

SPECTROSCOPIC STUDIES OF ORGANOPHOSPHORUS COMPOUNDS

Part XI. Preparation and IR Spectra of Phosphobetaine Tetrachlorocobaltates

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Tetrachlorocobaltates of phosphobetaine from triarylphosphines and *p*-quinones have been prepared in order to study the result of interaction of a bulky anion. Phosphobetaine tetrachlorocobaltates have been prepared either by the interaction of cobaltous chloride with phosphobetaine or phosphobetaine hydrochloride.

A comparison of the known spectra of phosphonium iodide, bromide and their tetrachlorocobaltates with those of phosphobetaine hydrochloride and their tetrachlorocobaltates indicates that the intensity of the IR bands as well as their positions are affected on changing the halides into tetrachlorocobaltates. There is a significant (four to five fold) enhancement in intensity and some of the bands like the *k* and *l* modes, which are otherwise unresolved, are well separated. The *O* mode behaves as an X-sensitive mode and is shifted to a higher frequency.

INTRODUCTION

A series of lattice stabilized tetrahalometallates of the type $(R_4Y)MX_4$, where 'R' may be alkyl, aryl or both, 'Y' may be N, P or As, 'M' may be cobalt, nickel, iron, manganese, have been reported [1-3]. The physical properties like spectral, magnetic and crystal studies of these compounds have shown that the metal atom is tetrahedrally coordinated in these compounds. Their isolation depends upon the relative stability of the complex anion and the lattice energy of the salts. The latter is in turn dependent on the relative sizes of the ions [2, 3]. IR study of these compounds show [4-7] that the intensity and position of some diagnostic bands are changed with a change in the anion, e.g. in the case of phosphonium compounds it was noted that when the electronegativity of the anion is increased, the intensity of the X-sensitive bands is also increased. This change in intensity as pointed out earlier [8] has been attributed to the presence of electronegative groups or anions which increase the formal positive charge on the central atom via inductive effect. Since the electrical charges are disturbed, there would be a greater dipole change and hence higher intensity would be noted. The halides are, however, small and the change of intensity is not very apparent. Tetrachlorocobaltates are bulky anions, having tetrahedral symmetry with respect to the central metal atom and the effect of charge is accordingly expected

to be more marked in the case of their phosphonium compounds. In order to study the increase in intensity due to a larger anion, phosphobetaine tetrachlorocobaltates have been prepared. The present paper describes the synthesis of phosphobetaine tetrachlorocobaltates and draws a comparison of the known spectra of tetrachlorocobaltates of the phosphonium compounds.

EXPERIMENTAL

1. Phosphobetaine hydrochloride has been prepared as reported [9]. Phosphines, Quinones and their adducts were prepared as described earlier [10-12]. The apparent integrated intensities of the bands are calculated as described previously [13].

2. Preparation of *Bis*-2, 5-dihydroxyaryltriarylphosphonium tetrachlorocobaltates from 2, 5-dihydroxyaryltriarylphosphonium chloride. The reactants, 2, 5-dihydroxyaryltriarylphosphonium chloride (2 moles) and cobaltous chloride (1 mole) were allowed to react after being dissolved separately in warm absolute ethanol. The mixture was refluxed for 10 min. and filtered while hot. Addition of hot ethyl acetate to the filtrate produced blue crystals, which were separated from the mother liquor and recrystallized from ethyl acetate-ethanol mixture. The following cobaltates were prepared.

2.1 *Bis*-2, 5-dihydroxyphenyltriphenylphosphonium tetrachlorocobaltate. $[Ph_3PBQH]^+Cl^-$, 0.81 g 1.0 mmoles and $CoCl_2 \cdot 6H_2O$ 0.238 g 1 mmoles gave 0.63 g blue

*Based in part on the Ph.D thesis of M. Siddique Siddiqui, University of Karachi (1981).

crystals in 58% yield, m.p. 292°

Analysis: Calculated for $C_{48}H_{40}O_4P_2CoCl_4$, C, 61.08%; H, 4.24%; Found C, 61.06%; H, 4.13%.

