

Synthesis and Spectroscopic Characterization of Eight Chloro Cyclopentadienyl Titanium Bis (O, O-Dialkyl and Alkylene Dithiophosphate) Compounds

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Abstract

A series of new complexes, $\text{CpTiCl}[\text{S}_2\text{P}(\text{OR})_2]_2$ (where R) Et, n-Pr, i-Pr, Bu¹, Ph) and $\text{CpTiCl}[\text{S}_2\text{POGO}]_2$ (where G) -CH₂CMe₂CH₂-, -CMe₂CMe₂-) have been prepared by the drop wise addition of the appropriate O, O'-dialkyl or -alkylene dithiophosphoric acid to cyclopentadienyl titanium trichloride in 1:2 molar ratio and refluxed in benzene solution. The new compounds were characterized by molecular weight measurements elemental analyses and spectroscopic studies (¹H, ¹³C, and ³¹P NMR, and infrared). We suggest a distorted tetrahedron structure of these new complexes and the dithioligand behaves as bidentate ligand.

Keywords

Chloro Cyclopentadienyl Titanium (IV) Bis Dialkyl, Alkylene Dithiophosphate

1. Introduction

Dithiophosphate complexes of both transition and non-transition elements have received considerable interest due to their wide diversity in chemical [1]-[6] and biological systems [7] [8] O, O-Dialkyl and alkylene dithiophosphate ligands can coordinate to metal atoms in a monodentate or anisobidentate fashion [9] [10]. More recent applications of thio compounds are in the production of nanoparticles of metal sulfides [11] [12]. Metal thio compounds are extensively used as pesticides [13], corrosion inhibitors [14], agricultural reagents [15], and quite

recently in therapy for HIV infections [16]. Moreover, some of these thio complexes are also known to show antitumor properties [17] [18] and their antioxidant properties could be of importance for treating Alzheimer's disease [19]. A survey of literature on dithiophosphate derivatives of titanium and organotitanium reveals that only simple derivatives (e.g., those containing organic and halo substituents on titanium in addition to the dithiophosphate group) have been described [20] [21]. In this paper and continuation of our work we hereby report some novel cyclopentadienyl titanium chloro dithiophosphate complexes.

2. Experimental

All the reactions were carried out in air and moisture-free conditions. Solvents (benzene and chloroform) and alcohols (ethanol, n-propanol, isopropanol and iso-butanol) were dried before use, by standard methods. CpTiCl₃ (Sigma Aldrich) was used as such. dialkyl and alkylenedithiophosphoric acids or their sodium/ammonium salts were prepared by the methods reported in the literature. Sulfur was estimated gravimetrically as barium sulfate. Chlorine estimated by Volhard's method.

3. Measurements

Molecular weights were determined cryoscopically in benzene. IR spectra, using CsI cells, were recorded as neat liquids or in the form of Nujol mulls (in case of solid compounds) on a Perkin-Elmer 577 spectrometer in the range 4000 - 200 cm⁻¹. ¹H and ¹³C spectra were recorded on a Jeol-FT NMR spectrometer-LA300 spectrophotometer using TMS as an external standard. ³¹P NMR (proton decoupled) spectra were recorded in CDCl₃ using 85% H₃PO₄ as an internal standard on the same instrument.

4. Results and Discussion

Chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate) have been prepared by the reaction of cyclopentadienyl titanium trichloride with acid or sodium salt of O, O-dialkyl and alkylenedithiophosphoric acids in 1:2 molar ratios in refluxing benzene as in Equations (1) and (2).



where R = Et, Pr-n, Pr-i, Bu-i and Ph



G = -CH₂CMe₂CH₂-, -CH₂CEt₂CH₂- and -CMe₂CMe₂.

The color of the reaction medium changed yellow-brown color with the progress of reaction. Chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate) derivatives are brown color solids in these complexes. The new complexes are soluble in common organic solvents like benzene, chloroform and dichloromethane. The molecular weight of all these compounds determined by cryoscopic method in benzene indicated the monomeric nature of these pro-

ducts (Table 1).

