JOURNAL OF CHEMICAL RESEARCH 2006 NOVEMBER, 727–729 RESEARCH PAPER 727

Synthesis and spectroscopic studies of some heterobinuclear derivatives of bismuth(III)

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The reactions of chlorobis(dialkyldithiophosphato)bismuth(III), $[\{(RO)_2P(S)S\}_2BiCl]$ with sodium tetraisopropoxoborate(III), $NaB(OPr^i)_4$ in 1: 1 molar ratio in refluxing benzene solution yield the corresponding heterobinuclear derivatives of the type $[\{(RO)_2P(S)S\}_2Bi(\mu\text{-}OPr^i)_2B(OPr^i)_2]$ [where R = Me(**1**); Et(**2**); Pr^i(**3**); Pr^n(**4**); and Bu^n(**5**)]. All these derivatives are moisture sensitive, dark-brown, viscous liquids that are soluble in common organic solvents and have been characterised by elemental analyses and molecular weight measurements. Probable structures for **1–5** have been proposed on the basis of spectroscopic studies.

Keywords: chlorobis(dialkyldithiophosphato)bismuth(III), heterobinuclear derivatives, bidentate ligands, tetracoordinated boron

There has been renewed interest in bismuth alkoxides as potential precursors for sol-gel and chemical vapor deposition preparations of bismuth-containing superconductors[1,2] and ferroelectrics.[3-5] Although during the last two decades there has been significant development in the chemistry of heterobimetallic alkoxides of bismuth,[6-11] heterobinuclear alkoxides of bismuth involving deprotonated dithiophosphates and isopropoxymetallate groups have not yet been reported, whereas mixed-ligand dialklydithiophosphate derivatives of bismuth have appeared.[12-20] We now report the synthesis of a unique class of heterobinuclear alkoxide derivatives of bismuth(III). All these compounds have been characterised on the basis of elemental analyses (C,H, S, B and Bi) and molecular weight measurements, as well as IR and NMR (^1H, ^{13}C, ^{31}P and ^{11}B) spectroscopic evidences.

Results and discussion

Heterobinuclear derivatives of bismuth(III) supported by dialkyldithio-phosphate ligands have been synthesised by the reactions of sodium tetraisopropoxoborate(III), $NaB(OPri)_4$ with the corresponding monochlorobis-(dialkyldithiophosphato)bismuth(III)compound, $[\{(RO)_2P(S)S\}_2BiCl]$ (prepared by the reactions of metal trichloride with sodium dialkyldithiophosphates in 1 : 2 molar ratio[12]) in 1 : 1 molar ratio in refluxing benzene solution for ~ 2 h:

$$[(RO)_2P(S)S]_2BiCl+NaB(OPr^i)_4 \xrightarrow[\text{Reflux}]{C_6H_6} [(RO)_2P(S)S]_2Bi(\mu\text{-}OPr^i)_2B(OPr^i)_2 + NaCl\downarrow$$

1–5

where R= Me(**1**); Et (**2**); Pr^i(**3**); Pr^n(**4**); and Bu^n(**5**).

All the newly-synthesised heterobinuclear derivatives, **1–5,** are moisture sensitive, dark-brown coloured, viscous liquids which are soluble in common organic solvents. Molecular weight determinations of these compounds reveal their monomeric nature in freezing benzene solution.

IR spectra

Although IR data cannot distinguish between the various structural possibilities of the dialkyldithiophosphate ligands, since it is difficult to assign P = S and P–S stretching frequencies with certainty,[21-22] tentative assignments of the important characteristic bands in the IR spectra of these heterobinuclear derivatives have been made by comparing them with the IR spectral data reported earlier,[12,21-28] A strong band, due to (P=S) stretching vibrations in the region 640–690 cm^{-1} in the spectra of corresponding free dialkyldithiophosphoric acids or their sodium salts, is shifted towards lower wave number in the spectra of **1–5** and appears at 620–675 cm^{-1}.[23-24] This shifting may be attributed to the bidentate chelation of dithiophosphate moieties at the bismuth(III) centre.[12] The bands of weak to medium intensity present in the regions 535–585 cm^{-1} and 375–390 cm^{-1} have been assigned to (P–S)[23-24] and (Bi–S)[25] stretching vibrations, respectively. The medium intensity bands in the regions 945–1020 cm^{-1} and 750–840 cm^{-1} are assigned to [(P)–O–C] and [P–O–(C)] stretching modes respectively.[22,26] These heterobinuclear derivatives also show additional bands in the regions 495–520 cm^{-1} and 1280–1310 cm^{-1} due to Bi–O[27] and B–O[28] stretching vibrations, respectively.