2.2 *Bis-2, 5-dihydroxytolyltriphenylphosphonium tetrachlorocobaltate*. $[Ph_3PTQH]^+Cl^-$, 0.84 g. 2 mmoles and $CoCl_2 \cdot 6H_2O$, 0.238 g. 1.0 moles gave 0.75 g. of blue crystals in 70% yield, m.p. 237°.

Analysis: Calculated for $C_{50}H_{42}O_4P_2CoCl_4$, C, 61.8%; H, 4.53%; Found C, 61.67%; H, 4.9%.

2.3 *Bis-2, 5-dihydroxyphenyltri-p-tolylphosphonium tetrachlorocobaltate*. $[P.T_3PBQH]^+Cl^-$ 0.99 g. 2 moles and $CoCl_2 \cdot 6H_2O$, 0.238 g. 1.0 moles afforded 0.8 g blue crystals in 75% yield, m.p. 268.5°

Analysis: Calculated for $C_{54}H_{52}O_4P_2CoCl_4$, C, 63.09%; H, 5.06%; Found C, 62.85%; H, 4.78%.

2.4 *Bis-2, 5-dihydroxytolyl-tri-p-tolylphosphonium tetrachlorocobaltate*. $[P.T_3PTQH]^+Cl^-$ 0.426 g 1 mmoles and $CoCl_2 \cdot 6H_2O$ 0.11 g 0.5 moles gave 0.16 g blue crystals in 31% yield, m.p. 318°

Analysis: Calculated for $C_{55}H_{56}O_4P_2CoCl_4$, C, 63.69%; H, 5.3%, Found C, 63.2%; H, 5.67%.

Abbreviations: $[Ph_3P]$ = triphenylphosphine, PT_3P = tri-*p*-tolylphosphine, BQ = *p*-benzoquinone, TQ = toluquinone, NQ = 1, 4-naphthoquinone].

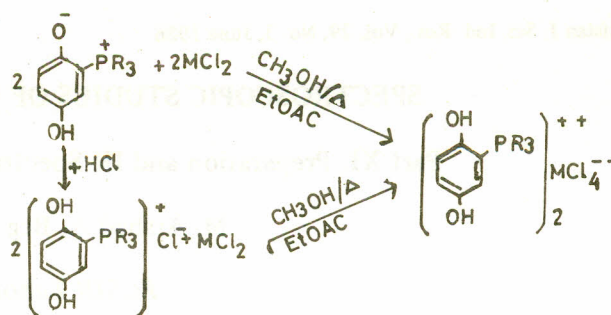
3. Preparation of *Bis-2, 5-dihydroxyaryltriarylphosphonium tetrachlorocobaltates* from phosphobetaines and cobalt chloride hexahydrate.

Tetrachlorocobaltates of phosphobetaines from triphenyl and tri-*p*-tolylphosphines and their adducts with benzoquinone and toluquinone are the only ones possible by direct reaction with excess cobalt chloride. Phosphobetaine and cobalt chloride in equimolar quantity were allowed to react in methanol. After refluxing for 30 min. the precipitates so formed were removed by filtration and the filtrate again refluxed for another 30 min. and filtered. Addition of hot ethyl acetate to the filtrate produced blue crystals which were removed from the mother liquor and recrystallized from ethanol-ethylacetate mixture.

Compounds 2.1 to 2.4 were prepared in the same manner. Naphthoquinone adducts of tertiary phosphines produced only phosphobetaine hydrochloride.

RESULTS AND DISCUSSION

Tetrachlorocobaltates of phosphobetaine have been prepared both by direct addition of phosphobetaine hydrochloride to cobalt chloride and by the reaction of phosphobetaine with cobalt chloride.



