2-Phenyl-2-oxo-5,6-benzo-1,3,2-dioxaphosphepine (I). A solution of 3 g of $PhP(O)Cl_2$ [11] in 10 ml of abs. CH_2Cl_2 was added by drops in 50 min to a mixed solution of 2.12 g of phthalyl alcohol and 3.1 g of NEt_3 in 20 ml of abs. CH_2Cl_2 while cooling to 0°C. The precipitated $NEt_3 \cdot HCl$ salt was filtered off, and the solution was washed with water acidified with HCl and then with Na_2CO_3 solution and water. After drying with anhydrous Na_2SO_4 and evaporation of the solvent, 3.67 g (92%) of white crystals was obtained. Recrystallization from benzene separated 2.8 g (70%) of pure product with mp 133-134°C. Found, %: C 64.55, H 5.01, P 12.01. $C_{14}H_{13}O_3P$. Calculated, %: C 64.62, H 5.03, P 11.90; M 260.22.

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BASICITY AND CONFORMATIONAL ANALYSIS OF CARBAMOYLMETHYLPHOSPHORYL COMPOUNDS

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547.1'118

The basicity in nitromethane of carbamoylmethylphosphoryl compounds (CMP), $RR'P(O)CHR'''C(O)NR_2''$, containing different substituents at the phosphorus or nitrogen atoms and in the methylene bridge and binary reactants $(CMP)_2X$, where $X = (CH_2)_5$ or $p\text{-}CH_2C_6H_4CH_2$, was investigated. The values of pK_a are linearly correlated with $\Sigma\sigma P$, the constant of the substituents at the phosphorus atom. Conformational analysis of CMP and tetraphenylmethylenediphosphine dioxides substituted in the methylene bridge showed that bulky substituents reduce the population of complexing conformations.

Diphenyl(carbamoylmethyl)phosphine oxides (CMP) have attracted attention as efficient extracting agents [1, 2], which requires studying their properties and particularly their basicity. We previously established the structure of the products of protonation of CMP with perchloric acid [3]. The basicity of CMP containing different substituents R, R' at

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TABLE 1. Basicity of the CMP $RR'P(O)CH_2C(O)NEt_2$ (CH_3NO_2, 25°C)

Compound	R, R′	pK_a	$\Sigma\sigma^P$	Compound	R, R′	pK_a	$\Sigma\sigma^P$
(I)	t-Bu_2	7,0	−3,10	(IX)	(p-$CH_3OC_6H_4$)$_2$	4,5	−1,48
(II)	$(Et_2N)_2$	6,9	−3,08	(X)	p-Tol	4,1	−1,44
(III)	$(Me_2N)_2$	–*	−2,44	(XI)	Ph_2	3,9; 4,3 [3]	−1,18
(IV)	Bu_2	5,6; 5,7 [3]	−2,44	(XII)	Ph, BuO	3,1	−0,85 [4]
(V)	(c-C_6H_{11})$_2$	6,4	−2,68 †	(XIII)	$(BuO)_2$	3,1; 3,6 [3]	−0,74 [4]
(VI)	Et_2	5,1	−2,20	(XIV)	Ph, EtO	3,6	−0,69
(VII)	Ph, Bu	4,5	−1,70	(XV)	$(EtO)_2$	3,1	−0,42
(VIII)	Ph, Et	4,6; 4,7 [3]	−1,58				

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*Compound (III) is hydrolyzed in the conditions of titration.
†Based on more precise unpublished data.