NMR spectra

^1H NMR spectra of **1–5** (Table 1) exhibit two sets of signals for terminal and bridging isopropoxy groups in the regions 1.16–1.22, 1.33–1.48 ppm (doublets) $\{CH(C\mathbf{H}_3)_2\}$, and 4.26–4.75, 4.52–4.89 ppm (multiplets) $\{C\mathbf{H}(CH_3)_2\}$, respectively. All alkyl protons of the (RO) group attached to the P atom are observed at the expected positions. Splitting due to coupling between protons and phosphorus has been observed in the signals of the alkyl groups attached to the OP(S)S group (Table 1).

In the proton-decoupled ^{13}C NMR spectra (Table1) of **1–5,** an upfield shift has been observed in the positions of the signals of different alkyl groups of the dialkyldithiophosphate moieties as compared to their positions in corresponding parent mixed chlorobis-(dialkyldithiophosphato)bismuth (III) compounds. This shift may be due to the fact that the coordination number of bismuth has changed from five to six. Two sets of signals due to terminal and bridging isopropoxy groups have been observed in the spectra of **1–5** in the ranges 16.15–23.70, 25.34–31.26 ppm $\{CH(\mathbf{C}H_3)_2\}$ and 65.12–72.16, 72.85–73.59 ppm $\{\mathbf{C}H(CH_3)_2\}$, respectively.

The ^{31}P NMR spectra exhibit only one signal for each compound in the range 91.64–96.05 ppm (Table 1). The position of the ^{31}P NMR resonances in this range has been interpreted in terms of the bidentate nature of the dialkyldithiophosphate ligands[29] in **1–5**.

^{11}B NMR spectra also exhibit only one signal in the range –17.58 to –21.40 ppm (Table 1) for **1–5**. The occurrence of this signal in this range indicates the presence of tetracoordinated boron centre in these compounds.[30]

Structure elucidation

Although it is quite difficult to comment on the molecular structure of these new heterobinuclear derivatives in the solid state without actual X-ray crystal structure analysis of at least

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Table 1 ^1H, ^{13}C, ^{31}P and ^{11}B NMR spectral data (δppm) of the heterobinuclear derivatives $[\{(RO)_2P(S)S\}_2Bi(\propto\text{-}OPr^i)_2B(OPr^i)_2]$, **1–5**

