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ELECTRONIC EFFECTS IN CYCLIC PHOSPHATES, PHOSPHONATES, AND PHOSPHINATES

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The acid–base properties of acyclic phosphoryl compounds have been thoroughly studied [1-12]. On the other hand, there is very little data on the properties of the P=O group in phosphorus heterocycles; the proton-acceptor activity of the phosphoryl group of 2-oxo-1,3,2-dioxaphosphorinanes is largely determined by the P atom exocyclic substituent, the effect from the ring substituents being much less pronounced [13]. The effect of the geometry on the proton-acceptor activity of the cyclic phosphoryl compounds has been repeatedly noted [1, 14, 15].

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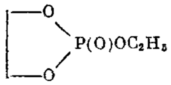
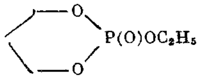
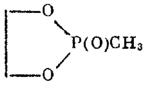
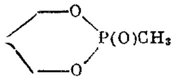
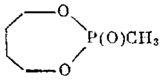
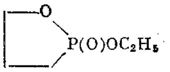
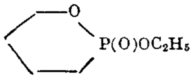
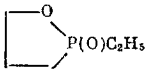
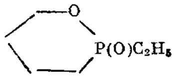
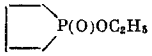
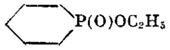
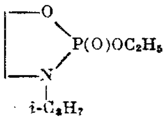
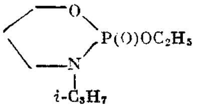
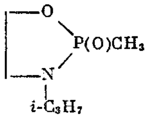
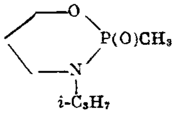
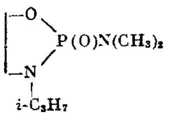
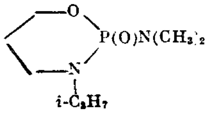
TABLE 1. Values of pK_a (CH_3NO_2) and the Thermodynamic and Spectral Characteristics of the Cyclic Phosphoryl—p-Fluorophenol H-Complexes

Compound	Formula	pK_a	$\Delta\nu_{OH}$, cm^{-1}	$\lg K_{as} \pm 0{,}02$	$\Delta H \pm 0.2$, kcal/mole	$\Delta S \pm 0{,}7$, eu
(I)		<2	275	2,29	5,4	7,7
(II)		<2	300	2,50	5,6	7,4
(III)		<2	305	2,56	5,7	7,4
(IV)		<2	360	2,81	6,2	8,1
(V)		<2	365	2,83	6,4	8,5
(VI)		<2	335	2,68	6,0	7,9
(VII)		<2	360	2,82	6,2	7,9
(VIII)		2,3	400	2,98	6,7	8,9
(IX)		2,8	425	3,13	7,0	9,2
(X)		2,2	400	2,94	6,8	9,4
(XI)		2,8	425	3,10	7,0	9,3
(XII)		–	350	2,74	6,1	8,0
(XIII)		<2	365	2,91	6,3	8,0
(XIV)		(7,7)	390	2,99	6,6	8,5
(XV)		(6,1)	420	3,18	7,0	8,9
(XVI)		(5,1)	410	3,06	6,9	9,2
(XVII)		4,1	440	3,26	7,2	9,2

We have evaluated the P=O group basicity by carrying out $HClO_4$ potentiometric titration of various compounds in **CH_3NO_2 solution, following** the procedure of [16]. It was impossible to evaluate the pK_a values of the cyclic phosphates (I) and (II), and the phosphonates (III)-(VII), the basicities of these compounds being so low that the titration curves did not show a region of abrupt potential change. On the other hand, the data of [17] indicate that the acyclic phosphates and phosphonates can be titrated with $HClO_4$ in CH_3NO_2 solution, their pK_a values being ∿1.9 and ∿3.2, respectively.* We have therefore, once more tested the possibility of titrating $(C_4H_9O)_3P = O$ and $(C_2H_5O)_2P(O)CH_3$ with $HClO_4$ in CH_3NO_2. The titration curves for these compounds also showed no region of abrupt potential change. From this it could be concluded that low basicity is characteristic of the phosphates and phosphonates, the cyclic compounds being no exception in this respect.

