

Once again the evidence indicates that the sulfonyl group is not capable of acting as a partner in conjugation.<sup>22</sup>

(22) Cf., however, spectra of *o*-HOC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>C<sub>6</sub>H<sub>5</sub> and *o*-HOC<sub>6</sub>H<sub>4</sub>SOC<sub>6</sub>H<sub>5</sub>, E. D. Amstutz, I. M. Hunsberger, and J. J. Chessik, *J. Am. Chem. Soc.* **73**, 1220 (1951).

*Acknowledgment.* The authors gratefully acknowledge financial support furnished for this work by the United States Atomic Energy Commission through Contract AT(30-1)-907. The preparation of the sulfones was supported by Callery Chemical Company under a contract from the Bureau of Aeronautics, Department of the Navy.

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[CONTRIBUTION FROM THE ST. LOUIS RESEARCH DEPARTMENT, ORGANIC CHEMICALS DIVISION, MONSANTO CHEMICAL CO.]

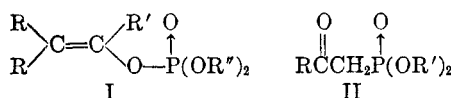
## Reactions of Phosphorus Compounds. I. Diethyl Carbamoylmethylphosphonates

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Received June 9, 1958

2-Chloroacetamides were found to undergo the Arbuzov reaction with triethyl phosphite to give diethyl carbamoylmethylphosphonates. The infrared and nuclear magnetic resonance spectra of these compounds were compared with those of known phosphonates and vinyl phosphates derived from halo ketones and esters.

The reaction of triethyl phosphites with 2-halocarbonyl compounds has been shown to give vinyl phosphates (I) and/or phosphonates (II).

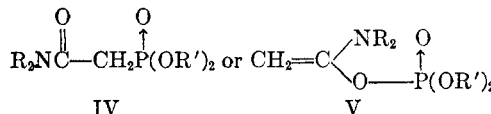
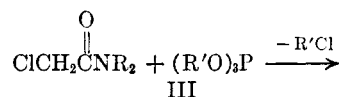


Mono-, di-, and trihaloaldehydes, di- and trihalo ketones, and trihaloesters gave the phosphate derivatives.<sup>1-7</sup> Monohaloesters<sup>8</sup> afforded the phosphonate compounds whereas monohalo ketones gave phosphonates or phosphates depending on the halogen atom. Thus, chloroacetone<sup>2b,6,7,8b,9</sup> gave predominantly the vinyl phosphate, bromoacetone<sup>7b,8b,9-11</sup> gave a mixture of phosphate and

phosphonate, and iodoacetone<sup>6,7b</sup> gave predominantly the phosphonate. 2-Haloacetoacetic esters and amides and 3-haloacetylacetone yielded phosphates.<sup>2b,7b,12</sup>

It was of interest, therefore, to determine whether the reaction of 2-haloamides with trialkyl phosphites would give phosphonates (IV) (normal Arbuzov<sup>13</sup> product) or phosphates (V) (Perkow rearrangement<sup>5</sup>).

In these laboratories, phosphonates (IV) rather than phosphates (V) were isolated from the reaction of trialkyl phosphites with 2-chloroacetamides.



- a R = R' = C<sub>2</sub>H<sub>5</sub>  
b R = CH<sub>2</sub>CH = CH<sub>2</sub>; R' = C<sub>2</sub>H<sub>5</sub>  
c R = *n*-C<sub>3</sub>H<sub>7</sub>; R' = C<sub>2</sub>H<sub>5</sub>

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(2) (a) J. F. Allen, S. K. Reed, O. H. Johnson, and N. J. Brunsvold, *J. Am. Chem. Soc.*, **78**, 3715(1956); (b) J. F. Allen and O. H. Johnson, *J. Am. Chem. Soc.*, **77**, 2871(1955).

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2-Chloro-*N,N*-diethylacetamide was allowed to react with triethyl phosphite (III, R' = ethyl) at 140°, and at which temperature ethyl chloride was vigorously evolved. Diethyl diethylcarbamoylmethylphosphonate (IVa, R = R' = ethyl) was isolated in a 78% yield. The infrared spectrum of IVa showed the following characteristics: C—O—P, 9.9μ; P—O—C<sub>2</sub>H<sub>5</sub>, 8.65μ; P→O, 8.05μ and (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>-