S. No.	Heterobinuclear derivative	^1H NMR chemical shift (δ ppm)[a]	^{13}C NMR chemical shift (δ ppm)	^{31}P NMR chemical shift (δ ppm)	^{11}B NMR chemical shift (δ ppm)
1	$[\{(CH_3O)_2P(S)S\}_2Bi$ $(\propto\text{-}OPr^i)_2B(OPr^i)_2]$	1.20,d,12H, $(CH(C\mathbf{H}_3)_2)_T$; 1.38, d, 12H, $(CH(C\mathbf{H}_3)_2)_B$; 3.77, d, 12H, $(OC\mathbf{H}_3)_{dtp}$; [$^3J(^{31}P\text{-}O\text{-}C\text{-}^1H) = 13.4$ Hz]; 4.62, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$; 4.87, m, 2H, $(C\mathbf{H}(CH_3)_2)_B$;	23.70 $(CH(\mathbf{C}H_3)_2)_T$; 25.35 $(CH(\mathbf{C}H_3)_2)_B$; 53.80 $(O\mathbf{C}H_3)_{dtp}$; 71.99 $(\mathbf{C}H(CH_3)_2)_T$; 73.32 $(\mathbf{C}H(CH_3)_2)_B$	91.64	– 17.58
2	$[\{(CH_3\,CH_2O)_2P(S)S\}_2Bi$ $(\propto\text{-}OPr^i)_2B(OPr^i)_2]$	1.19, d, 12H, $(CH(C\mathbf{H}_3)_2)_T$; 1.25, t, 12H, $(C\mathbf{H}_3)_{dtp}$; $^3J\,(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 1.37, d, 12H, $(CH(C\mathbf{H}_3)_2)_B$; 4.02, dq, 8H, $(OC\mathbf{H}_2)_{dtp}$; $^3J(^{31}P\text{–}O\text{–}C\text{–}^1H) = 9.5$, $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 4.33, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$; 4.60, m, 2H, $(C\mathbf{H}(CH_3)_2)_B$;	15.93 $(CH_2\mathbf{C}H_3)_{dtp}$; 16.15 $(CH(\mathbf{C}H_3)_2)_T$; 25.34 $(CH(\mathbf{C}H_3)_2)_B$; 64.79 $(O\mathbf{C}H_2)_{dtp}$; 65.12 $(\mathbf{C}H(CH_3)_2)_T$ 72.86 $(\mathbf{C}H(CH_3)_2)_B$	96.05	– 18.55
3	$[\{(CH_3)_2CHO\}_2P(S)S\}_2Bi$ $(\propto\text{–}OPr^i)_2B(OPr^i)_2]$	1.21, d, 12H, $(CH(C\mathbf{H}_3)_2)_T$; 1.34, d, 12H, $(CH(C\mathbf{H}_3)_2)_B$; 1.40, d, 24H, $(C\mathbf{H}_3)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}\,^1H) = 6.8$ 4.38, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$; 4.62, m, 2H, $(C\mathbf{H}(CH_3)_2)_B$; 4.90, m, 4H, $(OC\mathbf{H})_{dtp}$; $^3J(^{31}P\text{–}O\text{–}C\text{–}\,^1H) = 10.0$, $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.8$	23.54 $(CH(\mathbf{C}H_3)_2)_T$; 23.80 $(CH(\mathbf{C}H_3)_2)_{dtp}$; 25.37 $(CH(\mathbf{C}H_3)_2)_B$; 66.37 $(\mathbf{C}H(CH_3)_2)_T$; 73.20 $(\mathbf{C}H(CH_3)_2)_{dtp}$; 73.59 $(\mathbf{C}H(CH_3)_2)_B$	92.94	–21.40
4	$[\{(CH_3CH_2CH_2O)_2P(S)S\}_{2]}$ $Bi(\propto\text{–}OPr^i)_2B(OPr^i)_2$	0.72, t, 12H, $(C\mathbf{H}_3)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 1.19, d, 12H, $(CH(C\mathbf{H}_3)_2)_T$; 1.35, d, 12H, $(CH(C\mathbf{H}_3)_2)_B$; 1.64, m, 8H, $(C\mathbf{H}_2CH_3)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 4.04, dt, 8H, $(OC\mathbf{H}_2)_{dtp}$; $^3J(^{31}P\text{–}O\text{–}C\text{–}^1H) = 9.0$, $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 4.64, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$; 4.83, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$;	13.07 $(CH_2CH_2\mathbf{C}H_3)_{dtp}$; 22.38 $(CH(\mathbf{C}H_3)_2)_T$; 29.06 $(CH(\mathbf{C}H_3)_2)_B$; 32.15 $(CH_2\mathbf{C}H_2CH_3)_{dtp}$; 69.43 $(O\mathbf{C}H_2)_{dtp}$; 71.67 $(\mathbf{C}H(CH_3)_2)_T$; 72.85 $(\mathbf{C}H(CH_3)_2)_B$	93.60	–19.26
5	$[\{(CH_3CH_2CH_2CH_2O)_2P(S)S\}_2Bi$ $(\propto\text{–}OPr^i)_2B(OPr^i)_2]$	0.85, t, 12H, $(C\mathbf{H}_3)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 1.03, d, 12H, $(CH(C\mathbf{H}_3)_2)_T$; 1.26, d, 12H, $(CH(C\mathbf{H}_3)_2)_B$; 138, m, 8H, $(C\mathbf{H}_2CH_3)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 1.57, m, 8H, $(OCH_2C\mathbf{H}_2)_{dtp}$; $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 2.78, dt, 8H, $(OC\mathbf{H}_2)_{dtp}$; $^3J(^{31}P\text{–}O\text{–}C\text{–}^1H) = 9.0$, $^3J(^1H\text{–}C\text{–}C\text{–}^1H) = 6.0$ 4.46, m, 2H, $(C\mathbf{H}(CH_3)_2)_T$; 4.69, m, 2H, $(C\mathbf{H}(CH_3)_2)_B$;	13.53 $(CH_2CH_2CH_2\mathbf{C}H_3)_{dtp}$; 21.78 $(CH_2CH_2CH_2\mathbf{C}H_3)_{dtp}$; 23.72 $(CH(\mathbf{C}H_3)_2)_T$; 31.26 $(CH(\mathbf{C}H_3)_2)_B$; 33.37 $(CH_2\mathbf{C}H_2CH_2CH_3)_{dtp}$; 66.09 $(O\mathbf{C}H_2)_{dtp}$; 72.16 $(\mathbf{C}H(CH_3)_2)_T$; 73.25 $(\mathbf{C}H(CH_3)_2)_B$	91.58	–17.84