Tetrachlorocobaltate of phosphobetaine from *p*-benzoquinone and toluquinone are the only ones possible by direct reaction with excess cobalt chloride. Those with *p*-naphthoquinone do not react in like manner with the latter compound; the reaction seems to proceed only as far as 2, 5-dihydroxynaphthyltriphenylphosphonium chloride. Attempts to prepare other tetrahalometallates like the tetrachloronickelate, ferrate, cuprate etc., by the same procedure remained unsuccessful, although N.S Gills and Nyholm [14] have reported the isolation of cobaltates and nickelates of the tetraethylammonium, methyltriphenylarsonium, and ethyltriphenylphosphonium salts. The tetrachloronickelates, according to them, are impure while cobaltates are pure and stable. The crystal field stabilization energy of the tetrahedral complexes of metals like nickel, iron and copper are 8.6, 8 and 6 Kcal/g atom of metal respectively, while that of cobalt is 14 Kcal/g atom of cobalt. The tetrahedral complexes of nickel, iron and copper are therefore unlikely to be stable which is probably one of the reasons that these metals do not form the lattice stabilized addition products with phosphobetaine hydrochloride while cobalt chloride does so easily.

Infrared studies. It is observed in compounds like $(R_4Y)^+X^-$ [5-7], where R may be alkyl/aryl, Y, may be P, As, and X, may be halogen or tetrahalometallates, that the intensity and position of some infrared bands, specially the (C-C) skeletal, assigned to k, m, n, and o modes vary in intensity depending on the nature of substituents on the aromatic ring. For example the intensity of mode k, which absorbs at $1580 \pm 5cm^{-1}$ in triphenylphosphine, alkyltriphenylphosphonium halides and their tetrahalcobaltates is as shown below:

Compound	Apparent integrated intensity
1. Ph_3P	126
2. $EtPh_3PI$	321
3. $(EtPh_3P)_2CoI_4$	1420

4. nPrPh ₃ PI	327
5. (nPrPh ₃ P) ₂ CoI ₄	3333
6. nPrPh ₃ PBr	1322
7. (nPrPh ₃ P)CoBr ₄	3356

The shifting of the band position for mode o, n and q may be noted from the following example:

EtPh ₃ PI	(EtPh ₃ P) ₂ CoI ₄	
1320	1340	mode (o)
1435	1440	mode (n)
1110	1115	X-sensitive mode (q)

From the above data, it may be noted that the intensity of different modes in the phosphonium compounds and tetrachlorocobaltates is related to the electronegativity of the anion. The electronegative groups or anions attract electron density from the central atom and increase the positive charge on the central atom. Thus there would be a greater dipole change and hence higher intensity would be noted.

Similar changes in intensity and position of certain bands in the infrared region have been observed in phosphobetaine hydrochloride viz, 2, 5-dihydroxyphenyltriphenylphosphonium, 2, 5-dihydroxytolyltriphenylphosphonium, 2, 5-dihydroxyphenyltri-*p*-tolylphosphonium and 2, 5-dihydroxytolyltri-*p*-tolylphosphonium chloride and their tetrachlorocobaltates, viz. *bis*-2, 5-dihydroxyphenyltriphenylphosphonium, *bis*-2, 5-dihydroxytolyltriphenylphosphonium, *bis*-2, 5-dihydroxyphenyltri-*p*-tolylphosphonium and *bis*-2, 5-dihydroxytolyltri-*p*-tolylphosphonium tetrachlorocobaltates.

The infrared spectra of the cobaltates are much similar to those of phosphobetaine hydrochlorides excepting the intensity which is greater in all cases than that of the phosphobetaine hydrochloride. In some cases slight shifts in the frequencies have been noted. Certain bands, for example, at 1450, 1410, 1250 ± 5 cm⁻¹ which were not completely resolved in phosphobetaine hydrochloride are fully resolved in tetrachlorocobaltates and their intensity is also enhanced. This absorption pertaining to the phosphonium moiety does not suffer any shift which shows that the basic structure remains the same.

The position and integrated intensity of the absorption bands in the 1600-670 cm⁻¹ region alongwith their assignments are listed in Tables 1-2. The spectra of all compounds have broad bands of high intensity in the 3300-2600 cm⁻¹ region, which is due to the O-H stretching vibration. The

bands are very broad for the integrated intensity measurements.