$\begin{array}{c} \text{O} \\ || \\ \text{N}-\text{C}- \end{array}$ , 6.13μ. The intensity of the maximum at 6.13μ was typical for the amide carbonyl (absorbance, 1.44) and, hence, excluded the possibility of that band's representing the carbon-carbon double bond absorption<sup>2b</sup> (absorbance for Ia, R = H, R' = CH<sub>3</sub>, R'' = C<sub>2</sub>H<sub>5</sub>, and Ib, R = Cl, R' = OC<sub>2</sub>H<sub>5</sub>, R'' = C<sub>2</sub>H<sub>5</sub>, 0.33 and 0.43, respectively). The absence of the carbon-carbon double bond was also shown by bromine and iodine number determinations and by the failure of IVa to be hydrogenated at low pressures. However, diethyl diallylcarbamoylmethylphosphonate (IVb R = allyl; R' = ethyl) was easily hydrogenated to diethyl di-*n*-propylcarbamoylmethylphosphonate (IVc, R = *n*-propyl, R' = ethyl). Two molar equivalents of hydrogen were absorbed rapidly by IVb and, therefore, only two unsaturated linkages were present.

The infrared spectra of IVa and IVc differed significantly from that of IVb only at 10.11μ and 10.87μ which were due to the absorption of the —CH=CH<sub>2</sub> groupings of the allyl radicals.

The reaction of several 2-chlorocarbonyl compounds with triethyl phosphite was also investigated. Chloroacetone afforded a 75% yield of Ia (R = H, R' = CH<sub>3</sub>, R'' = C<sub>2</sub>H<sub>5</sub>) while bromoacetone gave a 21% yield of Ia and a 56% yield of IIa (R = CH<sub>3</sub>, R' = C<sub>2</sub>H<sub>5</sub>).

Ethyl 2-chloroacetate under similar reaction conditions yielded only the phosphonate (IIb, R = OC<sub>2</sub>H<sub>5</sub>, R' = C<sub>2</sub>H<sub>5</sub>) while ethyl 2,2,2-trichloroacetate gave only the vinyl phosphate (Ib, R = Cl, R' = OC<sub>2</sub>H<sub>5</sub>, R'' = C<sub>2</sub>H<sub>5</sub>). These results complement those of several investigators.<sup>2,6-10</sup>

Absorption bands were observed for phosphonate and carbonyl groups in the infrared spectra of IVa, b, c, IIa and b. Only the carbon-carbon double bond frequency was observed at 6.02μ and 6.0μ for Ia and Ib, respectively. Added evidence for the conclusion that phosphonates were obtained from the reaction of phosphite esters with 2-haloacetamides was obtained from a study of the nuclear magnetic resonance spectra of the compounds listed in Table I.

The chemical shifts observed for IVa and IVc were in a negative direction (−21.3 and −22 p.p.m., respectively) relative to orthophosphoric acid and a positive direction relative to dibutyl butylphosphonate (VI, −30.8 p.p.m.). Based on

TABLE I  
NUCLEAR MAGNETIC RESONANCE SPECTRA OF PHOSPHONATES AND PHOSPHATES

Compounds	Chemical Shifts, <sup>a</sup> P.P.M.
VI (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)C <sub>4</sub> H <sub>9</sub>	−30.8
IIb (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)CH <sub>2</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>5</sub>	−19.5
IIa (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)CH <sub>2</sub> C(=O)CH <sub>3</sub>	−19.9
IVa (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)CH <sub>2</sub> CON(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub>	−21.3
IVc (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)CH <sub>2</sub> CON(C <sub>3</sub> H <sub>7</sub> ) <sub>2</sub>	−22.0
Ia (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)OCH=CH <sub>2</sub>	+7.2
Ib (C <sub>2</sub> H <sub>5</sub> O) <sub>2</sub> P(=O)OCH=CCl <sub>2</sub>	+7.5
(C <sub>2</sub> H <sub>5</sub> O) <sub>3</sub> P→O	+0.9

<sup>a</sup> The radio frequency was 16.2 mc. Chemical shifts relative to 85% orthophosphoric acid.

data recently published,<sup>14</sup> the carbamoylmethylphosphonates (due to the electron withdrawing effect

$\begin{array}{c} \text{O} \\ || \\ \text{R}_2\text{N}-\text{C} \end{array}$  group) were expected to undergo a chemical shift in a more positive direction relative to that of VI and were expected to lie near that of the known phosphonates (IIa, −19.9 p.p.m. and IIb −19.5 p.p.m.). If the reaction products under discussion were phosphate esters of type V, the chemical shift should have occurred at a positive position relative to phosphoric acid. The chemical shifts for the known vinyl phosphates (Ia and Ib) were found to be +7.2 and +7.5 p.p.m., respectively. Since the chemical shifts of IVa and IVc were found to lie closer to those of IIa, IIb, and VI than to those of Ia and Ib, it was concluded that IVa and IVc were phosphonate derivatives.