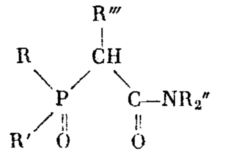
TABLE 2. Basicity of the CMP $Ph_2P(O)CH_2C(O)NR_2''$ (CH_3NO_2, 25°C)

Compound	R″	pK_a
(XVI)	Bu	4,3
(XVII)	Ph	<2,0

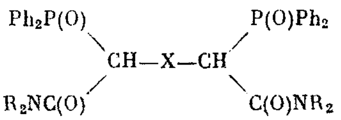
TABLE 3. Basicity of the CMP $Ph_2P(O)CHR'''C(O)NEt_2$ (CH_3NO_2, 25°C)

Compound	R‴	pK_a
(XVIII)	C_7H_{15}	4,3
(XIX)	CH_3	4,1
(XX)	Cl	3,5
(XXI)	CH_2CH_2COOEt	2,5
(XXII)	C_6H_5	2,6

the phosphorus atom (I)-(XV) (Table 1) or at the nitrogen atom R″ (XVI), (XVII) (Table 2), and in the methylene bridge R‴ (XVIII)-(XXII) (Table 3) and the binary reactants $(CMP)_2X$ was determined by potentiometric titration in nitromethane with perchloric acid



(I)–(XXII)



(XXIII)–(XXVI)

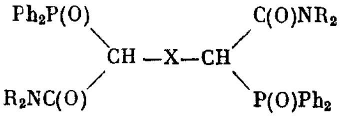
$X = (CH_2)_5$, p-CH_2—C_6H_4—CH_2; R = Et, Bu (Table 4).

Compounds (I)-(XVII) were obtained with the Arbuzov reaction from the corresponding phosphinite and perchloric acid dialkylamide [1, 5-7] substituted in the methylene bridge (XVIII)-(XXI) and the binary (XXIII)-(XXVI) reactants were obtained by alkylation of diphenyl-(diethylcarbamoylmethyl)phosphine oxide through the potassium or sodium derivatives. Diphenyl(diethylcarbamoylbenzyl)phosphine oxide (XXII) was synthesized from O-ethyldiphenylphosphinite and α-bromophenylacetic acid diethylamide. The constants, yields, and data from the analysis of the compounds obtained are reported in Table 5. The conformational composition of some of the compounds was investigated by the method of molecular mechanics.

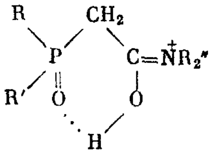
RESULTS AND DISCUSSION

The CMP molecules contain two functional groups capable of participating in protonation: phosphoryl $\left(\gt P{=}O\right)$ and carbamoyl $\left(\gt N{-}\overset{|}{C}{=}O\right)$. The CMP titration curves have one potential jump at the point of 1 equiv. $HClO_4$ in all cases, which indicates formation of products of protonation of 1:1 composition. According to the data from x-ray structural analysis, these products in the crystalline state have a structure with a protonated carbonyl group and a strong intramolecular H bond [3].

TABLE 4. Basicity of $(CMP)_2X$ $(CH_3NO_2$, 25°C)



Compound	X	R	pK_a
(XXIII)	$-(CH_2)_5-$	Et	5,3
(XXIV)	$p\text{-}CH_2C_6H_4CH_2-$	Et	5,3
(XXV)	$-(CH_2)_5-$	Bu	5,5
(XXVI)	$p\text{-}CH_2C_6H_4CH_2-$	Bu	5,7



(1)

The IR spectrum of the protonated salt in the crystalline state corresponds to the reported formula. However, in (MeCN) solution, the spectrum changes significantly and approaches the spectra of a complex of the BHB^+ type formed by phosphoryl compounds. For this reason, the structure of the protonated salt is different in solution: either the covalent and hydrogen bonds change sites – a proton is added to the phosphoryl oxygen atom and forms a hydrogen bond with the carbonyl group oxygen atom, or a structure arises which is symmetric with respect to the two oxygen atoms of the P=O and C=O group with the formation of the quasisymmetric complex $(BHB')^+$. The character of the dependence of the basicity of CMP on the nature of the substituents at the phosphorus atom is in agreement with such a change in the structure of the protonated salt in solution: the basicity of the donor substituents increases sharply, and the basicity of the acceptor substituents decreases. A good linear correlation with a relatively significant steep slope $\rho = -1.498 \pm 0.08$ is observed in Hammett coordinates of pK_a $(\Sigma\sigma^P)$.

$$pK_a = 2.16 - 1.498 \cdot \Sigma\sigma^P;\ n = 14,\ \ r = 0.983,\ s = 0.26,\ s_\rho = 0.08. \qquad (2)$$

For comparison, we indicate that the tangent of the slope of the analogous line for phosphine oxides is $\rho = -1.69 \pm 0.19$ [8]. Addition of a proton to the phosphoryl group with the formation of the $(BHB')^+$ complex thus corresponds to protonation of CMP in solution.