4.1. IR Spectra

IR spectra of chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate), have been recorded in the region 4000 - 400 cm^{-1} [22] [23]. The band shown by the parent acids in the region 2544 - 2400 cm^{-1} , due to SH stretching vibration, are absent for chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate) derivatives, indicating the formation of Ti-S bond with the appearance a new band in the regions 428 - 400 cm^{-1} [22] [24]. The bands present in the region 1104.0 - 1014.5 cm^{-1} and 937.3 - 800 cm^{-1} have been assigned to ν (P)-O-C and ν P-O-(C) stretching vibrations respectively. Strong to medium bands in the region 995 - 921.9 cm^{-1} are due to dioxaphosphorinane and dioxaphospholane ring vibrations [25] [26] [27]. The bands shown in the region 704.0 - 638.0 cm^{-1} can be assigned to ν P=S vibrations [28]. The bands in medium weak intensities in the region 602 - 513.0 cm^{-1} may be attributed to vibration of ν P-S asymmetric and symmetric vibrations [29]. Details regarding the individual bands have been included in Table 2.

4.2. ^1H NMR Spectra

The ^1H NMR spectrum of complexes 1 - 7 exhibit the characteristic resonance due to alkoxy and glycoxy (dithio moiety) protons (Table 3). The singlet peak at (3.1 - 3.5 ppm) in the parent ligand assigned to SH proton, is absent from the spectra of chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate) derivatives indicating deprotonation of SH group and forming of Ti-S bond [30].

4.3. ^{13}C NMR Spectra

The ^{13}C NMR spectra of a selection of chloro cyclopentadienyl titanium bis (di-

Table 1. Physical Properties and Analytical Data of Chloro Cyclopentadienyl Titanium Bis (O,O-Dialkyl and Alkylene Dithiophosphate) Compounds.

Comp. No.	Reactants (g) CpTiCl ₃ 2 NaS ₂ P(OR) ₂	Products	Yield %	Physical State	M. P. °C	Mol. Wt.	% H	% C	% S	% Ti
						Found/ (Calc.)	Found /(Calc.)	Found /(Calc.)	Found /(Calc.)	Found /(Calc.)
1	0.89 NaS ₂ P(OEt) ₂ 1.68	CpTiCl[S ₂ P(OEt) ₂] ₂ 2.09	92.4	Dark brown	154°	518.2/ (518.8)	4.3/ (4.8)	30.2/(30.0)	24.5 (24.7)	9.1/ (9.2)
						575.3/ (574.9)	5.8/ (5.7)	35.8/ (35.5)	22.6/ (22.3)	8.8/ (8.3)
2	0.97 NaS ₂ P (OPr-n) ₂ 2.08	CpTiCl[S ₂ P (OPr-n) ₂] ₂ 2.12	90.9	Yellow Powder	157°	576.9/ (574.9)	5.9/ (5.7)	35.4/ (35.5)	22.6/ (22.3)	8.2 (8.3)
						576.9/ (574.9)	5.9/ (5.7)	35.4/ (35.5)	22.6/ (22.3)	8.2 (8.3)
3	0.86 NaS ₂ P (OPr-i) ₂ 1.85	CpTiCl[S ₂ P (OPr-i) ₂] ₂ 1.97	87.5	Yellow Powder	176°	630.8/ (631.07)	6.3/ (6.5)	39.6/ (39.9)	20.4/ (20.3)	7.1/ (7.5)
						712.1/ (711.0)	3.6 (3.5)	48.3/ (48.9)	18.5/ (18.0)	6.8/ (6.7)
4	0.75 NaS ₂ P (OBU-i) ₂ 1.80	CpTiCl[S ₂ P (OBU-i) ₂] ₂ 1.91	88.8	Yellow Powder	159°	541.7/ (542.8)	4.8/ (4.6)	33.7/ (33.1)	23.1/ (23.6)	8.5/ (8.8)
						598.4/ (598.9)	5.3/ (5.5)	38.6/ (38.0)	21.6/ (21.4)	7.8/ (7.9)
5	0.64 NaS ₂ P(OPh) ₂ 1.77	CpTiCl[S ₂ P(OPh) ₂] ₂ 1.78	85.9	Yellow Powder	212°	571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)
						541.7/ (542.8)	4.8/ (4.6)	33.7/ (33.1)	23.1/ (23.6)	8.5/ (8.8)
6	0.54 NaS ₂ POCH ₂ CMe ₂ CH ₂ O 1.08	CpTiCl[S ₂ POCH ₂ CMe ₂ CH ₂ O] ₂ 1.24	93.2	Brown viscous	143°	598.4/ (598.9)	5.3/ (5.5)	38.6/ (38.0)	21.6/ (21.4)	7.8/ (7.9)
						571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)
7	0.67 NaS ₂ POCH ₂ CEt ₂ CH ₂ O 1.51	CpTiCl[S ₂ POCH ₂ CEt ₂ CH ₂ O] ₂ 1.64	90.1	Brown viscous	136°	571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)
						571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)
8	0.54 NaS ₂ POCMe ₂ CMe ₂ O 1.15	CpTiCl[S ₂ POCMe ₂ CMe ₂ O] ₂ 1.23	87.8	brown viscous	136°	571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)
						571.3/ (570.9)	5.7/ (5.1)	35.4/ (35.7)	22.8/ (22.4)	8.8/ (8.3)

Table 2. IR SPECTRAL Data (cm⁻¹) of Chloro Cyclopentadienyl Titanium Bis(O,O-Dialkyl and Alkylene Dithiophosphate Compounds.