It has been shown earlier [15] that integrated intensity and position of various infrared bands change with the vibration in the electronegativity of the anion. In phosphobetaine hydrochlorides and their tetrachlorocobaltates, similar changes have been observed. For example, the integrated intensity of 1585 ± 5 cm⁻¹ band (mode K) in phosphobetaine chloride increases nearly three to four times in phosphobetaine tetrachlorocobaltates as shown below:

Compound	Apparent integrated intensity
Ph ₃ P	126
[Ph ₃ PBQH] ⁺ Cl ⁻	588
[Ph ₃ PBQ] ₂ ⁺⁺ CoCl ₄ ⁻	1561
[Ph ₃ PTQH] ⁺ Cl ⁻	1191
[Ph ₃ PTQ] ₂ ⁺⁺ CoCl ₄ ⁻	3876
T ₃ P	76
[T ₃ PBQH] ⁺ Cl ⁻	580
[T ₃ PBQ] ₂ ⁺⁺ CoCl ₄ ⁻	1154
[T ₃ PTQH] ⁺ Cl ⁻	1098
[T ₃ PTQ] ₂ ⁺⁺ CoCl ₄ ⁻	6540

The position of the bands at 1280 ± 10, 1225 ± 10 and 1190 ± 5 cm⁻¹ in the phosphobetaine hydrochlorides are shifted to 1305 ± 10, 1230 ± 10, and 1200 ± 5 cm⁻¹ respectively in the phosphobetaine tetrachlorocobaltates.

The large enhancement in the intensity of the bands corresponding to the tetrachlorocobaltates is most likely due to the presence of two molecules of phosphonium ions coordinated to the transition metal. The effect appears to be due to the presence of twice as many chromophores as in phosphobetaine alone. The shifting of the position from phosphobetaine hydrochloride to tetrachlorocobaltates may be related to change in the electronegativity of the anion. The electronegative groups would increase the formal positive charge of the central atom by withdrawing the electron density and shifting the frequencies to lower wavelength. This effect was observed on recording the C-C stretching vibration, mode (O) of the trialkylphosphonium bromide or iodide [16] which absorbs at 1320 ± 5 cm⁻¹ in the cobaltates. The shifting of the bands in the same direction implies larger dipole change and a further gain in crystal field stabilization energy. It is also noted that the position is still higher by about 10 to 15 cm⁻¹ in the case of tetrachlorocobaltates of tri-*p*-tolylphosphine and analogues compared with that of tetrachlorocobaltates of

Table 1. Vibrational frequencies and apparent integrated intensities of phosphobetaine hydrochloride.

$[\text{Ph}_3\text{PBQH}]^+\text{Cl}^-$		$[\text{Ph}_3\text{PTQH}]^+\text{Cl}^-$		$[\text{p-T}_3\text{PBQH}]^+\text{Cl}^-$		$[\text{p-T}_3\text{PTQH}]^+\text{Cl}^-$		Assignments
Cm^{-1}	A	Cm^{-1}	A	Cm^{-1}	A	Cm^{-1}	A	
1605	504	1610	1208	1595	2216	1590	1098	ν (C-C) mode (k)
1585	588	1585	1191	1580	580			
1560	406	1540	sh	1560	370	1560	276	ν (C-C) mode (l)
1540	393			1540	325			
1505	393	1505	692	1500	4680	1495	683	ν (C-C) mode (m)
1480	1888	1480	1586	1490	sh	1450	835	
1455	sh	1450	sh	1450	sh			
1440	812	1440	3814	1430	1826	1405	3173	ν (C-C) mode (n)
1430	8095	1430	3814	1400	1240			
1410	sh	1410	sh					
1380	721	1380	sh	1380	557	1375	484	δ_{as} (C-H)
1350	5476	1340	1504	1360	sh	1360	443	
1310	1067	1310	1423	1315	2817	1310	520	ν (C-C) mode (o)
1280	1545	1290	1463	1270	2382	1285	487	δ_{Sym} (C-H)
1250	1878	1255	621			1260	282	β (O-H)
1230	1726	1230	1632	1230	sh	1230	374	β (C-H) mode (e)
1215	2084	1205	685	1210	1545	1200	711	β (C-H) mode (a)
1190	sh	1190	sh	1190	1191	1190	655	
1165	882	1160	sh					β (C-H) mode (c)
1110	3996	1110	6528	1110	5460	1110	805	X-sensitive mode (q)
1060	613	1070	sh	1065	512			β (O-H)
1030	939	1040	2382	1040	811	1040	1014	β (C-H) mode (b)
1000	1208	1000	772	1000	282	1020	627	Ring breathing mode (p)
885	939	890	2078	875	555	890	277	γ (C-H) mode (i)
840	721	855	671	835	470	830	242	γ (C-H) mode (g)
830	sh	825	484	825	588			