The effect of the substituents at the nitrogen atom is also apparently relatively strong. In any case, the compound with R" = Ph is characterized by such low basicity that no potential jump is observed on the titration curve in nitromethane.

CMP with substituents in the methylene bridge, $Ph_2P(O)CHR''C(O)NEt_2$, constituted the other group. The titration curves in nitromethane also have one potential jump at the point of one equivalent of $HClO_4$. The pK_a of this group of compounds are not correlated with either the constants of the substituents σ^* or with E_S or with any other constants from $\sigma\rho$ analysis. The effect of the substituents on the basicity is apparently somewhat complex here. It could consist of the superposition of a complex combination of polar effects and effects on the form and population of the conformations undergoing protonation.

In examining the results with respect to conformational analysis, it is necessary to note that the potential surface of chelating ligands is characterized by several minima in the general case, and a stable conformation corresponds to each one of them. Only the conformer having the optimum mutual direction of the functional groups should exhibit complexing properties, in the given case, P=O and C=O, corresponding to the edge of the coordination polyhedron of the cation. The larger the population of complexing conformations, the higher the thermodynamic stability of the complexes with all other conditions being equal.

Using the proposed approach, we conducted a conformational analysis of a series of CMP and tetraphenylmethylenediphosphine dioxides, $Ph_2P(O)CH(R'')P(O)Ph_2$, where R" = H (XXVII), $CH_2CH_2C(O)OMe$ (XXVIII), and $Ph_2P(O)CH(R'')C(O)NMe_2$, where R" = H (XXIX), $CH_2CH_2C(O)OMe$ (XXX).

TABLE 5. Yields, Physicochemical Constants, and Data from Analyses of CMP

Compound	Yield, %	Mp, °C Bp, °C (p, mm Hg)	Found, %				Empirical formula	Calculated, %				^{31}P, ppm (DMF)	IR spectrum, cm^{-1}	
			C	H	N	P		C	H	N	P		$\nu P{=}O$	$\nu C{=}O$
(III)	44,3	90(0,001) *	–	–	16,6	–	$C_{10}H_{24}N_3O_2P$	–	–	16,9	–	30,9	1205	1640
(IX)	76,0	123–124 (ether)	64,5	7,2	3,6	7,9	$C_{20}H_{26}NO_4P$	64,0	6,9	3,7	8,3	26,7	1182, 1195	1640
(XVII)	56,6	213–214 (benzene-alcohol, 4:1)	75,9	5,5	3,4	7,6	$C_{26}H_{22}NO_2P$	75,9	5,4	3,4	7,3	27,0	1189	1665
(XXI)	46,1	77–78 (benzene-petroleum ether)	66,4	7,5	3,5	7,4	$C_{23}H_{30}NO_4P$	66,7	7,2	3.4	7,5	28,7	1195	1640
(XXII)	75,0	220–221 (alcohol)	–	–	3,9	8,0	$C_{24}H_{26}NO_2P$	–	–	3,6	7,9	26,6	1190, 1215	1640
(XXIII)	44,2	198–200 (alcohol-acetone)	69,9	7,5	4,1	9,1	$C_{44}H_{52}N_2O_4P_2$	70,5	7,4	4.0	8,9	28,2	1205	1632
(XXIV)	54,7	276–278 (alcohol)	71,0	7.0	4,3	8,5	$C_{44}H_{50}N_2O_4P_2$	72,1	6,8	3,8	8,5	27,5	1210	1638
(XXVI)	59.2	170–171 (benzene-octane)	–	–	3,2	7,0	$C_{52}H_{66}N_2O_4P_2$	–	–	3,3	7,3	27,6	1210	1640

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*n_D^{20} = 1.4810.