No.	Compounds	v(P)-O-C	vP-O-(C)	Ring Vibration	v P=S	vP-S	v (Ti-S)	v (Ti-Cl)
1	CpTiCl[S ₂ P(OEt) ₂] ₂	1016.7 s	810.0 s	--	656.8 m	540.0 m	407.0 m	320
2	CpTiCl[S ₂ P (OPr-n) ₂] ₂	997.0 s	850.5 m	--	630.7 m	544.9 m	440.0 w	319
3	CpTiCl[S ₂ P (OPr-i) ₂] ₂	974.0 m	889.1 m	--	654.9 m	542.0 m	402.0 m	308
4	CpTiCl[S ₂ P (OBu-i) ₂] ₂	990.0s	820.6 s	--	650.0 m	532.0 m	413.0 w	325
5	CpTiCl[S ₂ P(OPh) ₂] ₂	1098.0 m	820.3 s	--	670.5 s	515.0 w	422.0w	326
6	CpTiCl[S ₂ POCH ₂ CMe ₂ CH ₂ O] ₂	1056.8 s	815.8 m	912.3 m	684.7 m	508.3 m	410.0 w	309
7	CpTiCl[S ₂ POCH ₂ CEt ₂ CH ₂ O] ₂	1020.4 s	830.0 m	940.0 m	660.0 m	530.0 m	407 0 m	316
8	CpTiCl[S ₂ POCMe ₂ CMe ₂ O] ₂	1010.7 s	858.8 s	923.9 m	661.7 m	580.0 m	409.0 m	326

s = strong, m = medium, w = weak and b = broad absorption bands.

Table 3. ¹H and ³¹P NMR Spectral Data of Chloro Cyclopentadienyl Titanium Bis (O, O-Dialkyl and Alkylene Dithiophosphate) Compounds.

No.	Compound	¹ H chemical shift in δ ppm CDCl ₃	³¹ P chemical shift in δ ppm (parent Acid)
1	CpTiCl[S ₂ P(OEt) ₂] ₂	1.19 t (<i>J</i> = 6.5 Hz), 12H(CH ₃)	92.9 (85.7)
		4.06, q (<i>J</i> = 6.0 Hz), 8H(OCH ₂)	
		6.46, s, 10H(C ₅ H ₅)	
2	CpTiCl[S ₂ P (OPr-n) ₂] ₂	0.76, t (<i>J</i> = 7.5 Hz), 12H(CH ₃)	102.7 (86.1)
		1.36, m (<i>J</i> = 6.5 Hz), 8H(CH ₂)	
		3.38 - 3.58, t (<i>J</i> = 7.5 Hz, 8H(OCH ₂)	
3	CpTiCl[S ₂ P (OPr-i) ₂] ₂	6.19, s, 10H(C ₅ H ₅)	93.4 (82.3)
		1.25, d (<i>J</i> = 6.6 Hz), 24H(CH ₃)	
		4.43 - 4.47, m (<i>J</i> (PH) = 12 Hz, 4H(OCH)	
4	CpTiCl[S ₂ P (OBu-i) ₂] ₂	6.32, s, 10H(C ₅ H ₅)	98.9 (85.7)
		0.77, d (<i>J</i> = 7 Hz), 24H(CH ₃)	
		1.72, m (<i>J</i> = 6.5 Hz) 4H(CH)	
5	CpTiCl[S ₂ P(OPh) ₂] ₂	3.40, d (<i>J</i> = 7 Hz), 8H(OCH ₂)	89.6 (79.9)
		6.30, s, 10H(C ₅ H ₅)	
		721 - 7.34, m, 20H(OC ₆ H ₅)	
6	CpTiCl[S ₂ POCH ₂ CMe ₂ CH ₂ O] ₂	6.72, s, 10H(C ₅ H ₅)	123.35 (77.3)
		0.84, s, 12H(CH ₃)	
		3.70, d, 8H(OCH ₂), <i>J</i> (PH) = 15.6 Hz	
7	CpTiCl[S ₂ POCH ₂ CEt ₂ CH ₂ O] ₂	6.35, s, 10H(C ₅ H ₅)	101.41 (78.50)
		0.63, t (<i>J</i> = 7.5 Hz), 12H(CH ₃)	
		1.11, q (<i>J</i> = 7.5 Hz), 8H(CH ₂)	
8	CpTiCl[S ₂ POCMe ₂ CMe ₂ O] ₂	4.04, d, 8H(OCH ₂), <i>J</i> (PH) = 16 Hz	103.7 (93.1)
		6.25, s, 10H(C ₅ H ₅)	
		1.06, s, 24H(CH ₃)	
		6.54 s, 10H(C ₅ H ₅)	

