600703

BIMONTHLY REPORT NO. 1

DEVELOPMENT OF FIRE-RESISTANT WATER BASE HYDRAULIC FLUID

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To
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I. INTRODUCTION

The broad objective of this contract is the development of a usable, fire-resistant water-base hydraulic fluid for shipboard use, in which the nonaqueous phase is fire resistant. The contract effort is restricted to water solutions as contrasted with emulsions and/or suspensions.

Fire resistance in the nonaqueous phase is important in hydraulic systems operating at 5000 psi, where the aqueous phase in certain segments of the system may, under special circumstances, evaporate, thereby leaving a hazardous explosive residue. and where spray leaks develop there may be flammable residues exposed to ignition sources.

The suggested, desirable, and tentative specifications for a fire-resistant water-base hydraulic fluid are:

1.	Autogenous Ignition Temperature	(AIT) > 900°F (nonaqueous phase)
2.	Flash Point	(AIT) > 450°F (nonaqueous phase)
3.	Fire Point	(AIT) > 550°F (nonaqueous phase)
4.	Viscosity cs.	850 (max) at 25°F and 25-31 at 150°F
5.	Pour Point	0°F (max)
6.	Shear Stability	±10% change at 150°F (max)
7.	Specific Gravity 60/60°F	1.0-1.2 (1.6 max)
8.	Lubrication	Shipboard screw and variable stroke piston pumps at 5000 psi
9.	Compatibility	
	(a) Metals	Steel, copper, copper-nickel, nickel-copper-bronze, phosphorus-bronze, and anodized aluminum (see metal specifications)
	(b) Elastomers	Buna N
	(c) Sea Water	Functional with 10%

10.	Stability	
	(a) Storage	-20 to +110°F
	(b) Use	+25 to 180°F
	(c) Filterable	5 Micron filter
11.	Foaming	No stable foam
12.	Toxicity	Non-hazardous
13.	Fluid Residues	Water soluble.

Our approach to this problem will be divided into four major phases:

(1) Selection, by synthesis and evaluation, of one or two fire-resistant pour point depressants.

(2) Selection of a water-soluble polymeric thickener compatible with the pour point depressant.

(3) Inhibition of the specified metals against corrosion in the liquid and vapor phases by the water solution of the pour point depressant and thickener.

(4) Compounding the finished fire-resistant, water-base hydraulic fluid.

II. SUMMARY

Partial alkyl ester alkali salts of phosphates and phosphonates are promising candidates as pour point depressants for fire-resistant, water-base hydraulic fluids. These compounds appear hydrolytically stable in alkaline solution at 200°F and possess high autoignition temperatures (AIT).

The alkylphosphoramidates, because of their low AIT values, are of no value for this application. Mono-chlorination of an n-alkyl phosphate also makes no contribution to the AIT of the trialkyl ester.

Water-soluble aromatic phosphates without hydrolytic stabilization are of no further interest.

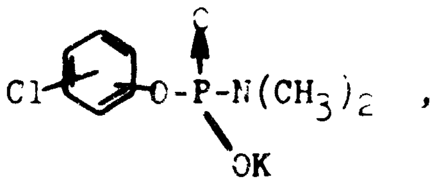
III. GENERAL DISCUSSION

In the present contract period numerous exploratory scouting experiments were conducted, along with a complementary synthesis program, in an effort to establish the utility of phosphorus esters, phosphoramidates and partial ester (alkali) phosphates, and phosphonates as fire-resistant, pour point depressants.

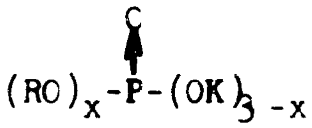
We have, in general, leaned heavily toward the alkyl phosphorus derivatives in preference to the aryl derivatives because of a reference in the literature by R. H. S. Plimmer and W. J. N. Burch (J. Chem. Soc., 1929, 279), in which these authors report the mono-, di- and tri-phenyl phosphates are completely hydrolyzed by dilute acid and by dilute alkali. Our results substantiate their data.

Further, as a result of the hydrolytic instability of the water-soluble triethyl phosphate (this report) and of the low AIT's of the phosphoramidates, we have preferred the partial alkyl ester (alkali) phosphates and/or phosphonates. The mono- and di-alkyl esters of phosphates have been reported by Plimmer and Burch (ibid) to be stable in alkaline solution and our results have also substantiated this finding.

The introduction of secondary amino groups into aryl phosphates to produce diaryl phosphoramidates has a stabilizing effect on the hydrolytic stability of the molecule. We are therefore synthesizing a molecule of the following structure,



to obtain, if possible, some improvement in the AIT above the level (approaching 900°F) shown by the



$$(RO)_x\text{-}\overset{O}{\overset{\uparrow}{P}}\text{-}(OK)_{3-x}$$

type molecules. Additional and more specific observations are made in the subsequent sections of this report.