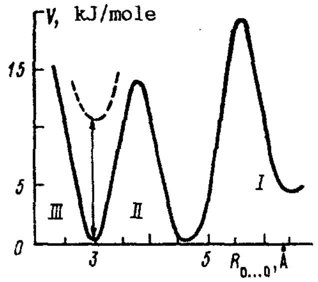


Fig. 1

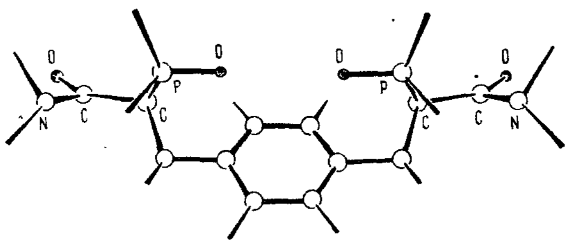


Fig. 2

Fig. 1. Dependence of the conformational energy of the ligand $Ph_2P(O)CH_2P(O)Ph_2$ on the distance between oxygen atoms ($R_{O...O}$); the increase in the energy of the complexing conformation on substitution of the H atom in the methylene bridge by the $CH_2CH_2C(O)OMe$ group is indicated by the broken line.

Fig. 2. Conformation of the binary reactant $(CMP)_2X$ with converging oxygen atoms of phosphoryl groups.

The conformational energy was calculated by the method of molecular mechanics as the sum of the energies of the nonvalence reactions (U_{nv}), the energies of the torsion potentials of rotation around ordinary bonds (U_{tors}), and the energies of angular deformations (U_{ang}). The parameters for the calculation were taken from [9, 10].

The potential surface for these ligands is essentially multidimensional (6 and 11 degrees of freedom of rotation around ordinary bonds, respectively, for ligands with a free and substituted CH_2 group). However, for chelation, the distance between oxygen atoms ($R_{O...O}$) and the corresponding changes in the conformational energy are the main characteristic of the ligand. For this reason, by using different zero-order approximations in minimization of the conformation energy with a given value of $R_{O...O}$, the multidimensional problem can be reduced to a one-dimensional problem. Penalty constraints in the form of the quasielastic law $U_{pen} = K(R_{ij} - R_{O...O})^2$ are added to the minimized function (U), where R_{ij} is the distance of the zero-order approximation and K is the force constant [11]:

$$U = U_{nv} + U_{tors} + U_{ang} + K(R_{ij} - R_{O...O})^2. \tag{3}$$

In this case, the calculated results are presented in the form of the function $U(R_{O...O})$. Not only the position of the local and global minima but also their energy are accurately reproduced.

For dioxide (XXVII), the dependence of the conformational energy on the distance between oxygen atoms (Fig. 1) is plotted with the data from the previously obtained conformation map [12]. There are three minima on the curve corresponding to stable conformers with values of $R_{O...O}$ = 6.5, 4.6, and 3.0 Å, and only minimum III corresponds to the complexing conformation with the required direction of the P=O groups.

The results of calculation of molecule (XXVIII) show that the effective volumes of the groups at the bridge carbon atom decrease the population primarily of the complexing conformation; its relative minimum is 10 kJ/mole higher than the global minimum (indicated by the broken line in Fig. 1). Similar results were obtained in calculating molecules (XXIX) and (XXX). Stable conformations are observed here for $R_{O...O}$ = 5.5, 4.5, and 3.0 Å, and the level of the complexing conformation increases by 6 kJ/mole.

The binary reactants $(CMP)_2X$ contain two fragments of CMP molecules which can each be protonated separately or simultaneously, producing two or one potential jump at the point of two neutralization equivalents on the titration curves; actually, the titration curves of these compounds in nitromethane have one potential jump at the point of one equivalent, and the pK_a are 1.4-1.2 log units higher than the basicity of the starting molecules (Table 4).

