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TETRAHEDRON

Derivatives of α-Phosphorylated Aldehydes

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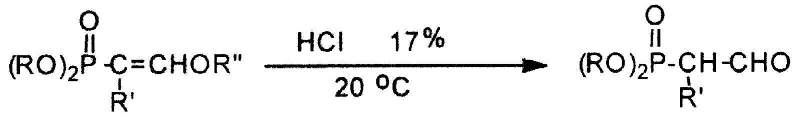
Abstract : Conditions for the selective chlorination of α-phosphorylated aldehydes as a means of synthesising α-monochloro- and α,α-dichlorosubstituted derivatives are described. Dichloro derivatives show high reactivity and easily add thiols, amides and ethyleneimine to give stable hemi-thioacetals, hemiamidals and hemiaminal. From the silyl ether of hemiisopropyl thioacetal above 140^{0}C , an α-ketophosphonate is obtained by the elimination of silane followed by the rearrangement of the oxirane intermediate. Alkylations of α-phosphorylated aldehydes with alkyl bromides gave enol ethers. However, dihalogenoalkanes such as 1,2-dibromoethane or 1,3-dibromopropane yielded phosphatecyclanes along with enol ethers, all in trans configuration. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords : Phosphonous aldehydes and derivatives, Phosphorus heterocycles, Alkylation

Although α- phosphorylated aldehydes were described comparitively long ago,[1] their chemical properties were not fully investigated. From a structural point of view they are the phosphorus analogues of α-formylcarbonyl compounds (α-formylketones and α-formylesters) and therefore, α-phosphorylated aldehydes can find their applications in organic synthesis for the preparation of polyfunctional heterocyclic compounds bearing phosphorus either in a ring or in lateral chains, which can act as complexing agents, analytical reagents etc.

α-Dialkoxyphosphoroylaldehydes were synthesized by hydrolysis of the corresponding enol ethers[2,3] and contrary to α- formylcarbonyl compounds their phosphorus analogues were stable.



$$(RO)_2\overset{O}{\overset{\|}{P}}-\underset{R'}{\underset{|}{C}}=CHOR'' \xrightarrow[20\ ^{o}C]{HCl\ \ 17\%} (RO)_2\overset{O}{\overset{\|}{P}}-\underset{R'}{\underset{|}{C}}H-CHO$$

It should be noted that α-phosphorylated aldehydes are coloured which is different to α-formylcarbonyls and their colors darken in alkaline media containing amines, alkali metals and their hydroxides.

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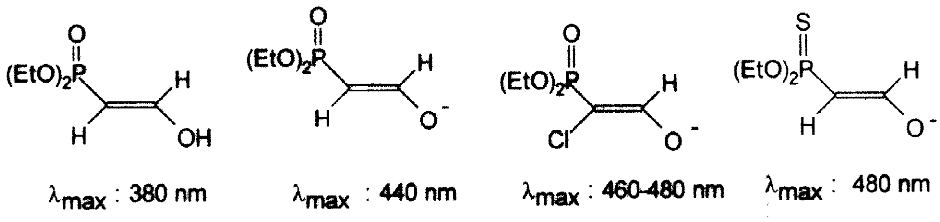
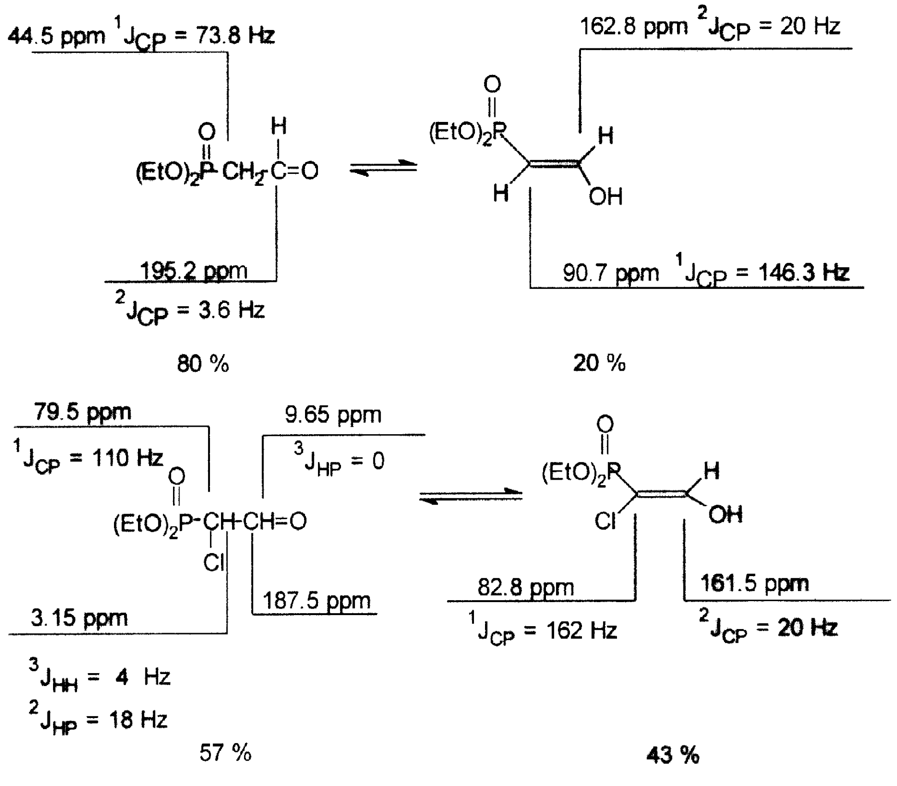
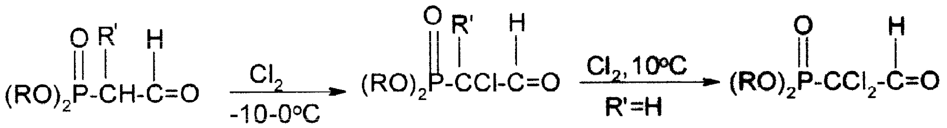


