H (NCH_2 of dtc) J = 7.0 Hz 3.78, q, 4H (OCH_2 of dtp) J = 7.0 Hz	11.97 (CH_3 of dtc) 15.65 (CH_3 of dtp) 42.13 (CH_2 of dtc) 62.3 (OCH_2 of dtp) 198.0 (NCS_2 of dtc)	94.82
7.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_3)_2$	1.22, t, 12H (CH_3 of dtc) J = 6.00 Hz 1.40, t, 6H (CH_3 of dtp) J = 6.00 Hz 1.70–1.88, m, 4H (CH_2 of dtp) 3.12, q, 8H (NCH_2 of dtc) J = 6.00 Hz 3.80, dt, 4H (OCH_2 of dtp) $J(OCH_2CH_2) = 6.00$ Hz $J(POCH_2) = 9.00$ Hz	11.18 (CH_3 of dtc) 12.30 (CH_3 of dtp) 42.26 (CH_2 of dtc) 27.81 (CH_2 of dtp) 67.76 (OCH_2 of dtp) 193.4 (NCS_2 of dtc)	95.646
8.	$[(C_2H_5)_2NCS_2]_2AsS_2P[(OCH(CH_3)_2]_2$	1.30, d, 12H (CH_3 of dtp) J = 6.00 Hz 1.42, t, 12H (CH_3 of dtc) J = 6.00 Hz 3.81, q, 8H (NCH_2 of dtc) J = 6.00 Hz 4.78, sep, 2H (OCH of dtp) J = 6.00 Hz	12.05 (CH_3 of dtc) 23.5 (CH_3 of dtp) 47.7 (CH_2 of dtc) 69.5 (OCH of dtp) 192.8 (NCS_2 of dtc)	98.05
9.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_2CH_3)_2$	0.85, t, 6H (CH_3 of dtp) J = 6.00 Hz 1.22, t, 12H (CH_3 of dtc) J = 6.00 Hz 1.28–1.43, m, 4H (CH_2CH_3 of dtp) 1.57–1.66, m, 4H (CH_2 CH_2O of dtp) 3.77, q, 8H (CH_2 of dtc) J = 6.00 Hz 4.02, dt, 4H (OCH_2 of dtp) J = 6.00 Hz $J(OCH_2CH_2) = 6.00$ Hz $J(POCH_2) = 9.00$ Hz	12.1 (CH_3 of dtc) 13.4 (CH_3 dtp) 18.6 (CH_2CH_3 of dtp) 31.9 (CH_2OCH_2 of dtp) 47.7 (CH_2 of dtc) 66.7 (OCH_2 of dtp) 192.8 (NCS_2 of dtc)	98.83
10.	$[(C_2H_5)_2NCS_2]_2AsS_2P[(OCH_2CH(CH_3)_2]_2$	0.88, d, 12H (CH_3 of dtp) J = 6.00 Hz 1.23, t, 12H (CH_3 of dtc) J = 6.00 Hz 1.88–1.97, m, 2H (CH of dtp) 3.07, q, 8H (CH_2 dtc) J = 6.00 Hz 3.78, dd, 4H (OCH_2 dtp) $J(OCH_2CH) = 6.00$ Hz $J(POCH_2) = 9.00$ Hz	12.1 (CH_3 of dtc) 18.8 (CH_3 of dtp) 28.5 (CH of dtp) 47.8 (CH_2 of dtc) 72.9 (OCH_2 of dtp) 192.8 (NCS_2 of dtc)	100.89

s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, sep = septet, dd = doublet of doublets, dt = doublet of triplets.

chemical shift for each compound in the range 94.82–100.89 δ ppm indicating the bidentate behavior of dithiophosphate ligands toward arsenic.

Structural Elucidation

Although it is quite difficult to comment on the molecular structure of these complexes in solid state without actual X-ray crystal structure analysis of at least one of the products, a strong and broad signal in the region 1020–1040 cm^{-1} due to (C=S) stretching vibrations in these derivatives indicate anisobidentate nature of dithiocarbamate ligands. A band due to (P=S) stretching vibrations present in the spectra of sodium salts of dialkyldithiophosphoric acids in the region 660–690 cm^{-1}, is shifted toward lower frequencies and appear at 615–650 cm^{-1}, indicating anisobidentate chelation of dithiophosphate moieties with arsenic which is further confirmed by the presence of only one proton decoupled ^{31}P chemical shift in the range 94.82–100.89 δ ppm. On the basis of the above spectral data it may be concluded tentatively that these ligands behave as anisobidentate mode of attachment to the metal, thus leading to a distorted octahedral geometry, with a stereochemically active lone pair of electrons.



Proposed Structure of the Complexes

where, R = CH_3 and C_2H_5 and R′ = Et, Pr^n, Pr^i, Bu^n, and Bu^i.

