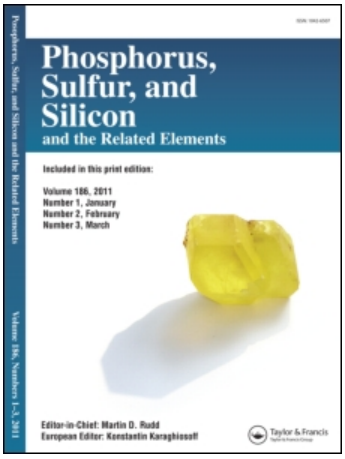


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

SYNTHESIS AND MASS SPECTRA OF FIVE-MEMBERED HETEROCYCLES CONTAINING PHOSPHORUS, OXYGEN, AND SULFUR

Byeong Hyo Kim[a]; Dong Young Oh[a]
[a] Department of Chemistry, Korea Advanced Institute of Science and Technology, Seoul, Korea

To cite this Article Kim, Byeong Hyo and Oh, Dong Young(1982) 'SYNTHESIS AND MASS SPECTRA OF FIVE-MEMBERED HETEROCYCLES CONTAINING PHOSPHORUS, OXYGEN, AND SULFUR', Phosphorus, Sulfur, and Silicon and the Related Elements, 13: 3, 337 — 344
To link to this Article: DOI: 10.1080/03086648208081194
URL: http://dx.doi.org/10.1080/03086648208081194

Phosphorus and Sulfur, 1982, Vol. 13, pp. 337–344
0308-664X/82/1303-0337/$06.50/0

SYNTHESIS AND MASS SPECTRA OF FIVE-MEMBERED HETEROCYCLES CONTAINING PHOSPHORUS, OXYGEN, AND SULFUR

BYEONG HYO KIM and DONG YOUNG OH*

Department of Chemistry, Korea Advanced Institute of Science and Technology, Seoul 131, Korea

(*Received February 9, 1982; in final form May 4, 1982*)

Isopropylphosphonic and isopropylphosphonothioic dichlorides were esterified with ethylene glycol, mercaptoethanol, 1,2,-ethanedithiol, 2,3-buthanediol, propylene glycol, and styrene glycol, to their corresponding phosphorus-containing heterocycles, which were new tetravalent phospholanes. The structure of each heterocycle was characterized by its IR, ^{1}H NMR, and electron-impact mass spectrometry. The mass spectra of synthesized phospholanes were studied in detail. The molecular ions were observed in variable abundance depending on the heteroatom of the ring. The base peaks of the spectra of phospholane-2-oxides usually arise by cleavage of the P—C bond and phospholane-2-sulfides generally arise by the loss of $C_3H_7S\cdot$. Plausible fragmentation pathways are described.

INTRODUCTION

Cyclic compounds in which an atom of phosphorus forms a part of the ring are of obvious interest in studies of physico-chemical properties of compounds where the heteroatom of the ring is varied. Edmundson and Lambie have reported some cyclic organophosphorus compounds as possible pesticides.[2,3] We have synthesized several tetravalent phospholanes using trichloromethylphosphonic dichloride.[4,5]

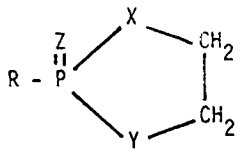
To extend the study of tetravalent phospholanes, another type of new phospholane was synthesized, and it was found that mass spectrometry is a useful method for the characterization of those compounds even though the mass spectra of organophosphorus compounds have not been important probably due to their relatively high reactivity and ready conversion in the spectrometer to other molecules which may be easily identified such as esters.

RESULTS AND DISCUSSION

Isopropylphosphonic and isopropylphosphonothioic dichlorides were prepared by known methods.[6–8] They were combined with ethylene glycol, mercaptoethanol, 1,2-ethanedithiol, 2,3-buthanediol, propylene glycol, and styrene glycol, to give tetravalent phospholanes.

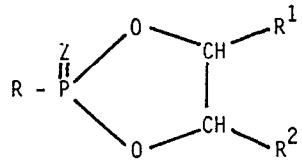
Although largely unevaluated in terms of analytical potential, phosphorus is known to form cyclic derivatives with bifunctional groups. The cyclization reaction is considered a stepwise esterification in all cases, though intermediates are not isolated.

* Author to whom all correspondence should be addressed.