On the basis of the data presented here, 2-chloroacetamides undergo the normal Arbuzov reaction.

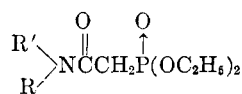
In Table II are listed the various types of phosphonates which were prepared in this investigation.

#### EXPERIMENTAL<sup>15</sup>

*Diethyl diethylcarbamoylmethylphosphonate* (IVa). Triethyl phosphite (67 g., 0.41 mole) was placed in a flask heated to 100° and 60 g. (0.4 mole) of 2-chloro-*N,N*-diethylacetamide was added dropwise. After about 5 ml. had been

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(15) The boiling points and melting points reported in this work are uncorrected. All spectra were taken in 2% w./w. chloroform solution in a 0.2-mm. NaCl cell using a Perkin-Elmer Model 21 instrument.

TABLE II  
 DIETHYL CARBAMOYLMETHYLPHOSPHONATES


R	R'	Yield, %	B.P. °C./Mm. Hg (M.P., °C.)	$n_D^{25}$	Formula	Analysis, %		Bromine Number <sup>a</sup>		
						Calcd.	Found	(Iodine number) Calcd.	Found	
Ethyl	Ethyl	77.5	135/1.2	1.4560	C <sub>10</sub> H <sub>22</sub> NO <sub>4</sub> P	N	5.58	5.55	64	0.12
						P	12.00	12.33	(101)	(2.5)
Allyl	Allyl	78.0	147/1.3	1.4718	C <sub>12</sub> H <sub>22</sub> NO <sub>4</sub> P	N	5.09	5.02	172	158
						P	11.25	11.23	(275)	(166)
Propyl <sup>b</sup>	Propyl	88.5	143/0.8	1.4550	C <sub>12</sub> H <sub>26</sub> NO <sub>4</sub> P	N	5.02	4.85	57	0.18
						H	70	(80-82) <sup>c,d</sup>	...	C <sub>6</sub> H <sub>14</sub> NO <sub>4</sub> P
H	<i>n</i> -Amyl	80	158/1.2	1.4550	C <sub>11</sub> H <sub>24</sub> NO <sub>4</sub> P	H	7.23	7.47		
						N	7.16	7.07		
						P	15.87	16.03		
						C	49.80	49.29	(104)	(4.4)
						H	9.12	8.84		
H	Cyclohexyl	85	(88-89) <sup>e</sup>	...	C <sub>12</sub> H <sub>24</sub> NO <sub>4</sub> P	N	5.28	5.33		
						P	11.67	11.54		
						C	51.97	52.10	57	0
						H	8.72	8.61		
						N	5.05	4.97		
H	Pentamethylene	64	156/2.0	1.4802	C <sub>11</sub> H <sub>22</sub> NO <sub>4</sub> P	H	8.42	8.63	(97)	(0)
						N	5.32	5.52		
						C	50.18	50.52		
						H	8.42	8.63		
						N	5.32	5.52		
H	3-Oxapentamethylene	72	167-170/1.4	1.4806	C <sub>10</sub> H <sub>20</sub> NO <sub>5</sub> P	C	45.28	45.31	(95)	(0)
						Hl	7.60	7.63		
						N	5.27	5.26		
						C	54.73	53.56		
						H	7.07	6.90	(89)	(9.2)
CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	84	160/0.6	1.5098	C <sub>13</sub> H <sub>20</sub> NO <sub>4</sub> P	N	4.91	4.87		
						C	62.24	62.04	(74)	(5.0)
						H	6.38	6.45		
C <sub>6</sub> H <sub>5</sub> <sup>e</sup>	C <sub>6</sub> H <sub>5</sub>	85	(67-68) <sup>d,f</sup>	...	C <sub>15</sub> H <sub>22</sub> NO <sub>4</sub> P	N	4.03	3.93		

<sup>a</sup> Theoretical bromine and iodine numbers for the phosphonates are zero except for IVb which are: Bromine No. 116 and iodine No. 182. The calculated values given here are those for the vinyl phosphates. The observed values for Bromine and Iodine numbers for the phosphonates are due the active methylene carbon. <sup>b</sup> Prepared by PtO<sub>2</sub> reduction of IVb. All other compounds were prepared according to the method described for IVa or IVb. <sup>c</sup> P. Nylen, *Ber.*, **57**, 1023(1924) reported m.p. 78-80°. <sup>d</sup> Recrystallized from petroleum ether (60-70°)-ethyl acetate mixture. <sup>e</sup> Recrystallized from petroleum ether (60-70°). <sup>f</sup> A. I. Razumov and N. P. Kashurina (*Chem. Abstr.*, **35**, 2474(1941)) reported m.p. 67-68°.