The titration curves for compounds (VIII)-(XI) each showed a region of abrupt potential change at 50% neutralization. In view of the data on the phosphine monooxides [11, 12], this suggested that protonation in these systems proceeds through the formation of a BHB^+ type complex

$$2B + H^+ \rightleftharpoons BHB^+ + H^+ \rightleftharpoons 2BH^+ \tag{1}$$

This being the case, the pK_a of the $BH^+ \rightleftharpoons B + H^+$ reaction can be calculated from the equation [13],

$$pK_a = 0.5\ (pH_{25} + pH_{75}) \tag{2}$$

On the basis of their pK_a (CH_3NO_2) values, 2.3-2.8 (cf. Table 1), the phosphinates (VIII)-(XI) are to be classified as weak bases. For this reason the titration curves for these compounds fail to show a second region of abrupt potential change at 100% neutralization [11, 12]. The titration curve of the more basic (XVII) ($pK_a = 4.1$) showed two clearly expressed regions of abrupt potential change, at 50 and 100% neutralization. Comparison of the pK_a values of the phosphinates (VIII)-(XI) showed that the size of the ring affects the basicity of the phosphoryl oxygen, the effect being most pronounced in the six-membered phosphinates.

Compounds (XIV)-(XVI) behaved differently, their titration curves showing a single, extremely abrupt alteration in the potential at 100% neutralization. Similar titration curves have been reported for the nitrogen bases where protonation occurs in a single step [18]: $B + H^+$ BH^+. It can be assumed that it is the N atom of the amide group which undergoes protonation here.†

The OH vibration regions in the IR spectra of the H-complexes of compounds (I)-(XVII) with p-chlorophenol are similar, being characterized by an absorption band whose position is determined by the immediate environment of the P atom. From this it could be concluded that H-bond formation involves the O atom of the phosphoryl group in every case, which is what one would anticipate in view of the considerable difference in the association constants (K_{as}) for the complexes of phenol with phosphoryl [1-7] and nitrogen [19] bases.

Study of the thermodynamic (log K_{as}, ΔH) and spectral ($\Delta\nu_{OH}$) characteristics of the H-complexes ($\gtrdot P = O\ldots H\text{—}OC_6H_4F$) shows that replacement of the phosphorus oxygen by C or N unmarkedly increases the proton-acceptor activity of the phosphoryl group, the situation here being similar to that met with acyclic compounds [1]. General linear log K_{as} vs $\Delta\nu_{OH}$ and log K_{as} vs ΔH relations were developed by comparing the characteristics of the H-complexes of p-fluorophenol with compounds (I)-(XVII) **and those for** complexes of the acyclic phosphoryl compounds which we have studied earlier [1]. A least-squares treatment of these

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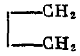
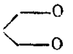
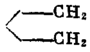
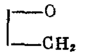
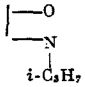
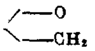
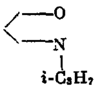
*These values are given in the nitromethane pH scale [16], the pK_a of diphenyl guanidine being 14.5.

†The problem of the point of protonation in the amides of the pentavalent phosphorus acids will be considered in more detail elsewhere.

TABLE 2. Values of the σ^P Constant for Various Cyclic Fragments

Cyclic fragment	Number of compounds	σ^P	$\Delta\sigma^P$ $(\sigma_6^P - \sigma_5^P)$	Cyclic fragment	Number of compounds	σ^P	$\Delta\sigma^P$ $(\sigma_6^P - \sigma_5^P)$
	2	+0,11 (±0,03)	0,67		1	−1,78	0,48
	2	−0,56 (±0,09)			1	−2,26	
	2	−1,01 (±0,01)	0,43		3	−1,17 (±0,04)	0,55
	2	−1,44 (±0,01)			3	−1,72 (±0,03)	

relations led to the equations

$$\lg K_{as} = 0.79 + 0.0055\Delta\nu_{OH};\quad n = 36,\quad r = 0.983,\quad S = 0.06,\quad S_\rho = 0.0002 \tag{3}$$

$$\lg K_{as} = -0.09 + 0.46\ (-\Delta H);\quad n = 36,\ r = 0.983,\quad S = 0.05, S_\rho = 0.02 \tag{4}$$