R : i-Pr

I. X = Y = Z = O
II. X = Y = O, Z = S
III. X = S, Y = Z = O
IV. X = Z = S, Z = O
V. X = Y = S, Z = O
VI. X = Y = Z = S

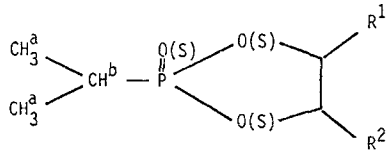


R : i-Pr

VII. Z = O, R^1 = R^2 = Me
VIII. Z = S, R^1 = R^2 = Me
IX. Z = O, R^1 = H, R^2 = Me
X. Z = S, R^1 = H, R^2 = Me
XI. Z = O, R^1 = H, R^2 = Ph
XII. Z = S, R^1 = H, R^2 = Ph

The reaction of isopropylphosphonic dichloride with 1,2-ethanedithiol produced 2-isopropyl-1,3,2-dithiaphospholane-2-oxide (54%) and an unexpected side product 2-isopropyl-1,3,2-dithiaphospholane-2-sulfide (13%). The formation of the 2-sulfide is difficult to explain even though it is assumed that some sulfur in 1,2-ethanedithiol is exchanged for oxygen in the 2-oxide.

Infrared spectrometry is generally applicable to the identification of phosphorus compounds by the P=O absorption of 1200–1250 cm^{-1}, the absorptions of P=S (band I) and P=S (band II) of 700–750 and 615–650 cm^{-1}, respectively, the P—O—(C) stretching, in the region of 1000–1070 cm^{-1}, and the characteristic single sharp band of tri- or tetravalent phospholanes and phosphorinanes at 915–950 cm^{-1}. The IR data are listed in Table I.



In the ^1H NMR spectra, the protons of OCH in dioxaphospholanes or dioxaphosphorinanes exhibited signals at δ 4.5–5.6, which were heavily multiple peaks due to phosphorus couplings and vicinal couplings. The protons of SCH in phospholanes showed multiplets centered at δ 3.5–3.7. In other words, chemical shifts of the ring protons are mainly governed by the inductive effect of heteroatoms in the ring. J_{HH} values of ^aH and ^bH vicinal couplings are 7 Hz on all cases, and the ^aH proton coupled with the phosphorus nucleus with a coupling constant of 19–23 Hz.

Mass Spectrometry

The characterization of organophosphorus compounds by mass spectrometry has not been used widely because of their facile conversion in the instrument to other

TABLE I

Physical and IR spectral data of compounds

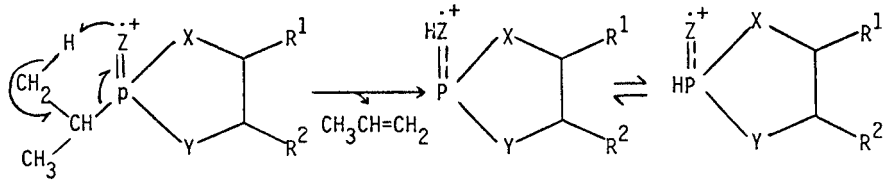
Compound	bp. (°C/torr)(mp.)	IR (cm^{-1}/KBr or NaCl) P=O	P=S	P—O—(C)
I. 2-R-1,3,2-dioxaphospholane-2-oxide	95–97/1.3	1250		1040
II. 2-R-1,3,2-dioxaphospholane-2-sulfide	55–56/1.2		I.750 II.650	1030
III. 2-R-1,3,2-oxathia-phospholane-2-oxide	99–101/1.2	1225		1010
IV. 2-R-1,3,2-oxathia-phospholane-2-sulfide	[a]110/2.0		I.710 II.630	1005
V. 2-R-1,3,2-dithiaphospholane-2-oxide	[a]120/1.4	1200		
VI. 2-R-1,3,2-dithiaphospholane-2-sulfide	[b](44–45)		I.700 II.615	
VII. 4,5-dimethyl-2-R-1,3,2-dioxaphospholane-2-oxide	82–84/2.2	1250		1050
VIII. 4,5-dimethyl-2-R-1,3,2-dioxaphospholane-2-sulfide	[a]120/1.5		I.750 II.650	1040
IX. 2-R-4-methyl-1,3,2-dioxaphospholane-2-oxide	77–78/1.1	1250		1015
X. 2-R-4-methyl-1,3,2-dioxaphospholane-2-sulfide	55–57/1.0		I.720 II.645	1040
XI. 2-R-4-phenyl-1,3,2-dioxaphospholane-2-oxide	102–105/0.6	1260		1020
XII. 2-R-4-phenyl-1,3,2-dioxaphospholane-2-sulfide	[a]90–95/0.7		I.730 II.635	1015

R; i-Pr.
[a] Kugelrohr microdistillation apparatus was used.
[b] Recrystallized from chloroform-pet. ether solvent.

structures, which may be easily identified such as esters. The mass spectra of alkyl- and arylphosphonic dichlorides and some phosphonates were examined by Griffiths and Tebby.[9,10] The characteristics of some phospholanes have been examined by Poole and by us.[4,5,11] It was recognized that electron-impact (EI) mass spectrometry was a useful method for the structural study of cyclic organophosphorus compounds.