These properties of the soluble phosphorylated aldehydes are dependent on the conjugation of the enol form and on the nature of the phosphorus heteroatom.

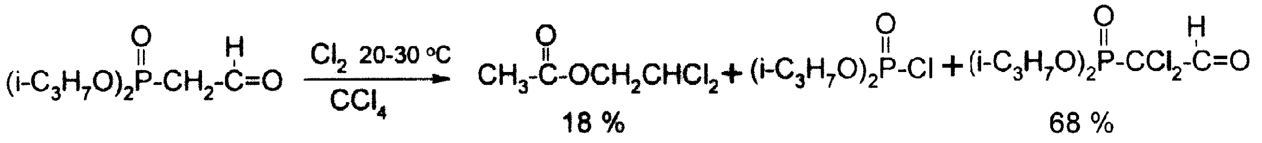
The data obtained during the investigation of the aldo-enol tautomerism of such aldehydes by ^{1}H, ^{13}C and ^{31}P-NMR spectroscopic methods confirmed the trans-configuration of their enol forms. The existence of $^{3}J_{HH}$=14 Hz indicated the trans configuration of the enol isomer.



Previously we have described preparative methods for the selective chlorination of α-phosphorylated aldehydes which yielded monochloro- and dichlorosubstituted derivatives.[4]



During the chlorination of phosphonacetaldehyde, increasing the reaction temperature caused the cleavage of the P-C bond and consequently the formation of β,β-dichloroethyl acetate and chlorophosphates was observed. The formation of such compounds can be considered as the condensation of acetaldehydes and dichloroacetaldehydes appearing from the decomposition of the initial nonchlorinated and chlorinated phosphonacetaldehydes during the reaction.

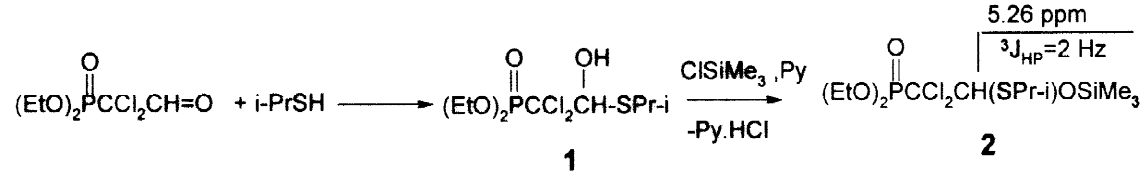


$$(i\text{-}C_3H_7O)_2\overset{O}{\overset{\|}{P}}\text{-}CH_2\text{-}\overset{H}{C}{=}O \xrightarrow[CCl_4]{Cl_2\ 20\text{-}30\,^{\circ}C} \underset{18\ \%}{CH_3\text{-}\overset{O}{\overset{\|}{C}}\text{-}OCH_2CHCl_2} + (i\text{-}C_3H_7O)_2\overset{O}{\overset{\|}{P}}\text{-}Cl + \underset{68\ \%}{(i\text{-}C_3H_7O)_2\overset{O}{\overset{\|}{P}}\text{-}CCl_2\text{-}\overset{H}{C}{=}O}$$

The formation of β,β-dichloroethyl acetate was confirmed by ^{1}H and ^{13}C-NMR (δ ppm, J Hz) : ^{1}**H** : 2.05 (s,CH_3) ; 3.85 (d,CH_2, $^3J_{HH}$= 6.0) ; 6.43 (t,CH).
^{13}**C** : 28.8 (CH_3) ; 46.9 (CH_2) ; 81.9 (CH) ; 178.8 (C=O).