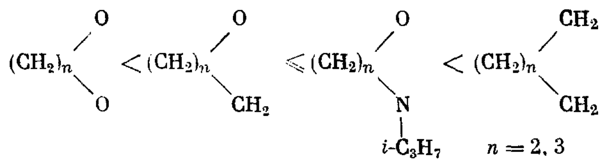
It has been pointed out [14] that the Badger–Bower ΔH vs $\Delta\nu_{OH}$ equation does not apply to the cyclic phosphoryl compounds. However, a relation of the following type does follow from Eqs. (3) and (4):

$$-\Delta H = 1.88 + 0.012\Delta\nu_{OH};\ n = 36,\ r = 0.985,\ S = 0.01, S_\rho = 0.0004 \tag{5}$$

The fact that the same linear thermodynamic parameter–spectral characteristic relations apply to the H-complexes of cyclic and acyclic phosphoryl compounds indicates that all of the complexes in question here belong to the same narrow reaction series. This permits calculation of values of the σ^P constants for the cyclic fragments, using the equation derived in [1]

$$\Sigma\sigma^P = 6.71 - 2.96 \lg K_{as};\ r = 0.989,\ S = 0.16,\ S_\rho = 0.11 \tag{6}$$

On the basis of the calculated values of the σ^P constant (Table 2) the fragments can be ordered in terms of increasing electron-donor activity



This sequence reflects the alteration of σ^P constants for common substituents at the phosphorus atom [20], the value for the six-membered ring (σ_6^P) fragment being approximately equal to the sums of the σ^P values for the two linear substituents.

One aspect of the electron effect of the ring appears on comparing the proton-acceptor activities of the phosphoryl oxygen of five- and six-membered compounds of identical composition. Considered pairwise, reduction of the size of the ring reduces the proton-acceptor activity, both in H-bond formation (log K_{as}, ΔH, $\Delta\nu_{OH}$) and in protonation of compounds (VIII)-(XI) (pK_a). In this respect the phosphoryl compounds in question here are similar to cyclic ketones, for which the basicity of the carbonyl group diminishes in passing from cyclohexanone to cyclobutanone [21].

The difference in the electronic effect of five- and six-membered phosphorus ring fragments can be measured by $\Delta\sigma^P = \sigma_6^P - \sigma_5^P$ (cf. Table 2), a quantity which changes only slightly in passing from rings containing elements with free electrons pairs to rings containing only carbon fragments. On this basis it can be seen that the alteration in proton-acceptor activity of the phosphoryl group resulting from a change in the size of the phosphorus heterocycle does not arise from differences in the $p_\pi - d_\pi$ interactions within the heterocyclic system. X-ray structural data [22] indicate that the P atom intracyclic angle is 6-8° less in the five-membered compounds than in the corresponding six-membered compounds. As a rule, the valence angles of the ring atoms linked to the phosphorus are also less. Obviously, the higher electronegativity of the five-membered ring fragments must trace back to the so-called stereoelectronic effect, i.e., to the alteration in the polarity of the ring phosphorus bonds resulting from changes in the hybridization of the individual atoms. Similar conclusions concerning the acceptor action of the five-membered rings follow from the fact that the deshielding of the phosphorus nuclei observed in the ^{31}P NMR spectra of the cyclic phosphates, phosphonates and phosphinates is much higher than that of the six-membered and acyclic compounds [23], and from the results of quantum-chemical calculations on the cyclic phosphates [24, 25]. This problem demands further detailed study.