IV. PHYSICAL TESTING

A. FLAMMABILITY PROPERTIES

If a water-base hydraulic fluid is to be obtained, in which the water-soluble portion has an AIT of 900°F or higher, then the pour point depressant should possess the maximum AIT to compensate for any potential lowering from the addition of a thickener. Therefore the AIT values of a variety of water-soluble phosphorus compounds were examined in this contract period and are reported in Table 1. From these data some broad conclusions can be made:

1. The aliphatic phosphoramidates are materially lower in AIT than the closely analogous esters (compare compounds 460, 462, and 423 with compounds 455, 437, and 467). These values for the phosphoramidates eliminate this class from further consideration per se.

2. The introduction of β-chlorine into triethyl phosphate contributed essentially nothing to the AIT of the ester class (compare Cpds. 437 and 456). This is probably due to the low decomposition temperature of the halogenated compound.

3. There is a suggestion that the mono- and di-alkyl (alkali) phosphates and the alkali alkylphosphonates may be higher in AIT than the neutral esters (compare Cpds. 455 with 442 and 443; Cpds 467 with 451 and 465).

4. From these AIT values the alkyl (alkali) phosphates and alkali alkylphosphonates are judged candidate, fire-resistant, pour point depressants. (See compounds 441, 442, 443, 451, 463, and 465).

5. A water-soluble phosphine oxide was low in AIT (Cpd. 438).

B. HYDROLYTIC STABILITY

As previously mentioned, Plimmer and Burch have reported the mono-, di- and tri-phenyl phosphates as hydrolytically unstable in dilute alkali and in dilute acid. The results (see Table 2) of our small-scale tests, both with organic bases and with alkali, corroborate their results. The alkali salt of phenyl phosphate at pH 7 shows only a trace of hydrolysis as measured by NMR (see Cpd. 433). The lower limit of detection by NMR of a different phosphorus environment is about 5%. This suggests that a mono-aryl phosphate, to have any utility, must be stabilized. Stabilization may be effected by conversion to the phosphoramidate, i.e.,