The introduction of a chlorine atom into the α-position of phosphorylated aldehydes increases the electrophilic capacity of the carbonyl group which results in relatively easier additions of nucleophilic reagents such as alcohols,[3] thiols, amides and ethyleneimine with the formation of accordingly stable hemiacetals, hemithioacetals, hemiamidals and hemiaminals.

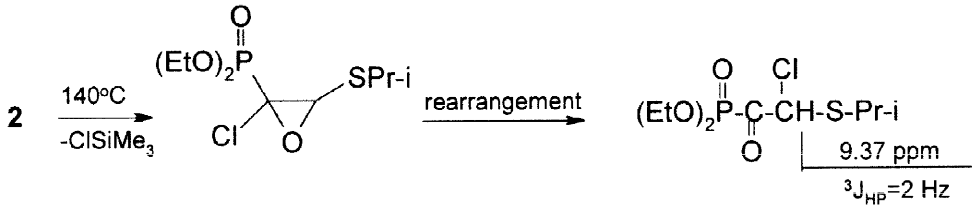
The addition of mercaptane to dialkoxyphosphoroyldichloroacetaldehyde proceeds at room temperature with the formation of the corresponding hemiacetal **1**, which forms a silyl ether **2** when reacted with trimethylchlorosilane in the presence of pyridine.



$$(EtO)_2\overset{O}{\overset{\|}{P}}CCl_2CH{=}O + i\text{-}PrSH \longrightarrow \underset{\mathbf{1}}{(EtO)_2\overset{O}{\overset{\|}{P}}CCl_2\overset{OH}{\overset{|}{C}}H\text{-}SPr\text{-}i} \xrightarrow[-Py.HCl]{ClSiMe_3\ ,Py} \underset{\mathbf{2}}{(EtO)_2\overset{O}{\overset{\|}{P}}CCl_2CH(SPr\text{-}i)OSiMe_3}$$

5.26 ppm, $^3J_{HP}$=2 Hz

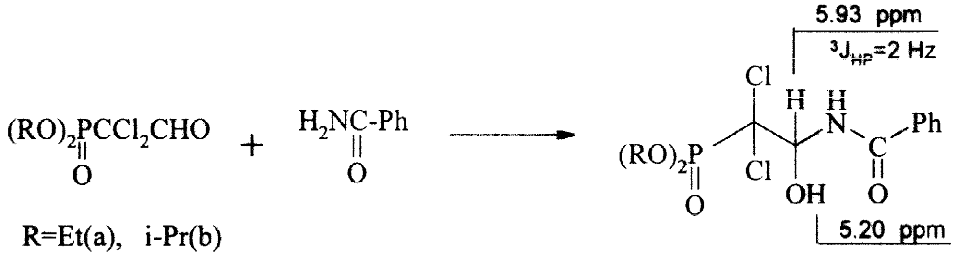
It was observed that above 140°C the silyl ether **2** eliminates trimethylchlorosilane with the formation of α-ketophosphonate **3**. We believe that the instable intermediate oxirane **4**, which easily undergoes the rearrangement into ketophosphonate **3**, should be the primary product of the trimethylchlorosilane elimination reaction.



3 **4**

The structures of compounds 2 and 3 were determined by NMR and IR-spectroscopic methods.

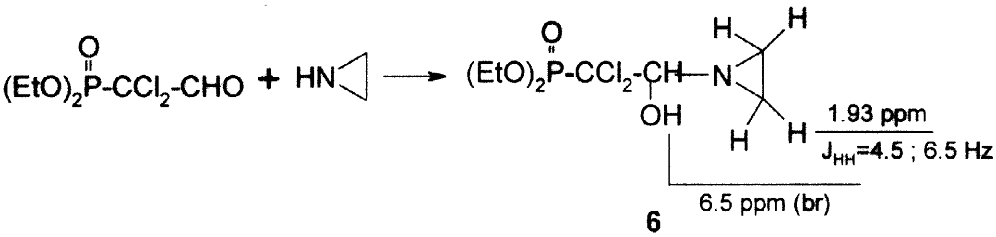
It has been shown for the first time that the condensation of phosphonchlorals with benzamide in benzene solution at 40°C results in the formation of stable phosphorylated hemiamidals **5a,b**.