EXPERIMENTAL

The H-bond characteristics were determined in CCl_4 solution, working at concentrations in the 0.001-0.003 mole/liter range. Measurements of K_{as} were carried out with a Hitachi EPS-3T spectrophotometer using a thermostated quartz cell, 10 cm deep [scanning time for the entire spectrum, 6 min; scale T = 90-110% (transmission coefficient); 25 ± 0.1°C]. Values of K_{as} were calculated from the formula

$$K_{as} = x/(a - x)(b - x)$$

x being the concentration of the H-complex (mole/liter) as obtained from the intensity of the free OH band at 1420 nm, and a and b being the respective formal concentrations (mole/liter) of proton donor and acceptor. The value of ΔH was obtained from the integral intensity of the H-complex OH band, following the procedure of [26]. The IR spectra were obtained with a UR-20 spectrometer, using a one-piece NaCl cell, 1 cm deep, and working by compensation of solvent and phosphoryl compound (scanning rate, 64 cm^{-1}/min). The error in the ΔH determination was ± 0.2 kcal/mole. Details of the potentiometric titration have been given in [16]. The value of pK_a (CH_3NO_2) was determined with an electrode pair calibrated against a picrate buffer, the accuracy of determination being ~0.1 pK_a unit.

The δ_{31P} chemical shifts of compounds (XI)-(XVII) were obtained from the ^{31}P NMR spectra in CCl_4 solution, using a Bruker HX-90 spectrometer operated with noise suppression of the interaction between the P and H nuclei, an 85% H_3PO_4 solution serving as the external standard.

The cyclic ethers (I)-(X) were synthesized by the methods of [27-31]; the measured values of the their physical constants agreed well with those reported in the literature. The n_D^{20} value of the phosphinate (XI), 1.4700, was considerably higher than the 1.4489 reported in the literature [32]. Because of this, this compound was also prepared by a second procedure, the product obtained being identical with that from the first in values of the IR spectral constants, and ^1H and ^{31}P NMR spectra. Both procedures for the preparation of (XI) are given below, together with general methods for synthesizing the previously undescribed 2-oxo-1,3,2-oxazaphospholanes and phosphorinanes (XII)-(XVII). As determined by GLC, each of these compounds was at least 98% pure.

1-Oxo-1-ethoxyphosphorinane (XI). (Procedure 1). Over a period of 5 h, 21.2 g, (0.1 mole) of the ethyl dichlorophosphate and a solution of the Grignard reagent, prepared by reacting 35 g (0.15 mole) of pentamethylene dibromide with 7.4 of Mg turnings, were added simultaneously to 350 ml of gently boiling, intensely agitated abs. ether. The solution remaining at the end of the reaction was broken down with a saturated K_2CO_3 solution. The organic layer was then separated from the precipitate, which was carefully extracted with five 50-ml portions of alcohol. The extracts were consolidated with the ether solution and the solvent evaporated. The resulting product was dissolved in 10 times its own volume of benzene and dried over Na_2SO_4. Evaporation followed by vacuum distillation gave 2.4 g (11%) of 1-oxo-1-ethoxyphosphorinane, bp 115°C (9 mm); n_D^{20} 1.4700; d_4^{20} 1.0743; δ_{31P} 46.2 ppm. Found: P 19.01%. $C_7H_{15}O_2P$. Calculated: P 19.10%.

Procedure 2. A solution containing 24 g (0.16 mole) of the Na salt of diethyl phosphate in 100 ml THF was added to a solution containing 57.5 g (0.25 mole) of pentamethylene dibromide in 150 ml THF, the mixture agitated for 3 h, and the solvent then evaporated off. Benzene (200 ml) was added to the residue and the mixture washed with 50 ml H_2O. The product

TABLE 3. Synthesis and Properties of 2-Oxo-1,3,2-oxazaphospholanes and Phosphorinanes