Table 1

FLAMMABILITY PROPERTIES

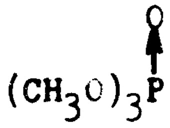
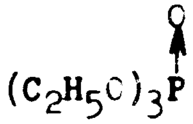
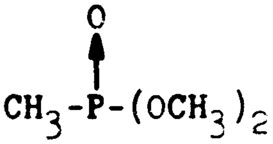
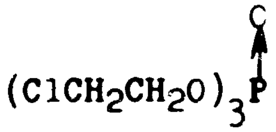
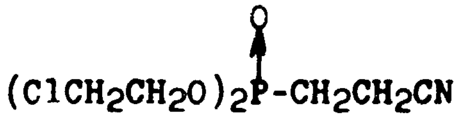
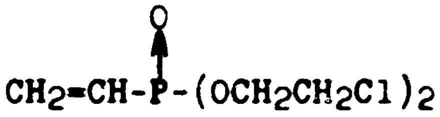
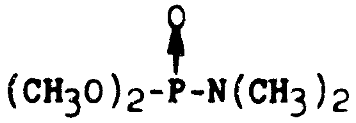
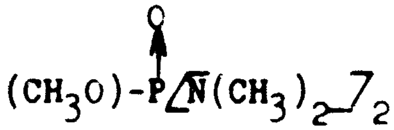
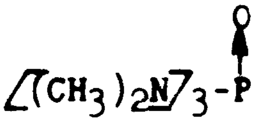
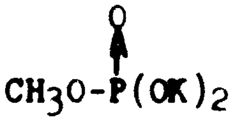
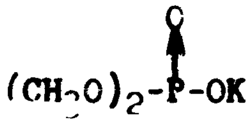
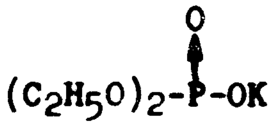
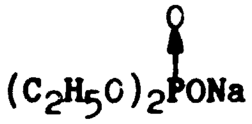
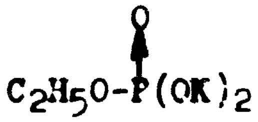
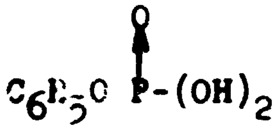
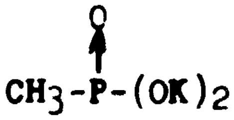
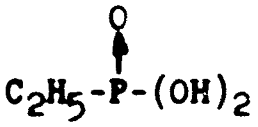
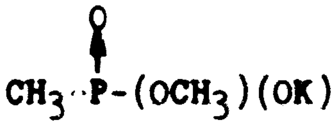
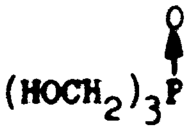
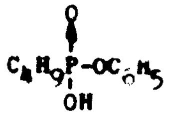
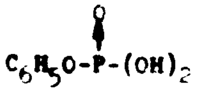
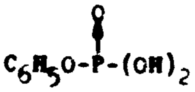
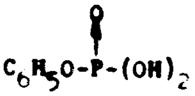
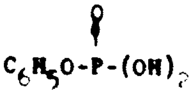
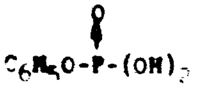
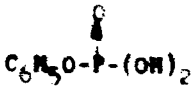
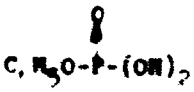
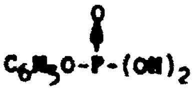
MRC No.	Compound	AIT °F.	AIT Time Lag Sec.	Micro Flash °F.	Micro Fire °F.
455	 $(CH_3O)_3\overset{O}{\overset{\uparrow}{P}}$	725	13	-	-
437	 $(C_2H_5O)_3\overset{O}{\overset{\uparrow}{P}}$	840	5	-	-
467	 $CH_3-\overset{O}{\overset{\uparrow}{P}}-(OCH_3)_2$	700	22	-	-
456	 $(ClCH_2CH_2O)_3\overset{O}{\overset{\uparrow}{P}}$	850	3	518	608
444	 $(ClCH_2CH_2O)_2\overset{O}{\overset{\uparrow}{P}}-CH_2CH_2CN$	805	5	507	no fire 581
447	 $CH_2=CH-\overset{O}{\overset{\uparrow}{P}}-(OCH_2CH_2Cl)_2$	745	6	-	-
460	 $(CH_3O)_2-\overset{O}{\overset{\uparrow}{P}}-N(CH_3)_2$	500	10	-	-
462	 $(CH_3O)-\overset{O}{\overset{\uparrow}{P}}[N(CH_3)_2]_2$	495	5	-	-
423	 $[(CH_3)_2N]_3-\overset{O}{\overset{\uparrow}{P}}$	485	9	265	360
443	 $CH_3O-\overset{O}{\overset{\uparrow}{P}}(OK)_2$	925	18	-	-

Table 1 (cont'd)

		AIT		Micro	
MRC No.	Compound	°F.	Time Lag Sec.	Flash °F.	Fire °F.
442	 $(CH_3O)_2-\overset{O}{\overset{\uparrow}{P}}-OK$	840	15	446	no fire 644
468	 $(C_2H_5O)_2-\overset{O}{\overset{\uparrow}{P}}-OK$	-	-	-	-
441	 $(C_2H_5O)_2\overset{O}{\overset{\uparrow}{P}}ONa$	830	9	493	604
463	 $C_2H_5O-\overset{O}{\overset{\uparrow}{P}}(OK)_2$	875	17	-	-
433	 $C_6H_5O\ \overset{O}{\overset{\uparrow}{P}}-(OH)_2$	1165	4	370	no fire 682
465	 $CH_3-\overset{O}{\overset{\uparrow}{P}}-(OK)_2$	1140	53	-	-
339	 $C_2H_5-\overset{O}{\overset{\uparrow}{P}}-(OH)_2$	910	5	750	no fire 760
451	 $CH_3-\overset{O}{\overset{\uparrow}{P}}-(OCH_3)(OK)$	880	17	-	-
434	$[C_6H_5]_2POH$	1160	4	594	no fire 743
438	 $(HOCH_2)_3\overset{O}{\overset{\uparrow}{P}}$	625	4	464	500

HYDROLYSIS(1) OF

MRC No.	Compound	Ratio Cpd/H$_2$O
466	 $C_4H_9-\overset{\overset{O}{\|}}{\underset{OH}{P}}-OC_6H_5$	3.1/1.21
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	3.6/1.22
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	2.0/2.0
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	2.0/2.0
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	2.0/2.0
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	2.0/2.0
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	2.0/2.0
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	1/1
433	 $C_6H_5O-\overset{\overset{O}{\|}}{P}-(OH)_2$	1/1

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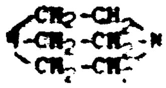
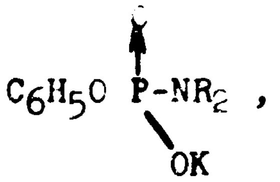
(1) Small-scale experiments