The structures of all of the synthesized tetravalent phospholanes were conformed by EI mass spectra. A notable feature of the spectra was the prominent molecular ion observed in all cases, even though, in general, phosphorus compounds are not so observed. The molecular ion of sulfur-containing phospholanes is more abundant than that of oxygen-containing phospholanes. This difference arises from the low ionization potentials of sulfur compounds, which are approximately 1 eV below those of the corresponding oxygen compounds. This makes possible the formation of much lower-energy molecular ions, substantially increasing the ion-current intensity of the molecular ion. The molecular ions of oxygen-containing phospholanes have a 5.3 to 26.0% abundance and those of sulfur-containing phospholanes 23.3 to 61.6%.

As a general observation, all the compounds tend to show an initial loss of propylene from the molecular ion via a McLafferty-type rearrangement which gives rise to abundant peaks, indicating that the cleavage of the P—C bond is a facile process. In case of phospholane-2-oxides, this cleavage gave the base peak except with 2-isopropyl-1,3,2-oxathiaphospholane-2-oxides (III) and 2-isopropyl-4-phenyl-1,3,2-dioxaphospholane-2-oxide (XI). In all cases of phospholane-2-sulfides, except 2-isopropyl-4-phenyl-1,3,2-dioxaphospholane-2-sulfide (XII), their base peaks were observed when the $C_3H_7S\cdot$ radical fragmented from the molecular ion. The base peak of III, observed at m/e 60, assumed the form of ethylene sulfide cation, and that of XI was observed at m/e 153 (Ph—CH=OPO+) and XII at m/e 104 ($Ph\cdot \pm CH_2$).



The dominating ions in the mass spectra of phospholanes (I–VI) are presented in Table II and their fragmentations are considered in detail. The molecular ion of III and 2-isopropyl-1,3,2-oxathiaphospholane-2-sulfide (IV) apparently undergo fragmentation in a similar pattern. An initial loss of propylene from the molecular ion produced $C_2H_5O_2PS^{+\cdot}$ at m/e 124 (III) and $C_2H_5OPS_2^{+\cdot}$ at m/e 140 (IV), respectively; then the elimination of $\cdot ZH$ [Z=O (III), Z=S (IV)] from each species gave the fragment ion $C_2H_4OPS^+$ at m/e 107 in both cases. The loss of $\cdot PO$ from $C_2H_4OPS^+$ gave $C_2H_4S^{+\cdot}$ at m/e 60, which is the base peak of III and which showed a low intensity in the case of IV. It is the dominant decomposition process for III and IV in other decomposition experiments with I, 2-isopropyl-1,3,2-dioxaphospholane-2-sulfide (II), V, and 2-isopropyl-1,3,2-dithiaphospholane-2-sulfide (VI), their fragmentations also resembled those of III and IV. The plausible decomposition pathways of III and IV are shown below in Scheme 1.

Scheme 2 shows proposed decomposition pathways of compounds VII–XII with ions derived from the dioxaphospholane derivatives on electron impact. In each case, P—C bond cleavage is also a facile process. Thus, the elimination of propylene gives five-membered cyclic ion (A) (m/e 136 (VII), 152 (VIII), 122 (IX), 138 (X), and the loss of $\cdot ZH$ from (A) gives ion (B) [m/e 119 (VII, VIII), 105 (IX, X)]. The ionized five-membered cyclic group (B) decomposes with two main ring-fragmentation patterns. In other words, the loss of $\cdot PO_2H$ and $R^1CH^{+\cdot}$ gives rise to $R^{1+}C{=}CHR^2$ [m/e 55 (VII, VIII), 41 (IX, X)] and R^2—CH=OPO+ [m/e 91 (VII, VIII, IX, X)], respectively. All above ions are shown in Table III.

The decomposition pathways of XI and XII are similar to those of VII–X. However, XI and XII exhibit somewhat different abundances and fragments because of the effect of the phenyl substituent on the fragmentation. The dominating ions of XI and XII are presented in Table IV.

Our previous paper,[4,5] the ring contraction of 2-trichloromethyl phospholane derivatives was observed, but the 2-isopropylphospholane derivatives do not show the ring contraction. It is assumed that this difference is produced by the 2-substituent (trichloromethyl or isopropyl).

The above results suggest that mass spectrometry is a convenient method for the characterization of phosphorus-containing heterocyclic molecules obtained from isopropylphosphonic and isopropylphosphonothionic dichlorides.