The conformational energy of the free molecule is calculated for the analysis of the observed effects

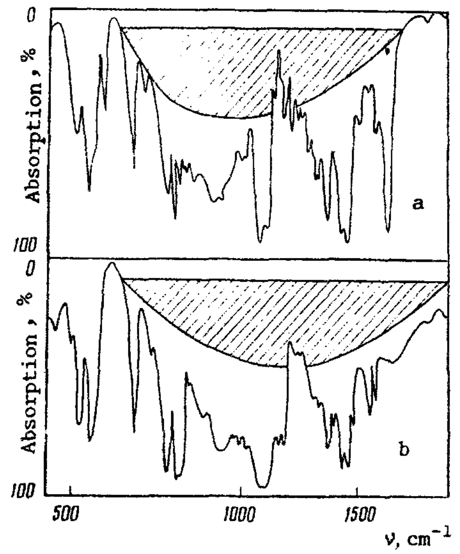
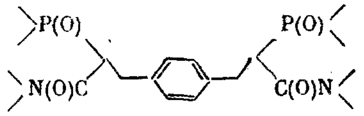


Fig. 3. IR spectra of the products of protonation: a) (XXVI)·$HClO_4$, b) $Ph_2P(O)CH_2CH_2P(O)Ph_2$·$HClO_4$ (petrolatum).



(arbitrarily set at 0) and its protonated forms, where the reaction of the proton of the type $\overline{\llcorner P{=}O\ldots H^{+}\ldots O{=}P \lrcorner}$ (form A) and $\overline{\llcorner C{=}O\ldots H^{+}\ldots O{=}P \lrcorner}$ (form B) can take place. The structure in which the chelating proton is positioned above the plane of the benzene ring is stable for both forms (Fig. 2). The molecular structure for each of these forms was optimized for the two defined distances between the oxygen atoms: 2.8 and 2.5 Å. The calculation showed that form (A) with corresponding values of 2 and 6 kJ/mole has the lowest stress energy; the stress energy of form (B) is 4 and 9.5 kJ/mole, respectively.

Forms (A) and (B) presuppose a different mechanism of protonation. The protonated salt of the binary reactant (XXVI) with $HClO_4$ (1:1) was prepared and its IR spectrum was investigated to elucidate this question (Fig. 3). Note the presence of a low-frequency continuum of OH absorption (hatched region of the spectrum) and the intense band in the 900 cm^{-1} region characteristic of complexes of the BHB^{+} $(\gt P{=}O..H..O{=}P\lt^{+})$ type of phosphoryl compounds [13]. For comparison, the spectrum of protonated tetraphenylethylenediphosphine dioxide characteristic of phosphoryl compound BHB^{+} complexes is shown in Fig. 3.

These IR spectra show that the mechanism of protonation changes in protonation of the binary reactants $(CMP)_2X$ in comparison to protonation of the starting CMP. This is also apparently the cause of the elevated basicity of the binary reactants.

On the whole, the results of the analysis show that the basicity of carbamoylmethylphosphoryl compounds and the related alkylenediphosphine dioxides is not only a function of the electronic effects of the substituents but also the conformational composition of the compounds.

EXPERIMENTAL

The CMP studied here were synthesized by the well-known methods. Compounds (I), (II), (VI), (X), (XIV), and (XV) were synthesized and purified as described in [5], compounds (IV), (V), (VIII), (XI)-(XIII), and (XVI) were synthesized as described in [1], compound (VII) was synthesized as in [6], and compounds (XIX), (XX), and (XXV) were synthesized as described in [7].

Bis(N,N-dimethylamido)(N',N'-diethylcarbamoylmethyl)phosphonate (III). Here 11.0 g (0.067 mole) of O-ethyldi(N,N-dimethylamido)phosphinite was added to a solution of 10.1 g (0.067 mole) of N,N-diethylchloroacetamide in 20 ml of abs. xylene at 140-180°C (bath temperature). Then 7.4 g (44.3%) of (III) was obtained.