Table

OF ARYL PHOSPHATES AND ARYL PHOSPHONATES

o [illegible]	Salt	pH	Temp. F	Time In Hours	[illegible] Analysis % Hydrolyzed
.11	None	Acid	1[illegible]	1[illegible] 1[illegible]	None None
.2[illegible]	None	Acid	1[illegible]	1[illegible]	[illegible]
.0	Morpholine		[illegible]	[illegible]	Nearly complete
.0	Triethylamine	[illegible]	[illegible]	1[illegible]	Nearly complete
.[illegible]	NaOH		[illegible]	[illegible]	Trace
.0	Benzyltrimethyl ammonium hydroxide		[illegible]	[illegible]	Nearly complete
.0	Dabco 		[illegible]	1[illegible]	Complete
	NaOH	[illegible]	[illegible]00	1[illegible]	[illegible]
	Dabco	[illegible]	[illegible]	[illegible]	[illegible]



$$C_6H_5O\,\underset{\diagdown OK}{\overset{\overset{O}{\uparrow}}{P}}\text{-}NR_2\,,$$

which is one additional type molecule currently under investigation in this class.

The apparent hydrolytic stability of compound 466 is probably due to its insolubility.

The hydrolytic data on alkyl phosphates and their salts are shown in Table 3. Water-soluble, low molecular weight trialkyl phosphates are of insufficient hydrolytic stability for this application (see Cpd. 437). Although anomalies exist in these data, they do indicate that the alkyl (alkali) phosphates have adequate stability for this application.

C. FREEZING POINT

Solutions were made of diethyl potassium phosphate (Cpd. 468) as shown in Table 4. From these data the attainment of satisfactory pour points and freezing points for the objectives of this contract, using alkali salts of the alkyl phosphates, appears to be no problem.

D. CORROSION

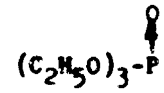
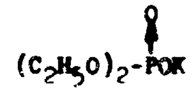
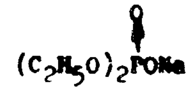
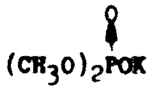
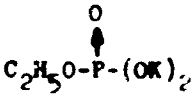
Preliminary corrosion screening tests were conducted with disodium ethylphosphonate as shown in Table 5. Although the quality of this acid has now become questionable, it does suggest that this type of molecule in the absence of sea water is not corrosive to steel. Experiments have begun which will determine, more firmly, if the alkali salts of alkylphosphonates and alkyl phosphates are noncorrosive and at what p^H.

In this preliminary experiment corrosion does take place rapidly in synthetic sea water. Inhibition against sea water corrosion will be investigated. The synthetic sea water was formulated according to Interim Federal Specifications in P-S-610 (Navy Ships), 28 March 1952. We are proposing to use natural sea water in forthcoming tests.

Ta

HYDROLYSIS OF ALKYL PHOSPHA

(Coke Bottle Te

MRC No.	Compound	Ratio Gms Cpd/H$_2$O	Temp °F.	Time hrs	I
437	 $(C_2H_5O)_3\text{-}\overset{O}{\overset{\uparrow}{P}}$	75/25	200	48	
468	 $(C_2H_5O)_2\text{-}\overset{O}{\overset{\uparrow}{P}}OK$	37.5/37.5	200	48	
		37.5/37.5	200	48	
		25/25	200	48	1
		25/25	200	48	
441	 $(C_2H_5O)_2\overset{O}{\overset{\uparrow}{P}}ONa$	25/25	200	48	
442	 $(CH_3O)_2\overset{O}{\overset{\uparrow}{P}}OK$	25/25	200	48	
463	 $C_2H_5O\text{-}\overset{O}{\overset{\uparrow}{P}}\text{-}(OK)_2$	25/25	200	48	

Specification limits

(1) Compound 437 was run using standard Coke Bottle Ratios. Other runs were on a smaller scale.

Table 3

PHATES AND ALKYL METAL PHOSPHATES

Test Mil-H-19457)(1)

Initial pH	Final pH	Total Acidity Mg KOH/g.	Copper Strip Wt. Change Mg/cm²	Copper Strip Corrosion
-	-	12.67 4.1% hydrolyzed	1.2	Etched badly
7	8.5		0.855	Etched badly
7	[illegible].6		0.[illegible]14	Copper brown, no etching
10	-		0.09	Copper dark brown, no etching
9.1	8.2	0.039	-	Copper brown, no etching
6.0	-	-	0.571	Appeared etched
8.8	9.0	0.44	[illegible]	Copper brown, no sign of etching
9.35	8.8	0.039	0.[illegible]	Tarnished and etched
		0.[illegible] mg	0.3 mg/cm²	