Compound	Method of synthesis	Yield, %	bp, °C (p, mm Hg)	n_D^{20}	d_4^{20}	$\delta_{^{31}P}$, ppm	Found, %	Calculated, %
(XII)	$C_2H_5OP(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_2OH$	57	86(1)	1,4500	1,1269	—17,8	15,96	16,03 ($C_7H_{16}O_3NP$)
(XIII)	$C_2H_5OP(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_3OH$	65	98(1) bp 27—28	1,4556	1,1123	—1,6	15,06	14,95 ($C_8H_{18}O_3NP$)
(XIV)	$CH_3P(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_2OH$	47	84(1)	1,4633	1,1146	—39,7	19,09	18,98 ($C_6H_{14}O_2NP$)
(XV)	$CH_3P(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_3OH$	37	92—94(1) bp 57 (ether–hexane)	—	—	—24,9	17,49	17,48 ($C_7H_{16}O_2NP$)
(XVI)	$(CH_3)_2NP(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_2OH$	48	100—102(2) bp, 37 (ether–hexane)	—	—	—23,7	16,04	16,12 ($C_7H_{17}O_2N_2P$)
(XVII)	$(CH_3)_2NP(O)Cl_2 +$ $+ i\text{-}C_3H_7\underset{H}{N}(CH_2)_3OH$	45	95(1)	1,4703	1,0915	—12,6	15,17	15,02 ($C_8H_{19}O_2N_2P$)

obtained after drying over Na_2SO_4 and evaporation of the benzene was vacuum distilled. This procedure gave 12.1 g (28%, calculated as Na diethyl phosphite) of diethyl-5-bromoamylphosphonate, bp 120-123°C (2 mm):n_D^{20} 1.4660.

To 1.1 Mg turnings, activated by I_2, in 50 ml of gently boiling abs. ether 12 g of the product diethyl-5-bromoamylphosphonate was added in the course of 1.5 h. At the end of this time, the solution was heated and stirred for another 2 h, after which 100 ml of freshly distilled anisole was added, the ether gradually evaporated off, and the temperature raised to 130-135°C. After 1.5 h, the solution was cooled and broken down with a saturated K_2CO_3 solution. The product was separated out by the procedure described in Procedure 1. This gave 2.1 g (31%) of 1-oxo-1-ethoxyphosphorinane, bp 72°C (1 mm), n_D^{20} 1.4705; d_4^{20}1.0751, $\delta_{^{31}P}$ 46.3 ppm. Found: P 19.13%.

2-Oxo-1,3,2-oxazaphospholanes (Phosphorinanes). To a solution containing 0.1 mole of N-isopropylaminoethanol (propanol) and 0.2 mole of dry triethylamine in 150 ml abs. ether there was added 0.1 mole of freshly distilled dichloride in 30 ml ether (Table 3). The resulting mixture was agitated for 30 min at 0-5°C, and the agitation then continued for another 12 h at ∿20°C. The precipitated $N(CH_3)_3 \cdot HCl$ was then separated out and the solvent evaporated off in vacuum. This procedure was repeated three more times, after which the compound was either distilled or recrystallized from ether–hexane solution (cf. Table 3). The crystalline compounds (XIII), (XV), and (XVI) were hydroscopic. Synthesis of each compound was carried out in an atmosphere of dry Ar.

CONCLUSIONS

1. A study has been made of the acid–base properties of the five- and six-membered phosphates, phosphonates, and phosphinates. Thermodynamic spectral characteristics of the H-complexes with p-fluorophenol have been measured in CCl_4 solution; using the method of potentiometric titration with $HClO_4$, the pK_a (CH_3NO_2) values have been obtained for the more basic of these compounds.

2. General linear correlation equations have been obtained for the H-complexes of p-fluorophenol with cyclic and acyclic phosphoryl compounds.

3. The σ^P values have been calculated for the phosphor ring fragments. The difference between the σ^P constants for the five- and six-membered rings remains unchanged in moving through the series of compounds studied here. Passage from a six-membered to a five-membered cycle reduced the proton-acceptor activity of the phosphoryl group in every case.

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