[a] Coupling constants are in Hz. $_B$ = bridging, $_T$ = terminal, $_{dtp}$ = dialkyldithiophosphate, d = doublet, t = triplet, m = multiplet, dt = doublet of triplets, dq = doublet of quartets.

Table 2 Physical and analytical data for the heterobinuclear derivatives, $[\{(RO)_2P(S)S\}_2Bi(\mu\text{-}OPr^i)_2B(OPr^i)_2]$, **1–5**

Entry	Heterobinuclear derivative	Empirical formula and yield (%)	Colour/physical state	Analysis (%) found (calcd)					Mol. wt. found (calcd)
				Bi	S	B	C	H	
1	$[\{(CH_3O)_2P(S)S\}_2Bi\ (\mu\text{–}OPr^i)_2B(OPr^i)_2]$	$C_{16}H_{40}O_8P_2S_4BBi$ 90	Dark brown/viscous liquid	26.50 (27.12)	16.33 (16.65)	1.26 (1.40)	24.54 (24.94)	5.13 (5.24)	768 (770.48)
2	$[\{(CH_3CH_2O)_2P(S)S\}_2Bi\ (\mu\text{–}OPr^i)_2B(OPr^i)_2]$	$C_{20}H_{48}O_8P_2S_4BBi$ 93	Dark brown/viscous liquid	25.06 (25.28)	15.23 (15.52)	1.22 (1.31)	28.92 (29.06)	5.60 (5.86)	822 (826.58)
3	$[\{(CH_3)_2CHO\}_2P(S)S\}_2Bi\ (\mu\text{–}OPr^i)_2B(OPr^i)_2]$	$C_{24}H_{56}O_8P_2S_4BBi$ 94	Dark brown/viscous liquid	23.17 (23.68)	14.18 (14.53)	1.15 (1.22)	32.36 (32.65)	6.18 (6.41)	877 (882.69)
4	$[\{(CH_3CH_2CH_2O)_2\ P(S)S\}_2Bi(\mu\text{–}OPr^i)_2B(OPr^i)_2]$	$C_{24}H_{56}O_8P_2S_4BBi$ 92	Dark brown/viscous liquid	23.23 (23.68)	14.15 (14.53)	1.12 (1.22)	32.26 (32.65)	6.22 (6.41)	875 (882.69)
5	$[\{(CH_3CH_2CH_2CH_2O)_2P(S)S\}_2(\mu\text{–}OPr^i)_2B(OPr^i)_2]$	$C_{28}H_{64}O_8P_2S_4BBi$ 93	Dark brown/viscous liquid	22.02 (22.26)	13.40 (13.66)	1.07 (1.15)	35.48 (35.82)	6.76 (6.88)	935 (938.79)

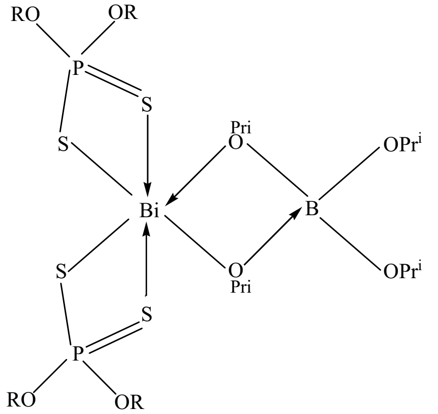

Fig. 1 Proposed structure of compounds **1–5.** [R=Me **(1)**; Et (**2**); (**3**); Pr^n (**4**); and Bu^n (**5**)]

one of the compounds, in view of the ^{31}P NMR data, which indicate the bidentate nature of the dithiophosphate ligands in these compounds, and the presence of terminal and bridging isopropoxy groups and tetracoordinated boron, the following structure (Fig. 1) may be tentatively proposed for **1**–**5**.