Table 4

FREEZING POINTS OF AQUEOUS PR

MRC No.	Compound	% Concentration in Aqueous Solution
468	Diethyl potassium phosphate	50
		40.7

Table 5

PRELIMINARY CORROSI

MRC No.	Compound	Solvent	C
469	Disodium ethylphosphonate	Distilled water	
470	Dipotassium ethylphosphonate	Distilled water	
	None	Artificial sea water	
339	Ethylphosphonic Acid	Artificial sea water	

le 4

PHOSPHORUS ACID SALTS

in on	°F. Pour Point	Remarks
	-50	Sample crystallized
	-20	Sample crystallized - melted at -14°F. Will supercool to -30°F.

e 5

ROSION DATA

Concentration %	pH	Metal Used		Temp	Remarks
		52-100 Steel	Copper		
30	8.0	X		Room	No corrosion after [illegible] weeks
30	8.0	X		[illegible]00°F	No corrosion after [illegible] weeks
-	6.5	X		[illegible]°F	Corrosion severe in one hour
30	6.5	X		[illegible]00°F	Solution [illegible] - corrosion severe after 18 hrs

V. SYNTHESIS

The following syntheses were carried out to support this program.

Phosphine Oxides

Tris(hydroxymethyl)phosphine Oxide (Cpd. 438) was prepared in near quantitative yield by the hydrolysis of tetrakis-(hydroxymethyl)phosphonium chloride,

$$(HOCH_2)_4\text{-}P\text{-}Cl \;+\; BaCO_3 \xrightarrow[\Delta]{H_2O} (HOCH_2)_3P{=}O \;+\; BaCl_2$$

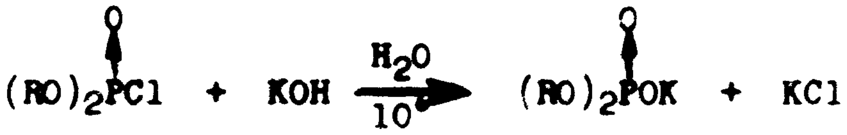
The barium was removed from the dilute solution by precipitation with H_2SO_4, followed by concentration to remove the hydrochloric acid and some formic acid. The neutral product was finally obtained by passing the solution through an anion exchange column.

Phosphates

Diethyl Potassium Phosphate (Cpd. 468) and Dimethyl Potassium Phosphate (Cpd. 442) were prepared by the following sequence of reactions:

$$(RO)_2\overset{O}{\overset{\uparrow}{P}}H \;+\; Cl_2 \xrightarrow{88\%} (RO)_2\overset{O}{\overset{\uparrow}{P}}Cl \;+\; HCl$$

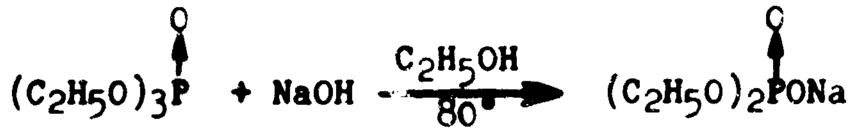
R = Et - bp 86°/10 mm, $n^{25}D$ 1.4150
R = Me - bp 81°/19 mm, $n^{25}D$ 1.4110



$$(RO)_2\overset{O}{\overset{\uparrow}{P}}Cl \;+\; KOH \xrightarrow[10^\circ]{H_2O} (RO)_2\overset{O}{\overset{\uparrow}{P}}OK \;+\; KCl$$

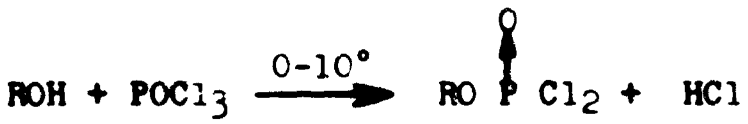
The dehydrated product was separated from the KCl by dissolving in a minimum of cold methanol, filtering, and subsequently removing the solvent under vacuum.

This two-step process was avoided in the preparation of the diethyl sodium phosphate (Cpd. 441) by direct hydrolysis of the triethyl phosphate:



$$(C_2H_5O)_3\overset{O}{\overset{\uparrow}{P}} + NaOH \xrightarrow[80°]{C_2H_5OH} (C_2H_5O)_2\overset{O}{\overset{\uparrow}{P}}ONa$$

Dipotassium Methyl Phosphate (Cpd. 443) **and Dipotassium Ethyl Phosphate** (Cpd. 463) were prepared by the following reactions:



$$ROH + POCl_3 \xrightarrow{0-10°} RO\overset{O}{\overset{\uparrow}{P}}Cl_2 + HCl$$

$$RO\overset{O}{\overset{\uparrow}{P}}Cl_2 + 4KOH \longrightarrow RO\overset{O}{\overset{\uparrow}{P}}(OK)_2 + 2KCl$$