added (15 min.), a rapid evolution of ethyl chloride began at 135°. Addition was continued at 135-140°, and completed in 55 min. The solution was then heated at 140-152° for 75 min. The product was distilled. A low boiling fraction [40-110° (0.6 mm.); wt. 16.1 g.] was collected and discarded. The main fraction distilled at 135° (1.2 mm.). A 77.5% yield of IVa (b.p. 106°/0.17 mm.,  $n_D^{25}$  1.4560) was obtained when this experiment was repeated. The infrared absorption spectrum was consistent in all respects (see Discussion) for a compound with the structure of IVa.

*Attempted reduction of IVa.* Ten grams of IVa were subjected to reduction at 60 p.s.i. with 0.5 g. of platinum dioxide in 100 ml. of absolute ethanol at room temperature. No hydrogen was consumed in 2 hr. The catalyst was filtered and 0.3 g. of fresh platinum dioxide was added to the filtrate. No reaction occurred under the same conditions in 2 hr. The catalyst was filtered, solvent was removed, and 9.1 g. of unchanged phosphonate (IVa) was recovered after distillation at 134° (1.0 mm.),  $n_D^{25}$  1.4556. Infrared absorption spectrum for IVa before and after attempted reduction was identical in all respects.

*Diethyl diallylcarbamoylmethylphosphonate (IVb).* To 33 g. (0.20 mole) of triethyl phosphite at 140°, 34.6 g. (0.20 mole) of *N,N*-diallyl-2-chloroacetamide was added over 1 hr. Ethyl chloride was evolved during the addition and heating was continued at 140-160° for another hour. The product

was then distilled; b.p. 147° (1.3 mm.). Infrared absorption spectrum was almost identical with that of IVa except for absorption at 10.11 $\mu$  and 10.87 $\mu$  which were due to the -CH=CH<sub>2</sub> groupings of the allyl radicals. The amide group absorbed at 6.11 $\mu$  (absorbancy, 1.00).

*Diethyl dipropylcarbamoylmethylphosphonate (IVc).* Ten grams (0.036 mole) of IVb were reduced at 60 p.s.i. with 0.5 g. of platinum dioxide in 100 ml. of absolute ethanol at room temperature. Two mole equivalents of hydrogen corresponding to the saturation of the allyl groups were absorbed in 2 min. The catalyst was filtered, the solvent evaporated and the product was distilled; b.p. 143° (0.8 mm.).

*Diethyl 1-methylvinyl phosphate<sup>2b</sup> (Ia)* was prepared by the procedure of Allen and Johnson<sup>2b</sup> from triethyl phosphite and chloroacetone in 74.5% yield; b.p. 65-66° (1.5 mm.),  $n_D^{25}$  1.4178. The infrared spectrum showed the vinyl group absorption at 6.02 $\mu$ ; no carbonyl function was evident. The intensity of the vinyl group absorption (absorbancy 0.33) was significantly different from that of the intensity of the disubstituted amide group in IVa (absorbancy 1.44).

*Diethyl acetylphosphonate (IIa)* was prepared according to the procedure of Kreutzkamp and Kayser<sup>10</sup> from triethyl phosphite and bromoacetone. There was isolated 21.4% of Ia ( $n_D^{25}$  1.4165) and 56% of IIa; b.p. 81° (0.45 mm.),  $n_D^{25}$  1.4340. The infrared spectrum of IIa showed absorption at 5.85 $\mu$  for the carbonyl group, none in the 6.1 $\mu$  region

and was quite similar to the phosphonate product obtained in the chloroacetamide reactions.

*Diethyl ethoxycarbonylmethylphosphonate*<sup>8a,c</sup> (IIb) was prepared from ethyl chloroacetate according to the same procedure for Ia. The phosphonate was obtained in 76% yield; b.p. 103° (1.2 mm.),  $n_D^{25}$  1.4299. The infrared spectrum was almost identical with the phosphonate (IVa) except for the different absorptions of the ester (5.78 $\mu$ ) and amide (6.13 $\mu$ ) groups.