5a,b

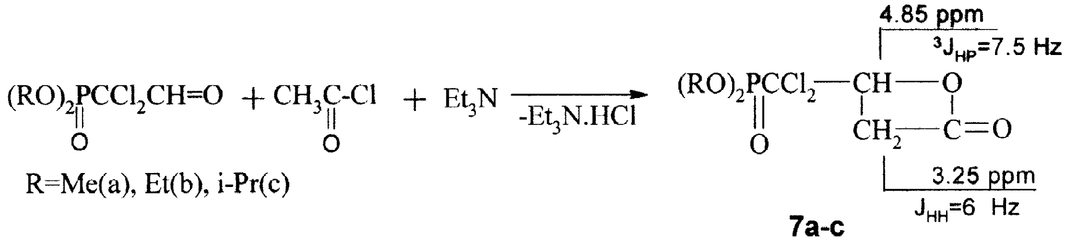
The stabilities of hemiacetals, hemithioacetals, hemiamidals are, apparently, caused by both the presence of the dialkoxyphosphoryldichloromethyl group (by analogy with chloralhydrate) and the intramolecular hydrogen bonds.

Dialkoxyphosphoroyldichloroacetaldehyde has reacted very easily with ethyleneimine at 5-10°C to give hemiaminals (hydroxyaziridines), which are crystalline compounds stable at room temperature for a long time.



6

Dialkoxyphosphoryldichloroacetaldehydes undergo a [2+2] cycloaddition with ketene which was obtained *in situ* from acetyl chloride and triethylamine, with the formation of β-phosphoryldichloromethyl-β-propiolactones **7a-c** which are yellowish viscous liquids.

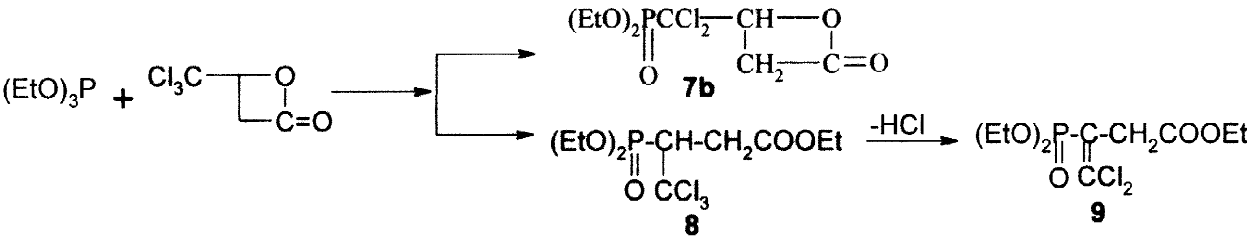


7a-c

The composition and structure of β-lactones **7a-c** were determined on the basis of their elemental analysis , IR and NMR (^{1}H and ^{31}P) spectral data.

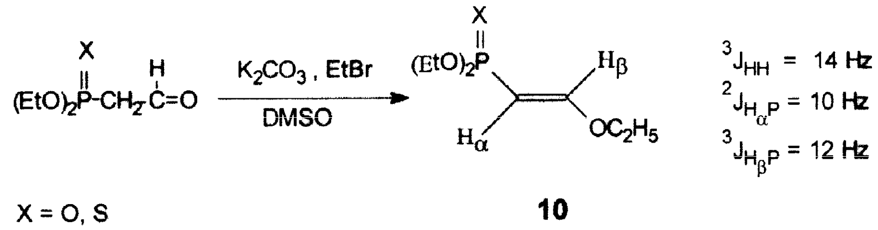
The absorption bands in the ranges of 1270-1280 cm^{-1} and 1860-1870 cm^{-1} were found in the IR spectra of compounds **7a-c** which correspond to the stretching vibrations of the phosphoryl group (P=O) and of the carbonyl vibrations[5] of β-propiolactones, respectively. Besides the signals of alkoxyl groups, a resonance which appeared as a doublet at δ 3.25 ppm (2H,-CO-CH$_2$, $^3J_{HH}$=6 Hz), and a quartet at δ 4.85 ppm (1H,CH-O-, $^3J_{HP}$=7.5 Hz), were observed in the ^{1}H-NMR spectra of the products 7**a-c**. The chemical shifts of phosphorus in compounds 7**a-c** were in the range of 7.5 - 8.5 ppm in the ^{31}P NMR spectra.

We also obtained the product **7b** by an alternative synthesis, namely the interaction of triethylphosphite with β-trichloromethyl-β-propiolactone. It was established that the reaction proceeds mainly in two directions, namely by the mechanism of the Arbuzov reaction and by opening of the lactone ring.