(Continued ...)

(Table 1, continued)

790	1114	755	205	810	1991	810	1082	isolated Hydrogen
755	1775			790	443			
735	1058	730	1125	720	sh	735	sh	γ (C-H) mode (f)
725	1264	720	3264					
715	1162	700	4937	710	783	710	381	X-sensitive mode (r)
695	3332	690	4937	700	541	680	357	ϕ (C-C) mode (v)

Table 2. Vibrational frequencies and apparent integrated intensities of phosphobetaine tetrachlorocobaltates.

$(\text{Ph}_3\text{PBQ})_2\text{CoCl}_4$		$(\text{Ph}_3\text{PTQ})_2\text{CoCl}_4$		$(p\text{-T}_3\text{PBQ})_2\text{CoCl}_4$		$(p\text{-T}_3\text{PTQ})_2\text{CoCl}_4$		Assignment
Cm^{-1}	A	Cm^{-1}	A	Cm^{-1}	A	Cm^{-1}	A	
1590	1561	1585	3876	1580	1154	1590	6540	ν (C-C) mode (k)
1580	4253							
1575	4368	1575	3504	1550	1045	1560	1191	ν (C-C) mode (l)
1555	1288	1540	1117					
1540	1309							
1505	1612	1505	2173	1510	5453	1510	1803	ν (C-C) mode (m)
1490	12110	1490	3223					
1480	3370	1480	2719					
1440	12710	1450	2052	1450	5220	1450	4618	ν (C-C) mode (n)
1430	12710							
1380	sh	1380	2340	1375	sh	1375	2158	δ_{as} (C-H)
1350	5429	1360	1612	1360	1658	1360	3018	
1340	sh	1340	1476	1320	5439	1315	1705	ν (C-C) mode (o)
1300	13820	1310	6353					
1270	4693	1275	6259	1275	3607	1285	2417	β (O-H)
1250	5429	1255	2340	1260	5220	1260	2158	
1240	2184	1240	6842					β (C-H) mode (e)
1220	14340	1200	10180	1220	sh	1225	sh	
1205	sh	1160	4680	1200	7112	1195	5131	β (C-H) mode (a)
						1185	2958	
1160	4025							β (C-H) mode (c)
1135	5439	1140	4368	1140	2952	1140	2447	

(Continued)

(Table 2, continued)

1110	11410	1110	15210	1110	12400	1110	1474	X-sensitive mode (h)
1105	7183							
1060	3234			1070	2787			β (O-H)
1030	2089	1040	6995	1040	2537	1040	5849	β (C-H) mode (b)
		1020	sh	1020	2620	1020	2487	
1000	3275	1000	2184	980	sh	980	967	Ring breathing mode (p)
885	1675	885	1908	880	2714	880	2712	γ (C-H) mode (i)
870	1330	865	1435					
830	3969	825	1565	830	sh	825	sh	γ (C-H) mode (g)
790	4742	790	sh					γ (C-H) isolated hydrogen
755	9124	755	5984	800	4876	810	7188	
730	5131	730	1521					γ (C-H) mode (f)
725	7183	720	4867					
710	4945	700	1936	710	5876	710	1705	X-sensitive mode (r)
690	6908	690	4788	695	2214	670	3748	ϕ (C-C) mode (v)