*Diethyl 1-ethoxy-2,2-dichlorovinyl phosphate*<sup>2b,4</sup> (Ib) was prepared in 57% yield from ethyl trichloroacetate according to the procedure of Allen and Johnson<sup>2b</sup>; b.p. 106° (0.7 mm.);  $n_D^{25}$  1.4495. About 35% of unreacted ester was recovered. The double bond absorbed at 6.0 $\mu$  with an absorbancy of 0.43.

*Diethyl carbamoylmethylphosphonate*. This is a general preparation of carbamoylmethylphosphonates when solid amides are used as starting materials. A slurry of 19.1 g.

(0.205 mole) of 2-chloroacetamide, 34 g. (0.205 mole) of triethyl phosphite, and 55 ml. of *o*-xylene was heated (132–143°) until the evolution of ethyl chloride was rapid. The rate of evolution of ethyl chloride was slower after about 1.25 hr., but the reaction mixture was kept at 143° for 2 hr. longer. The solvent was distilled *in vacuo* and the residue was recrystallized from petroleum ether and ethyl acetate mixture.

*Acknowledgments.* We are indebted to Dr. B. Katlafsky and Mr. O. Kinast for the infrared spectra, to Drs. C. F. Callis and D. P. Ames of our Inorganic Division for the NMR data and to Messrs. J. L. O'Sullivan and O. S. Kring for the analytical data.

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[CONTRIBUTION FROM THE LABORATORY OF ORGANIC CHEMISTRY, INSTITUTE OF TECHNOLOGY POLITECHNIKA AND INSTITUTE OF ORGANIC SYNTHESIS POLISH ACADEMY OF SCIENCES]

## Synthesis of 2-(Pyridyl)ethylphosphonic Acids and Esters<sup>1</sup>

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Received July 10, 1958

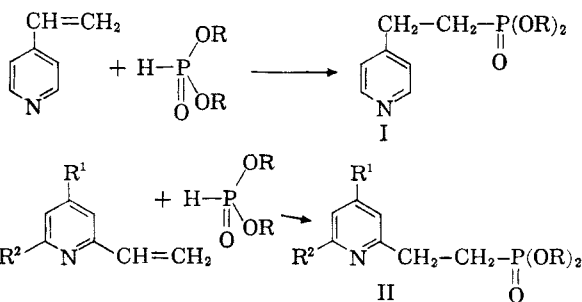
Esters of 2-(pyridyl)ethylphosphonic acids are obtained in good yield by adding dialkyl phosphites to  $\alpha$ - and  $\gamma$ -vinylpyridines. The esters are readily hydrolyzed to the corresponding phosphonic acids.

Among phosphonic acids and their esters few compounds are known in which the phosphono or dialkylphosphono group is attached directly to the heterocyclic ring or its side chain.<sup>2</sup> In the field of pyridine the only compounds of this type are: 2-dimethylamino-5-pyridylphosphonic acid described by Plažek and Sasyk,<sup>3</sup> 3-pyridylphosphonic acid described recently by Bennett, Burger, and Volk,<sup>4</sup> pyridylhydroxymethylphosphonic acids reported by Burger, Clements, Dawson, and Henderson,<sup>5</sup> and 2-pyridylmethylphosphonic acids and their esters obtained in this laboratory.<sup>6</sup>

In undertaking the synthesis of still unknown esters of 2-(pyridyl)ethylphosphonic acids (I and II) it might have been expected that the preparation of this kind of compound by the Arbuzov or Michaelis-Nylen methods would entail difficulties due to the properties of the starting  $\beta$ -haloalkylpyridines. The latter, endowed with a tautomeric electron-withdrawing group, would likely be susceptible to dehydrohalogenation by attack of

basic reagents, such as sodium dialkyl phosphites or trialkyl phosphites. This type of difficulty was noted by Arbuzov and Lugowkin in the course of the attempted synthesis of diethyl 2-(2'-pyridyl)ethylphosphonate by the Michaelis-Nylen procedure.<sup>7</sup>

In considering other methods possibly useful in synthesizing compounds I and II we focused our attention on pyridine derivatives possessing vinyl groups in position  $\alpha$  or  $\gamma$ . Such vinylpyridines behave like typical  $\alpha,\beta$ -unsaturated compounds and add nucleophilic reagents.<sup>8</sup> In view of these facts we expected to synthesize esters I and II by adding dialkyl phosphites, as nucleophilic reagents, to  $\alpha$  and  $\gamma$ -vinylpyridines. We found indeed that dialkyl phosphites add to 2 and 4-vinylpyridines as well as to 2-methyl-6-vinylpyridine and 2,4-



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