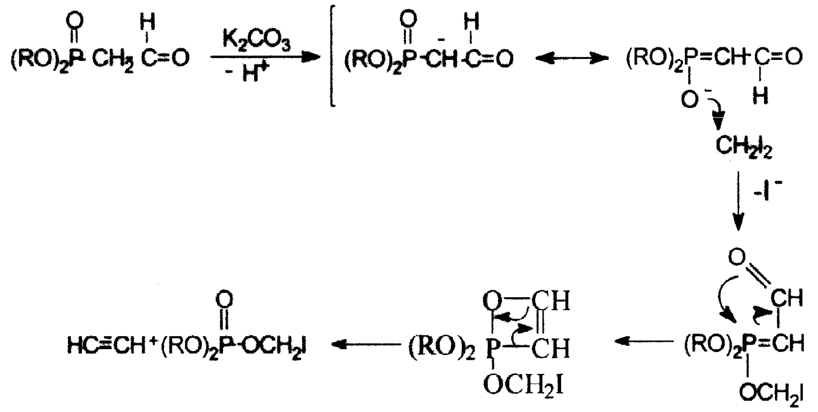


The product **8** which is formed by opening of the lactone ring, is thermally unstable and under the reaction conditions it dehydrochlorinates into the ethyl ester of 4,4-dichloro-3-diethoxyphosphoryl-but-3-enoic acid **9** .

It was found that the alkylation of dialkyl esters of phosphonacetaldehydes with ethyl bromide in DMSO or in DMF in the presence of K$_2$CO$_3$ proceeded with the formation of the products of *O*-alkylation. Thus, enol ether **10** was formed with trans configuration.



When dialkoxyphosphonacetaldehyde was alkylated with diiodomethane in the presence of potassium carbonate in DMSO, dialkoxyiodomethyl phosphates were isolated via elimination of acetylene. Acetylene was detected by passing the gaseous mixtures through bromine water. The reaction pathway is given as follows.



Diethyliodomethyl phosphate is a stable compound, b.p:88-90°C(0.5 Torr). NMR ^1H (CCl_4) (δ ppm ; J Hz): 1.6 ppm (t,6H,2C<u>H</u>$_3$, $^3J_{HH}$= 7 Hz) and 4.3 ppm (q, 4H,2C<u>H</u>$_2$O, $^3J_{HH}$=$^3J_{HP}$=7 Hz), 4.3 ppm (s, 2H, OC<u>H</u>$_2$I, $^3J_{HP}$=6 Hz).

We have proposed before[6] that the alkylation of dialkoxyphosphorylacetaldehydes by dibromoethane under the indicated conditions proceeds with the primary formation of an enol ether which either dealkylates under the reaction conditions or during the fractional distillation to give the seven membered phosphopene heterocycle containing a phosphorus atom in the ring. However, in recent experiments the enol ether did not yield the heterocyclic product , indicating that our former proposal is incorrect.

The reactions between dialkoxyphosphorylacetaldehydes and polyelectrophilic reagents, such as 1,2-dibromoethane or 1,3-dibromopropane, were more complicated, since CH_2, P=O and C=O groups were simultaneously involved in the reactions. At the end of the reactions, novel enol ether compounds (Table 1) and phosphatecyclanes all in trans configuration (Table 2) were isolated.

The nature of the substituents both at phosphorus and in the halogenated reagent directs the alkylation reaction. Thus, we suppose that the product of a cyclic structure is formed from the alkylation on the phosphoryl oxygen and the enol ethers are formed by the direct alkylation of carbonyl oxygen. The isolation of enol ethers and dialkyliodomethylphosphate is the experimental confirmation of the above-stated theory.

EXPERIMENTAL

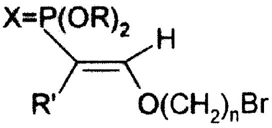
^1H and ^{31}P NMR spectra were recorded on a Bruker WR-80 using internal TMS or H_3PO_4 85 % references. Infrared spectra were recorded on a UR-20 spectrophotometer.

Chlorination of diisopropoxyphosphorylacetaldehyde by molecular chlorine at 20-30°C

Dry chlorine was bubbled through a solution of diisopropoxyphosphorylacetaldehyde (20.8 g, 0.1 mol) in CCl_4 (100 mL) at 20-30°C until a greenish coloration appeared.