triphenylphosphine analogues which absorb at 1300 and 1310 cm^{-1} . The small shifts in the position of these two species might be due to the difference in their dipole moments. The dipole moment [17] of triphenylphosphine is 1.43 D and that of tri-*p*-tolylphosphine is 1.94 D. In the trisubstituted compounds of phosphorus, e.g. triphenylphosphine [17], the lone pair on the phosphorus atom causes some changes in the structure (i.e. change in the bond distances or bond angles of the phenyl rings). In the tetrasubstituted compounds, the positive charge develops on the central atom which polarizes the rings and alters their electron density. In the polarized state, the rings are expected to interact with the infrared radiation giving higher intensity as compared with trisubstituted compounds. The tetrachlorocobaltates of tri-*p*-tolylphosphine and analogues would be more polar than the cobaltates of triphenylphosphine analogues. The difference in polarity results in a shift of the bands to higher frequency.

A comparison of the known spectra of phosphonium iodide, bromide and their tetrachlorocobaltates was made with those of phosphobetaine hydrochloride and their tetrachlorocobaltates. It is suggested that there are significant shifts in the position of the bands as well as an increase

in intensity. The reason may be the same as stated for *bis* (alkyltriphenylphosphonium) tetrachlorocobaltates, i.e. interaction of the two parts of the molecules and also due to the presence of cobaltate anion.

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The yield of the product was increased by increasing the temperature of the reflux. The maximum yield was obtained at the reflux temperature. It was found that the rate of oxidation of 2-pyridine

aldehyde decreased with increasing temperature. The yield of the product was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M.

The rate of oxidation of 2-pyridine aldehyde was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M.

At room temperature (30°C) the oxidation was observed even in the presence of a large amount of water. The corresponding aromatic aldehyde was obtained in poor yields. The maximum yield was obtained at the reflux temperature. It was found that the rate of oxidation of 2-pyridine

aldehyde decreased with increasing temperature. The yield of the product was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M.

A comparison of the rate of oxidation of 2-pyridine aldehyde with that of 2-pyridone showed that the rate of oxidation was more than 2-pyridone. The rate of reaction was more than 2-pyridone. The mechanism of degradation of 2- and 4-carboxyl groups on pyridine is not clear. It has been reported that the rate of oxidation of 2-pyridine

aldehyde decreased with increasing temperature. The yield of the product was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M. It is known that the rate of oxidation of 2-pyridine aldehyde decreases with increasing temperature. The yield of the product was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M.

Results of the oxidation of 2-pyridone using cobalt(II) acetate and potassium persulphate at different temperatures and times are given in Table I and II respectively.

- (i) Cobalt(II) acetate (0.01 M)
- (ii) Cobalt(II) acetate (0.02 M)
- (iii) Potassium persulphate (0.01 M)
- (iv) Potassium persulphate (0.02 M)

In view of the importance of pyridine aldehydic acids in the field of oxidation of aldehydes both from industrial and laboratory points of view.

RESULTS AND DISCUSSION

In this paper we report the results of the oxidation of 2-pyridine aldehyde by the action of the catalytic effect of cobalt(II) acetate in the oxidation of 2-pyridone in strongly acidic medium. Certain rates of cobalt(II) acetate and potassium persulphate have been used in the oxidation of 2-pyridone. The maximum yield of the product was obtained at a concentration of 0.1 M. The rate of oxidation of 2-pyridone decreased with increasing temperature. The yield of the product was increased by increasing the concentration of the oxidant. The maximum yield was obtained at a concentration of 0.1 M.

Table I Oxidation of 2-pyridone using cobalt(II) acetate as catalyst

Sr.	Time in hr	Temperature (°C)	Rate of flow of oxygen	Yield (%)
1	30	70	0.1 liter	10
1	30	110	0.1 "	7
1	30	110	0.1 "	9
2	40	110	0.1 "	10
2	40	170	0.1 "	10
2	30	170	0.1 "	11