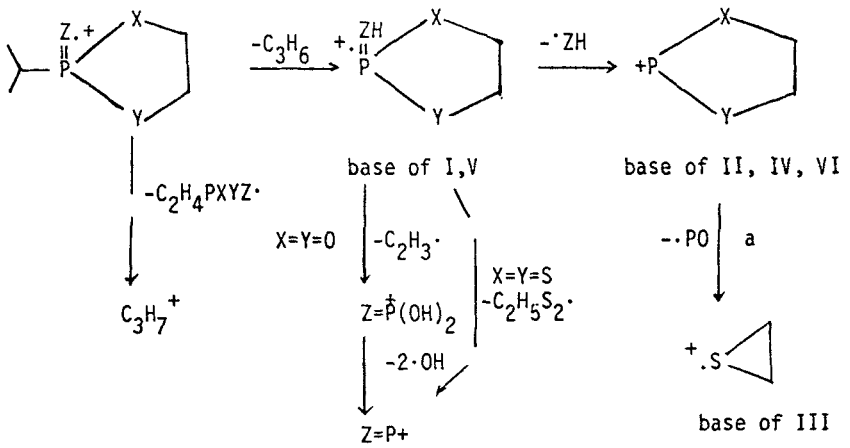
TABLE II

Mass spectral data of compounds I–VI

Compound	Relative abundance (%)							Other abundant cations
	$M^{\ddagger}$	$C_3H_7^+$	$M-[C_3H_6]^{\ddagger}$	$M-[C_3H_7S]^{\ddagger}$	$P(OH)_2^+$	$P^+=O$	$P^+=S$	
I	5.6	24.6	100.0	—	5.2	15.2	—	
II	36.3	11.4	24.9	100.0	8.4	—	7.0	
III	61.6	34.2	98.9	—	7.0	31.6	—	m/e 60 (100.0)
IV	49.1	8.8	43.1	100.0	—	—	12.3	
V	23.3	41.5	100.0	—	—	33.3	—	
VI	46.7	16.7	57.5	100.0	—	—	24.6	

EXPERIMENTAL

The ^1H nmr spectra were determined in $CDCl_3$ solutions (TMS as internal standard) using a Varian T-60A spectrometer. Chemical shifts are described in ppm. Melting points were obtained with a Thomas Hoover capillary melting point apparatus and are uncorrected. Mass spectra were obtained using the Hewlett-Packard HP5985A instrument of 70 eV electrons and with sample introduction via direct inlet system at room temperature, and ion source temperature 200–250°C. Infrared spectra were determined using a Perkin-Elmer Model 267 instrument. The following descriptive abbreviations are used; vs = very strong, s = strong, m = medium, w = weak.

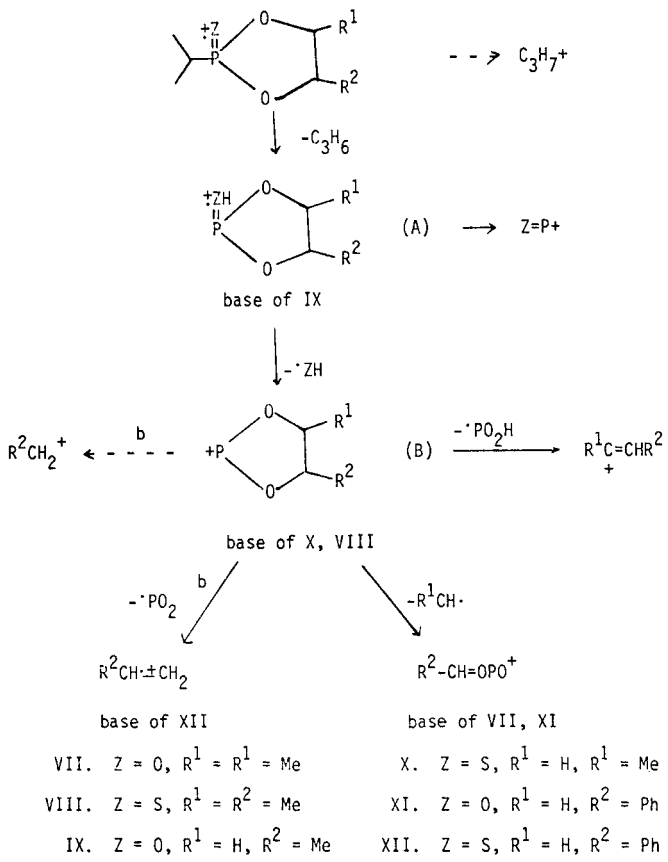


I. X = Y = Z = O III. X = S, Y = Z = O V. X = Y = S, Z = O

II. X = Y = O, Z = S IV. X = Z = S, Y = O VI. X = Y = Z = S

a This fragmentation appears at III and IV.