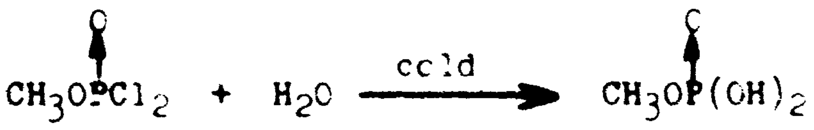
The properties and yields of the first two intermediates were:

$CH_3O\overset{O}{\overset{\uparrow}{P}}Cl_2$, bp 47.5°/13 mm; $n^{25}D$ 1.4332; yield 72%

$C_2H_5O\overset{O}{\overset{\uparrow}{P}}Cl_2$, bp 56.5°/13 mm; $n^{25}D$ 1.4323; yield 84%

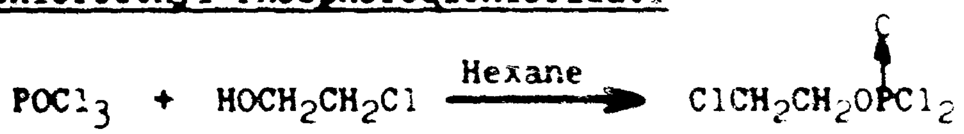
Purification of the partial ester salts was effected by treating the dry salt with a minimum of cold methanol in which the KCl is essentially insoluble.

Attempted hydrolysis of the methyl phosphorodichloridate to the acid using the stoichiometric quantity by water resulted in hydrolysis of some methoxy ester linkage, as evidenced by the presence of phosphoric acid as shown by NMR.



$$CH_3O\overset{O}{\overset{\uparrow}{P}}Cl_2 + H_2O \xrightarrow{cold} CH_3O\overset{O}{\overset{\uparrow}{P}}(OH)_2$$

β-Chloroethyl Phosphorodichloridate



$$POCl_3 + HOCH_2CH_2Cl \xrightarrow{Hexane} ClCH_2CH_2O\overset{O}{\overset{\uparrow}{P}}Cl_2$$

This reaction was carried out in an attempt to get both β-chloroethyl phosphorodichloridate and bis(β-chloroethyl) phosphorochloridate as intermediates. A yield of 68.8% of the dichloridate was obtained and little or no monochloridate.

When the molar ratio of ethylene chlorohydrin to $POCl_3$ was 2:1, a 15.7% yield of dichloridate and 18.7% of monochloridate was obtained. Experiments using triethylamine as HCl scavenger resulted in a viscous,undistillable residue formation.

In view of the low AIT value (850°F) for the tris(β-chloroethyl) phosphate (Cpd. 456), these two intermediates will be held in abeyance.

Phosphonates

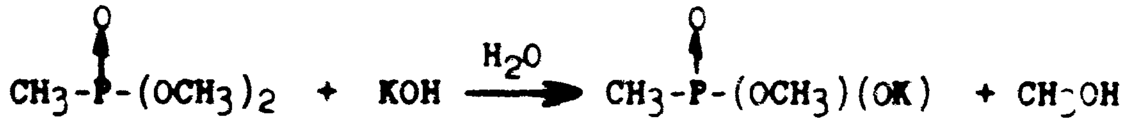
Methylphosphonic Acid (Cpd. 448)

This compound was prepared by HCl catalyzed hydrolysis of dimethyl methylphosphonate. A 59% yield of product, mp 102-103°C (acetone-ether), was obtained.

$$CH_3\overset{O}{\overset{\uparrow}{P}}(OCH_3)_2 \;+\; 2HOH \xrightarrow{HCl} CH_3\text{-}\overset{O}{\overset{\uparrow}{P}}(OH)_2 \;+\; 2CH_3OH$$

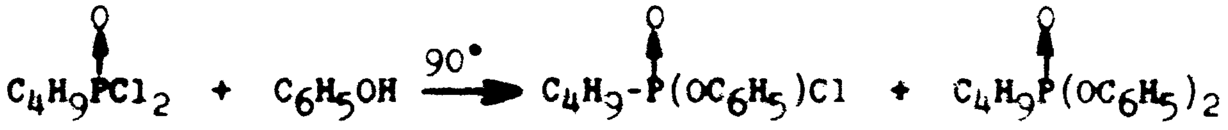
The dipotassium methylphosphonate (Cpd. 465) was made by titrating the methylphosphonic acid (Cpd. 448) with aqueous potassium hydroxide to a p^H of 10 and evaporating to dryness under high vacuum at 80°.