Table 1

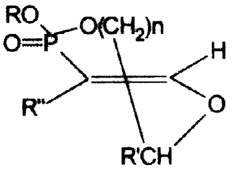
Substitued Vinylphosphonates



No	N	R	R'	X	Empirical Formula	Analyses (%)				B.p.(°C) (p, Torr)	d_4^{20} (g/mL)	n_D^{20}	Yield %
						Calcd.		Found					
						Br	P	Br	P				
1	2	Me	H	O	$C_6H_{12}BrO_4P$	30.92	11.93	30.52	11.61	120-121 (0.7)	1.4038	1.4720	23
2	2	Et	H	O	$C_8H_{16}BrO_4P$	27.87	10.80	27.34	10.58	143-145 (0.5)	1.3287	1.4685	23
3	2	Et	H	S	$C_8H_{16}BrO_3PS$	24.31	9.42	24.11	9.32	148 (0.5)	1.3393	1.4795	34
4	2	*i*-Pr	H	O	$C_{10}H_{20}BrO_4P$	25.43	9.81	25.01	9.73	132-134 (0.08)	-	1.4600	41
5	2	*n*-Pr	H	O	$C_{10}H_{20}BrO_4P$	25.49	9.81	25.14	9.52	148-149 (0.08)	-	1.4660	39
6	2	Et	Et	O	$C_{10}H_{20}BrO_4P$	25.43	9.81	25.08	9.41	153-155 (0.5)	1.2417	1.4768	40
7	3	*i*-Pr	H	O	$C_{11}H_{22}BrO_4P$	24.30	9.40	23.82	9.13	136-138 (0.5)	1.2447	1.4675	42

Table 2

Phosphatecyclanes



No	n	R	R'	R"	Empirical Formula	Analyses P (%)		B.p.(°C) (p,Torr)	d_4^{20} (g/mL)	n_D^{20}	Yield %
						Found	Calcd.				
1	1	Me	H	H	$C_5H_9O_4P$	18.82	18.90	82.5-84 (0.1)	1.2227	1.4450	38
2	1	Et	H	H	$C_6H_{11}O_4P$	17.18	17.42	93-95 (0.5)	1.1909	1.4420	48
3	1	Me	Me	H	$C_6H_{11}O_4P$	16.95	17.42	93-94 (0.5)	1.1510	1.4505	51
4	1	*i*-Pr	H	H	$C_7H_{13}O_4P$	16.01	16.33	92-94 (0.7)	1.1511	1.4460	28
5	1	*n*-Pr	H	H	$C_7H_{13}O_4P$	16.12	16.33	102-103 (0.5)	1.1708	1.4490	14
6	1	Et	Me	H	$C_7H_{13}O_4P$	15.92	16.33	115-117 (0.5)	1.0822	1.4420	48
7	2	*i*-Pr	H	H	$C_8H_{15}O_4P$	15.00	15.05	90-91 (0.5)	0.9978	1.4355	15
8	1	Et	H	Et	$C_8H_{15}O_4P$	15.11	15.05	94-96 (0.5)	1.1041	1.4478	8

After distillation of the solvent, by fractional distillation β,β-dichloroethylacetate (2.8 g, 18%) was isolated along with diisopropoxyphosphoryldichloroacetaldehyde (18.8 g, 68%), colourless liquid, b.p.: 90-92°C(0.5 Torr); d^{20}_4 1.2548 ; n_D^{20} 1.4535.[3,4]

Addition of isopropylmercaptan to diethoxyphosphoryldichloroacetaldehyde
Isopropylmercaptan (2g , 26.3 mmol) was added to diethoxyphosphoryldichloroacetaldehyde (7 g, 28.1 mmol). The mixture was warmed up. The product **1**, pale yellow liquid, with d_4^{20} 1.2061 , n_D^{20} 1.4840 was obtained. [Found: Cl, 21.64; P, 9.04. $C_9H_{19}Cl_2O_4PS$ requires Cl, 21.84; P, 9.20 %] ν_{max} (liquid film) 1270 (P=O), 3300-3400(OH) cm^{-1}.