alkyl and alkylenedithiophosphate) derivatives were recorded in deuterated chloroform at ambient temperature. These compounds did not show any shift compared to corresponding carbons in the dithiophosphoric acid and salt (**Table 4**).

4.4. ³¹P NMR Spectra

Decoupled ³¹P NMR spectra for these products give a singlet. The observation of

only one ^{31}P singlet for all compounds reflects the equivalent nature of phosphorous nuclei and the purity of the compound. The values of chemical shifts according to Glidewell's [31] observation indicates bidentate chelating behavior of the ligand (Table 3).

5. Structural Elucidation

Molecular weight determination of chloro cyclopentadienyl titanium bis (dialkyl and alkylenedithiophosphate) derivatives showed monomeric nature of these compounds in benzene. Thus, on the basis of our observations for IR, NMR (^1H , ^{13}C and ^{31}P) and molecular weight determinations, the following structures are proposed for these new complexes, as shown in Figure 1 and Figure 2.

6. Conclusion

We have successfully prepared and characterized the chloro cyclopentadienyl ti-

Table 4. ^{13}C NMR Spectral Data of Some Chloro Cyclopentadienyl Titanium Bis (O, O-Dialkyl and Alkylene Dithiophosphate) Compounds.

Sl No	Compound	^{13}C Chemical shift, in ppm					
		CH_3	CH_2	CH	C	CO	C_5H_5
1.	$\text{CpTiCl}[\text{S}_2\text{P}(\text{OEt})_2]_2$	15.9 s				63.03	128.
2.	$\text{CpTiCl}[\text{S}_2\text{P}(\text{OPr-}i)_2]_2$	23.0s				72.4 s	117.3 118.0 d
3.	$\text{CpTiCl}[\text{S}_2\text{POCH}_2\text{CMe}_2\text{CH}_2\text{O}]_2$	19.9			32.9 s	77.0 77.1d	117.4 s

s = singlet, d = doublet and t = triplet.

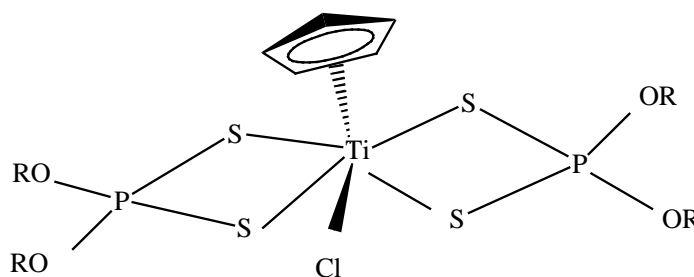


Figure 1. Suggested structure for chloro cyclopentadienyl titanium bis (dialkyldithiophosphate) complexes.

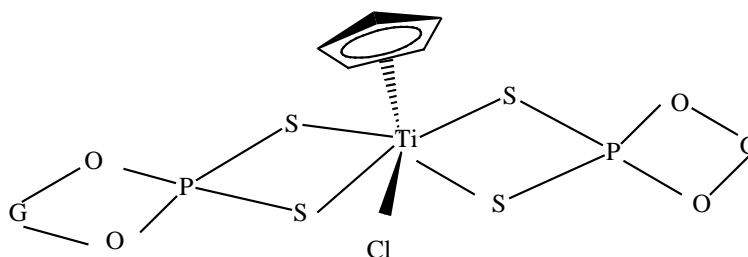


Figure 2. Suggested structure for chloro cyclopentadienyl titanium bis (alkylene dithiophosphate) complexes.

tanium bis (alkylene dithiophosphate) complexes. The IR, ^1H , ^{13}C and ^{31}P NMR spectra of all of these titanium complexes and the molecular structures of open chain $\text{CpTiCl}[\text{S}_2\text{P}(\text{OR})_2]_2$ and cyclic compounds $\text{CpTiCl}[\text{S}_2\text{POGO}]_2$ were determined. There are chemical shift differences between the ligand acids and the organotitanium complexes. These changes can be attributed to bidentate phosphorodithioate.

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