Methyl Potassium Methylphosphonate (Cpd. 451) was prepared by hydrolysis of dimethyl methyl phosphonate for 1.5 hrs at 20° in water solution. The final p^H was 8.1.



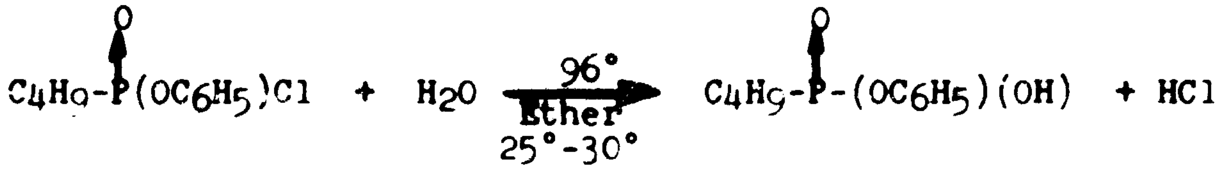
$$CH_3\text{-}\overset{O}{\overset{\uparrow}{P}}\text{-}(OCH_3)_2 \;+\; KOH \xrightarrow{H_2O} CH_3\text{-}\overset{O}{\overset{\uparrow}{P}}\text{-}(OCH_3)(OK) \;+\; CH_3OH$$

Phenyl Hydrogen Butylphosphonate (Cpd. 466) was synthesized by the following sequence of reactions:



$$C_4H_9\overset{O}{\overset{\uparrow}{P}}Cl_2 \;+\; C_6H_5OH \xrightarrow{90^\circ} C_4H_9\text{-}\overset{O}{\overset{\uparrow}{P}}(OC_6H_5)Cl \;+\; C_4H_9\overset{O}{\overset{\uparrow}{P}}(OC_6H_5)_2$$

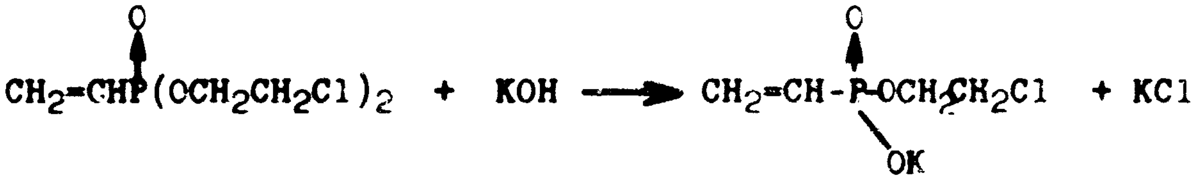
bp 100°/0.04-0.06mm   bp 142-3°/0.06 mm
$n^{25}D$ 1.5093-1.5095 $n^{25}D$ 1.5349-1.5351



$$C_4H_9\text{-}\overset{O}{\overset{\uparrow}{P}}(OC_6H_5)Cl + H_2O \xrightarrow[\text{Ether}\ 25^\circ\text{-}30^\circ]{96^\circ} C_4H_9\text{-}\overset{O}{\overset{\uparrow}{P}}\text{-}(OC_6H_5)(OH) + HCl$$

This product was characterized by NMR.

Attempted Partial Saponification of Bis(β-chloroethyl) Vinylphosphonate in accordance with the equation,

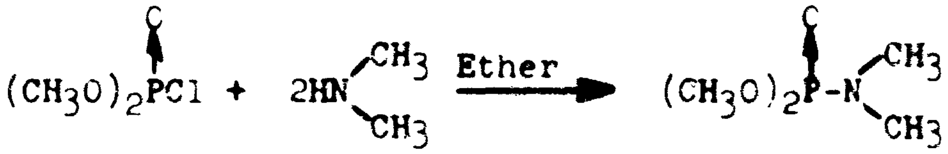


$$CH_2{=}CH\overset{O}{\overset{\uparrow}{P}}(OCH_2CH_2Cl)_2 + KOH \longrightarrow CH_2{=}CH\text{-}\underset{OK}{\overset{O}{\overset{\uparrow}{P}}}\text{-}OCH_2CH_2Cl + KCl$$

at 20-25°C resulted in a viscous liquid/solid mixture (after dehydration). It appeared that dehydrohalogenation, instead of saponification, had occurred.