2-Isopropylthio–2-trimethylsiloxy-1,1-dichloroethylphosphonousacid diethyl ester (2)
Isopropylmercaptane (1.5 g, 19.7 mmol) was added dropwise during mixing at 0-5°C to diethoxyphosphoryldichloroacetaldehyde (5 g, 20.1 mmol). The reaction mixture was mixed for 2 hrs at room temperature. A hemithioacetal **1** was obtained. Following the addition of pyridine (1.6 g, 20.2 mmol) and anhydrous methylene chloride (25 mL), the mixture was cooled up to 0°C and trimethylchlorosilane (2.3 g, 21.2 mmol) was added dropwise during mixing . The mixture was kept for a night and the residue was separated. Fractional distillation of the residue gave the product **2** (4.8 g, 66%), colourless oil, b.p.:124-125 °C (1Torr), [Found: Cl, 18.7; P, 8.15. $C_{12}H_{27}Cl_2O_4PSSi$ requires Cl, 19.4; P, 8.4 %] n_D^{20} 1.4805; ν_{max}(liquid film) 1050 (br,P-O-C),1260(P=O) cm^{-1}. δ_H ($CDCl_3$) 0.07 (s, 9H, $SiMe_3$), 1.22 (m, 12H, $4CH_3$), 3.13 (q, *J* 6.2 Hz,1H, SCH), 4.23 (m, *J* 6.4, 8.0 Hz, 4H, $2OCH_2$), 5.26 (d, 1H, $^3J_{HP}$ 2.0 Hz, $PCCl_2CH$). δ_P ($CDCl_3$) 9.96 ppm.

2-Isopropylthio-2-chloro-1-oxoethylphosphonous acid diethyl ester (4)
Ester **2** (4 g, 11 mmol) in o-xylene (15 mL) was refluxed for 8 hrs. The solvent was removed in vacuum. Product **4** (1.7 g, 55%) was obtained by fractional distillation of the residue. Pale yellow liquid,b.p.:104-105 °C (0.08 Torr); [Found: Cl, 12.6; P, 10.5. $C_9H_{18}ClO_4PS$ requires Cl, 12.3; P,10.72 %]; n_D^{20} 1.4876; ν_{max} (liquid film) 1035(P-O-C), 1275 (P=O), 1735 (C=O) cm^{-1}; δ_H ($CDCl_3$) 1.25 (m, 12H, $4CH_3$), 3.15 (q , *J* 6.2 Hz, 1H, SCH), 4.22(m, *J* 6.4, 8.0 Hz, 4H, $2OCH_2$), 9.37(s, $^3J_{HP}$ 2.0 Hz, 1H, ClCHS); δ_C ($CDCl_3$) 15.0(CH_3), 23.4 (CH), 64.2 (CH_2O), 76.5 (d,CHCl, $^2J_{CP}$ 10.0 Hz), 183.9 (C=O); δ_P ($CDCl_3$) 9.5 ppm.

N-(1-Hydroxy-2,2-dichloro-2-dialkoxyphosphorly)ethylbenzamides (5a,b)
The mixture of phosphonchloral (0.20 mol) and benzamide (21.8 g, 0.18 mol) in dry benzene (100 mL) was kept at 40-45 °C for 24 hrs. The solvent was evaporated, the crystals of compound (**5a** or **b**) were filtered, washed by ether, dried *in vacuo*.
N-(1-Hydroxy-2,2-dichloro-2-diethoxyphosphorly)ethylbenzamide(5a):Amorphous powder (72.5 g, 98%), m.p. 91-92 °C; [Found: Cl, 18.91; N, 3.95; P, 8.12. $C_{13}H_{18}Cl_2NO_5P$ requires Cl, 19.19; N, 3.78; P, 8.37 %]; ν_{max}(KBr) 1025(P-O-C), 1230(P=O), 1670(NC=O), 3180(OH), 3320(NH) cm^{-1}; δ_H ($(CD_3)_2CO$) 1.3(t, *J* 6.4 Hz, 6H, $2CH_3$), 4.32(m, *J* 6.4, 8.0 Hz, 4H, $2OCH_2$), 5.20(d ,1H, OH), 5.93(d.d, $^3J_{HP}$ 2.0 Hz, 1H, CH), 7.45 (m, 3H, Ph), 7.85 (m, 2H, Ph), 8.35 (br.d, 1H, NH); δ_P ($CDCl_3$) 10.0 ppm.

N-(1-Hydroxy-2,2-dichloro-diisopropoxyphosphorly)ethylbenzamide(5b):Amorphous solid (77.2 g, 97%); m.p. 69.5-71 °C; [Found: Cl, 17.40; N, 4.00; P, 7.75. $C_{15}H_{22}Cl_2NO_5P$ requires Cl, 17.84; N, 3.52; P, 7.79 %]; ν_{max}(KBr) 1030(P-O-C), 1260(P=O), 1670(NC=O), 3160(OH), 3320(NH) cm^{-1}; δ_H ($(CD_3)_2CO$) 1.32 (d.d, J 6 Hz, 12H, $4C\underline{H}_3$), 4.92 (m, 2H, $2OC\underline{H}$), 6.10 (d.d, $^3J_{HP}$ 2.0 Hz, 1H, $C\underline{H}$), 6.75 (br, 1H, $O\underline{H}$), 7.42 (m, 3H, $\underline{Ph}$), 7.90 (d.d, 2H, $\underline{Ph}$), 8.47 (br.d, 1H, $N\underline{H}$); δ_p ($CDCl_3$) 8.00 ppm.