Di(p-anisyl)(diethylcarbamoylmethyl)phosphine Oxide (IX). From 2.8 g (0.02 mole) of N,N-diethylchloroacetamide and 5.8 g (0.02 mole) of O-ethyldi-(p-anisyl)phosphinite, 5.4 g (76%) of (IX) was obtained at 160°C.

Diphenyl(diphenylcarboylmethyl)phosphine Oxide (XVII). Here 4.5 g (0.02 mole) of Ph_2POEt was added to a melt of 5 g (0.02 mole) of N,N-diphenylchloroacetamide at 130-140°C for 2 h. The reaction mixture was held for 0.5 h at 160°C. The mixture crystallized on cooling. Then 6.5 g (56.6%) of (XVII) was obtained.

4-Diphenylphosphinyl-4-diethylcarbamoylbutyric Acid Ethyl Ester (XXI). A mixture of 2 g (0.06 mole) of diphenyl(diethylcarbamoylmethyl)phosphine oxide and 3 g (excess) of ethyl acrylate in 15 ml of benzene in the presence of catalytic amounts of metallic sodium was heated in an ampul for 8 h at 80°C. This produced 1.2 g (46.1%) of (XXI).

α-(Diphenylphosphinyl)phenylacetic Acid N,N-Dimethylamide (XXII). Here 8.6 g (75%) of (XXII) was obtained from 9 g (0.03 mole) of α-bromophenylacetic acid N,N-diethylamide and 7.7 g (0.03 mole) of O-ethyldiphenylphosphinite at 140-160°C.

Tetraphenyl-1,7-di(ethylcarbamoyl)heptamethylenediphosphine Dioxide (XXIII). Here 3.0 g (44.2%) of (XXVII) was obtained from 1 g (0.026 g-atom) of metallic potassium, 7.8 g (0.025 mole) of (XI), and 2.99 g (0.013 mole) of 1,5-dibromopentane in xylene at 140°C.

1,4-Xylenyl-bis-(N,N-diethyl(diphenylphosphinyl)acetamide) (XXIV). At 140°C, 3.3 g (54.7%) of (XXVIII) was obtained from 0.6 g (0.015 g-atom) of metallic potassium, 4.8 g (0.015 mole) of (XI), and 2.2 g (0.008 mole) of p-xylylene dibromide in xylene at 140°C.

1,4-Xylenyl-bis-[N,N-dibutyl(diphenylphosphinyl)acetamide] (XXVI). Here 6.9 g (59.2%) of (XXX) was obtained from 1.1 g (0.028 g-atom) of potassium, 10.5 g (0.028 mole) of (XVI), and 3.7 g (0.014 mole) of p-xylylene dibromide in xylene at 140°C.

Protonated Salt (XXVI)·$HClO_4$. A solution of 0.2 g ($2.4 \cdot 10^{-4}$ mole) of base (XXVI) in nitromethane was mixed with 0.0343 g ($2.4 \cdot 10^{-4}$ mole) of $HClO_4 \cdot 2H_2O$. The prepared solution was evaporated. The crystalline residue was filtered off, repeatedly washed with ether, and dried. Then 0.2109 g (90%) of (XXVI)·$HClO_4$ was obtained, mp 218-220°C. Found, %: N 3.4, P 6.1. $C_{52}H_{67}N_2O_8P_2Cl$. Calculated, %: N 2.9, P 6.5.

The basicity of CMP in nitromethane was determined by the method in [14] using a Radelkis OP-211/1 pH-meter with a glass electrode (Radelkis) and a Calomel electrode with contact through a porous ceramic (Radiometer K-401).

The ^{31}P NMR spectra were made on a Bruker HX-90 instrument (85% H_3PO_4 as the external standard). The IR spectra were made on a UR-20 (thin layer or pellets with KBr).

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