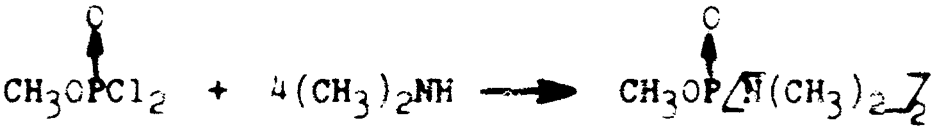
Phosphoramidates

Tetramethyl Phosphoramidate (Cpd. 460) (bp 81°/14 mm; $n^{25}D$ 1.4168) was prepared in 76.8% yield by the following reaction:



$$(CH_3O)_2\overset{O}{\overset{\uparrow}{P}}Cl + 2HN\begin{smallmatrix}CH_3\\CH_3\end{smallmatrix} \xrightarrow{\text{Ether}} (CH_3O)_2\overset{O}{\overset{\uparrow}{P}}\text{-}N\begin{smallmatrix}CH_3\\CH_3\end{smallmatrix}$$

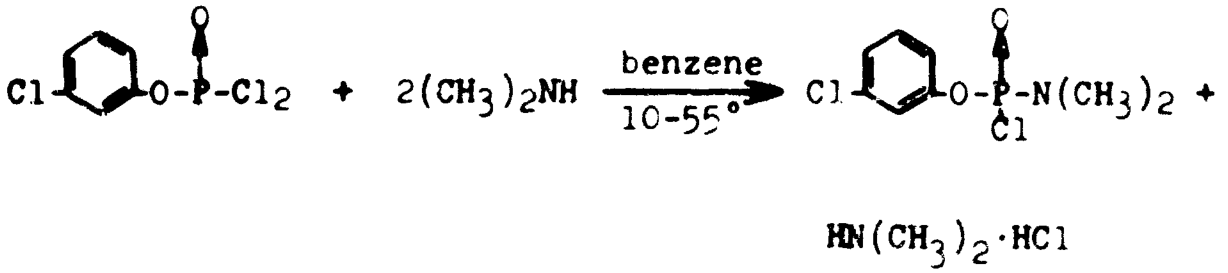
Methyl Tetramethylphosphorodiamidate (Cpd. 462) was prepared in an 83% yield according to the equation:



$$CH_3O\overset{O}{\overset{\uparrow}{P}}Cl_2 + 4(CH_3)_2NH \longrightarrow CH_3O\overset{O}{\overset{\uparrow}{P}}[N(CH_3)_2]_2$$

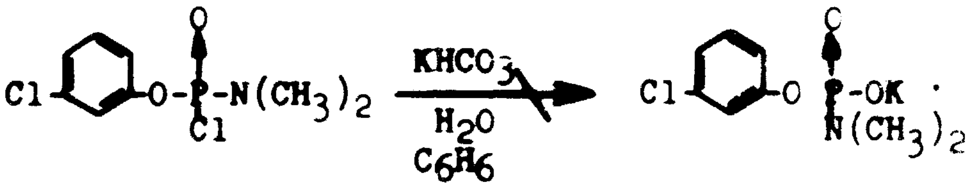
The water-soluble compound distilled at 99°/17 mm and the $n^{25}D$ was 1.4364.

m-Chlorophenyl Dimethylphosphoramidochloridate was synthesized by the reaction of one mole of m-chlorophenylphosphorodichloridate with two moles of dimethylamine, one mole of which was consumed as hydrogen chloride scavenger,



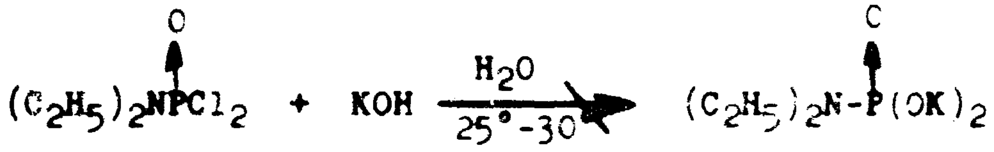
The product distilled at 124-129°/0.30-0.09 mm; $n^{25}D$ 1.5258, 71% yield.

Attempted hydrolysis of this dimethylphosphoramidochloridate to the potassium salt in water, using potassium bicarbonate, was unsuccessful.



NMR pattern and integrated area suggested that the sample was a mixture of the desired material and a contaminant containing no $(CH_3)_2N$- group.

Unsuccessful also was the hydrolysis of diethylphosphoramidic dichloride to the dipotassium N,N-diethylphosphoramidate



$$(C_2H_5)_2N\overset{O}{\overset{\uparrow}{P}}Cl_2 + KOH \xrightarrow[25°-30°]{H_2O} (C_2H_5)_2N-\overset{O}{\overset{\uparrow}{P}}(OK)_2$$

The molecule hydrolyzed completely.

VI. FUTURE PLANS

1. Determine the optimum compound as a pour point depressant in the class of phosphates and phosphonates.

2. Determine the compatibility of these two classes with paint.

3. Determine compatibility of the pour point depressant with sea water from a precipitation standpoint.

4. Give consideration to non-phosphorus halogenated organics.

5. Investigate thickeners.