Experimental

All reactions have been carried out in a moisture-free environment. Solvents (E.Merck) were carefully dried by standard methods before use. Mixed monochlorobismuth(III)dialkyldithiophosphates were prepared by the reactions of bismuth(III) chloride and sodium salts of corresponding dialkyldithiophosphoric acids in 1 : 2 molar ratio in refluxing benzene solution.[12] Sodium tetraisopropoxoborate, $NaB(OPr^i)_4$ was prepared according to the literature procedure.[31] Sulfur was estimated by Messenger's method.[32] Boron[32] and bismuth[32] were determined by the methylborate and complexometric methods respectively.

Elemental analyses (C and H) were determined on a Perkin Elmer Series II 2400 C,H,N analyser. Molecular weights were determined cryoscopically in freezing benzene solution using a Beckmann thermometer. The IR spectra were recorded on a Nicolet DX FT IR spectrophotometer as liquid films in a CsI cell in the range 4000–200 cm^{-1}. ^{1}H, ^{13}C, ^{31}P and ^{11}B NMR spectra were recorded in $CDCl_3$ solution with a JEOL FT AL 300 MHz spectrometer; chemical shift values of ^{1}H and ^{13}C are expressed in δ ppm relative to TMS as an internal reference and those of ^{31}P and ^{11}B in δ ppm relative to H_3PO_4[29] and $B(OCH_3)_3$[30] as external standards respectively.

Synthesis of heterobinuclear derivative **1**: Freshly prepared sodium tetraisopropoxoborate (0.76 g, 2.81 mmol) was mixed with a benzene solution of $[\{(MeO)_2P(S)S\}_2BiCl]$ (1.57 g, 2.81 mmol), and the resulting reaction mixture, after stirring at room temperature for ~ 4 h, was finally refluxed for ~2 h. The precipitated NaCl (0.16 g, 2.74 mmol) was removed by filtration. Removal of volatile components from the filtrate under reduced pressure yielded derivative **1,** 1.98 g (91.24%)**.** For purification, the crude compound was dissolved in a small amount of benzene and then *n*-hexane was added until the compound began to separate. This solution was kept overnight at ~ 0ºC. After decanting off the solvent, the compound was dried under vacuum at room temperature to give analytically pure derivative **1** in 89.9% yield (1.95 g).

A similar procedure was adopted for the syntheses and purification of all other new heterobinuclear derivatives, **2**–**5**. The physical and analytical data of these derivatives are summarised in Table 2. Amounts of the reactants actually used and the NaCl precipitated are shown below in brackets.

2: $NaB(OPr^i)_4$ (0.49 g, 1.81 mmol) and $[(EtO)_2P(S)S]_2BiCl$ (1.11 g, 1.80 mmol); NaCl (0.10 g).

3: $NaB(OPr^i)_4$ (0.69 g, 2.55 mmol) and $[(Pr^iO)_2P(S)S]_2BiCl$ (1.71 g, 2.55 mmol); NaCl (0.15 g).

4: $NaB(OPr^i)_4$ (0.34 g, 1.26 mmol) and $[(Pr^nO)_2P(S)S]_2BiCl$ (0.85 g, 1.27 mmol); NaCl (0.07 g).

5: $NaB(OPr^i)_4$ (0.39 g, 1.44 mmol) and $[(Bu^nO)_2P(S)S]_2BiCl$ (1.04 g, 1.43 mmol); NaCl (0.08 g).

The award of a Senior Research Fellowship to Pankaj Kumar Sharma by the University Grants Commission, New Delhi, is gratefully acknowledged.

Received 30 June 2006; accepted 15 September 2006
Paper 06/4055

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