β-Hydroxy-β-ethyleneimino-α,α-dichloroethylphosphonic acid diethyl ester (6)

Diethoxyphosphoryldichloroacetaldehyde (12 g, 48.2 mmol) was added during mixing at 5-10 °C to an ethereal solution of ethyleneimine (2.1 g, 48.8 mmol). The reaction mixture was mixed for an hour, and ether was removed. β-Hydroxy-β-ethyleneimino-α,α-dichloroethyl phosphonic acid diethyl ester **6** (13.95 g, 99%) was obtained as light yellow crystals. M.p.: 59-60 °C. [Found: N, 4.68; P, 10.47. $C_8H_{16}Cl_2NO_4P$ requires N, 4.79; P, 10.62 %]; δ_H (400 MHz, $CDCl_3$) 6.5 (b, 1H, OH), 5.75 (1H, d, $^3J_{HP}$ 2.0 Hz, CCl_2-$C\underline{H}$-N), 4.10 (4H, m, J 6.4, 8.0 Hz $(CH_3C\underline{H}_2O)_2P(O)$-), 1.93 (4H, dd, J 6.5, 4.1 Hz, $2C\underline{H}_2$ of ethyleneimine moiety), 1.27 (t, J 6.4 Hz, 6H, $(C\underline{H}_3CH_2O)_2P(O)$-).

β-Dimethoxyphosphoryldichloromethyl-β-propiolactone (7a)

Colourless oil.Yield:78%. B.p.128-130°C(0.08Torr); [Found: Cl, 20.49; P,10.40. $C_8H_{13}Cl_2O_5P$ requires Cl 20.96; P, 10.65 %]; n_D^{20} 1.4720.

β-Diisopropoxyphosphoryldichloromethyl-β-propiolactone (7b)

Colourless oil. Yield: 63%. B.p.: 123-125°C (0.07 Torr); [Found: Cl, 22.07; P, 8.00. $C_{10}H_{17}Cl_2O_5P$ requires Cl, 22.25; P, 9.07 %]; n_D^{20} 1.4780.

4,4-Dichloro-3-diethoxyphosphoryl-but-3-enoic acid ethyl ester (9)

A mixture of triethylphosphite (8.3 g, 50 mmol) and β-trichloromethyl-β-propiolactone (9.5 g, 50 mmol) was heated at 140-150°C during mixing for 4 hrs. Fractionating distillation gave three products. 1st fraction: 5.05 g (32%) product (**7b**), B.p.119-121°C (0.05 Torr), n_D^{20} 1.4725. 2nd fraction: 9 g, B.p.127-147°C (0.05 Torr) products of unstated structure. 3rd fraction was identified as product (**9**) (2.68g, 17 %), colourless oil, b.p.: 149-151°C (0.05 Torr); [Found: Cl, 22.1; P, 9.22. $C_{10}H_{17}Cl_2O_5P$ requires Cl, 22.2; P, 9.07 %]; n_D^{20} 1.4690;ν_{max}(liquid film) 1270(P=O), 1640($C=CCl_2$), 1690(C=O)cm^{-1}.δ_H (CCl_4) 1.20(t,J 6.4 Hz, 9H, $3C\underline{H}_3$), 3.95 (d, 2H, $C\underline{H}_2$), 4.25(m, 6H, $3C\underline{H}_2O$, $^3J_{PH}$ 7.8 Hz).

General procedure for the alkylation of α-phosphoncarbonyls by alkyl halides

α-Formylcarbonyl compound (0.1 mol), alkyl halide (0.2 mol) and potassium carbonate(0.4 mol) in DMSO (100 mL) were vigorously mixed at 40-60°C for 6-7 hrs. Following the addition of water, the reaction product was carefully extracted by ether. The organic layer was dried over anhydrous sodium sulfate. Ether was removed and the residue was vacuum distilled. By this procedure, enol ethers (Table 1) and heterocyclic organophosphorus compounds (Table 2) were obtained.

Diethyliodomethyl phosphate
Phosphoneacetaldehyde diethyl ester (18.0 g, 0.1 mol), methylene iodide (53.6 g, 0.2 mol) and potassium carbonate (55.2 g, 0.4 mol) in DMSO (100 mL) gave diethyliodomethylphosphate (5.8 g, 20%), colourless liquid, b.p.: 88-90 °C (0.5 Torr); d_4^{20} 1.5946; n_D^{20}1.4635.

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