EXPERIMENTAL

Instruments

All the experiments were carried out under moisture-free conditions. Solvents (benzene, alcohols, dichloromethane, etc.) were purified and

TABLE II Physical and Analytical Data of *Bis*(dialkyldithiocarbamato)arsenic(III) Complexes with Dialkyldithiophosphates

S. no.	Compound	Yield (%)	NaCl found (Calc.)	M.P. °C	Color & State	% Found (calculated)				
						As	S	C	H	N
1.	$[(CH_3)_2NCS_2]_2AsS_2P(OC_2H_5)_2$	85	0.09 g	127	Pale yellow	14.94	37.87	22.32	4.28	6.44
			(0.10 g)		solid	(14.96)	(38.47)	(23.99)	(4.39)	(5.59)
2.	$[(CH_3)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_3)_2$	86	0.09 g	185	Pale yellow	14.05	34.97	26.83	4.44	5.51
			(0.10 g)		solid	(14.16)	(36.43)	(27.26)	(4.92)	(5.33)
3.	$[(CH_3)_2NCS_2]_2AsS_2P[OCH(CH_3)_2]_2$	91	0.09 g	195	Pale yellow	14.48	34.98	23.62	4.23	5.96
			(0.09 g)		solid	(14.16)	(36.43)	(27.26)	(4.92)	(5.33)
4.	$[(CH_3)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_2CH_3)_2$	89	0.09 g	146	Pale yellow	13.39	32.96	29.28	5.22	5.11
			(0.09 g)		solid	(13.45)	(34.60)	(30.20)	(5.39)	(5.03)
5.	$[(CH_3)_2NCS_2]_2AsS_2P[(OCH_2CH(CH_3)_2]_2$	85	0.09 g	190	Pale yellow	13.16	33.56	30.06	5.18	4.91
			(0.09 g)		solid	(13.45)	(34.60)	(30.20)	(5.39)	(5.03)
6.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OC_2H_5)_2$	91	0.12 g	—	Yellow-Orange	13.32	34.16	28.94	5.20	5.14
			(0.13 g)		Vis. Liq.	(13.45)	(34.60)	(30.20)	(5.39)	(5.03)
7.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_3)_2$	84	0.11 g	—	Yellow-Orange	12.40	32.34	32.22	5.56	4.38
			(0.11 g)		Vis. Liq.	(12.81)	(32.94)	(32.87)	(5.80)	(4.79)
8.	$[(C_2H_5)_2NCS_2]_2AsS_2P[(OCH(CH_3)_2]_2$	84	0.15 g	—	Yellow-Orange	12.80	31.79	31.90	5.60	4.81
			(0.16 g)		Vis. Liq.	(12.81)	(32.94)	(32.87)	(5.80)	(4.79)
9.	$[(C_2H_5)_2NCS_2]_2AsS_2P(OCH_2CH_2CH_2CH_3)_2$	85	0.10 g	—	Yellow-Orange	12.13	30.45	33.01	6.13	4.60
			(0.10 g)		Vis. Liq.	(12.22)	(31.43)	(35.28)	(6.20)	(4.57)
10.	$[(C_2H_5)_2NCS_2]_2AsS_2P[(OCH_2CH(CH_3)_2]_2$	90	0.11 g	—	Yellow-Orange	12.03	30.23	34.96	6.00	4.49
			(0.11 g)		Vis. Liq.	(12.22)	(31.43)	(35.28)	(6.20)	(4.57)

dried by standard methods.[34] Phosphorus pentasulphide (Merck), sodium salt of dimethyldithiocarbamate (Fluka), sodium salt of diethyldithiocarbamate (BDH), and arsenic trioxide (Merck) were used as received without further purification. Thionyl chloride (Merck) was distilled before use. Melting points were determined on B10 TECH INDIA Melting Apparatus and are uncorrected. Elemental analysis (C, H, and N) was performed on a Heraeus Carlo Erba 1108 C, H, N analyzer at Sophisticated Analytical Instrument Facility of Central Drug Research Institute, Lucknow, India. Infrared spectra were recorded on Perkin Elmer Model 557 as KBr disc in the range 4000–200 cm^{-1}. ^1H, ^{13}C, and ^{31}P NMR spectra were recorded in C_6D_6 and $CDCl_3$ solutions on Bruker DRX300 (300 MHz FT NMR) spectrometers; chemical shift values ^1H and ^{13}C are expressed in δ ppm relative to TMS and that of ^{31}P relative to H_3PO_4.

The dialkyldithiophosphoric acids and their sodium salts[35,36], arsenic(III)*tris*(dialkyldithiocarbamato),[22] and arsenic(III)*bis*(dialkyldithiocarbamato) chloride[22] were prepared by reported methods. Arsenic trichloride was prepared by the reaction of arsenic trioxide and thionyl chloride.[32] Sulphur was determined gravimetrically as barium sulphate.[37] Arsenic was determined iodometrically.[25]

Reaction Between Bis(dimethyldithiocarbamato)- arsenic(III) Chloride and Diethyldithiophosphate in 1:1 Molar Ratio

The solution of *bis*(dimethyldithiocarbamato)arsenic(III) chloride (0.59 g, 1.70 mmol) in benzene (~30 ml) was added dropwise to sodium diethyldithiophosphate (0.35 g, 1.70 mmol). The contents were heated under reflux for ~4 hours. Precipitated sodium chloride (0.09 g) was removed by filtration. On removing the solvent from the filtrate, pale yellow solid obtained, was recrystallized by dichloromethane. Yield –0.72 g; 85%; M.P. 127°C (Table II).

All the other derivatives were prepared by adopting the above method. Partient analytical and physicochemical data for these complexes are listed in Table II.

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