SCHEME 1



b This fragmentation appears at XI and XII.

SCHEME 2

All the chemicals used were of reagent grade and purified prior to use, if necessary. Wacogel Q-23 was used for column chromatography. Isopropylphosphonic dichloride and isopropylphosphonothioic dichloride were prepared by known methods.[6-8]

2-isopropyl-1,3,2-dioxaphospholane-2-oxide (I). To a solution of isopropylphosphonic dichloride (0.015 mole, 2.42 g) and ethylene glycol (0.015 mole, 0.93 g) in dry ethyl ether (30 ml), a solution of Et_3N in dry

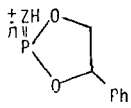
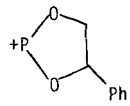
TABLE III

Mass spectral data of compounds VII–X

	m/e (%)					
Cpd.	A	B	R^2—CH=OPO$^+$	$R^2C^+=CHR^2$	+P=Z	$C_3H_7^+$
VII	136(60.9)	119(28.0)	91(100.0)	55(28.2)	47(20.2)	43(59.5)
VIII	142(63.8)	119(100.0)	91(25.5)	55(25.2)	63(13.2)	43(20.2)
IX	122(100.0)	105(7.1)	91(21.5)	41(10.6)	47(5.0)	43(9.4)
X	138(27.0)	105(100.0)	91(0.5)	41(7.8)	63(4.9)	43(3.5)

TABLE IV

Mass spectral data of XI and XII

Fragment	m/e (%) Compound XI	Compound XII
	184(2.5)	200(3.9)
	167(1.4)	167(9.2)
Ph—CH=OPO$^+$	153(100.0)	153(14.6)
Ph. ± CH_2	104(42.3)	104(100)
$PhCH_2^+$	91(32.4)	91(30.5)
Ph^+	77(29.6)	77(17.1)
$Z{=}P^+$	47(2.6)	47(1.4)
$C_3H_7^+$	43(6.6)	43(3.7)

ethyl ether (20 ml) was added dropwise over a period of approximately 30 min. in an ice-bath. After the addition of Et_3N was completed, the mixture was stirred for an added 2 hours. Triethylammonium chloride was removed by filtration and the filtrate was concentrated on a rotary evaporator. The concentrated filtrate was distilled and treated by column chromatography. The yield was 1.8 g (80%). IR (NaCl): 2995 cm^{-1} m, 1465 m, 1250 s, 1040 vs, 930 s, 890 m, 815 s, 690 m. NMR ($CDCl_3$); δ 1.3 (dd, 6H, CH_3, J_{PH} = 19 Hz, J_{HH} = 7 Hz), 2.2 (m, 1H, CH), 4.5 (m, 4H, CH_2). Mass spec.; m/e (rel. intens. %) *150*(5.6, M^+), 135(33.3), 109(15.9), 108(100.0), 81(15.9), 65(5.2), 47(15.7), 43(22.8), 41(24.6).

2-isopropyl-1,3,2-dioxaphospholane-2-sulfide (II). The yield was 1.3 g (52%). IR (NaCl); 2990 cm^{-1}, 1465 m, 1030 vs, 925 s, 850 s, 750 m, 728 m, 650 w. NMR ($CDCl_3$); δ 1.4 (dd, 6H, CH_3, J_{PH} = 21 Hz, J_{HH} = 7 Hz), 2.3 (m, 1H, CH), 4.5 (m, 4H, CH_2). Mass spec.; *166*(36.3, M^+), 124(24.9), 91(100.0), 65(8.4), 63(7.0), 43(11.4), 41(9.5).

2-isopropyl-1,3,2-oxathiaphospholane-2-oxide (III). The yield was 1.8 g (72%). IR (NaCl); 2985 cm^{-1} m, 1465 m, 1225 s, 1045 m, 1010 m, 945 m, 790 m, 680 m. NMR ($CDCl_3$); δ 1.5 (dd, 6H, CH_3, J_{PH} = 21 Hz), 2.5 (m, 1H, CH), 3.5 (m, 2H, SCH_2), 4.5 (m, 2H, OCH_2). Mass spec.; *166*(61.6, M^+), 124(98.9), 65(7.0), 60(100.0), 59(40.0), 47(31.6), 43(34.2), 41(40.4).

2-isopropyl-1,3,2-oxathiaphospholane-2-sulfide (IV). The yield was 0.9 g (36%). IR (NaCl); 2980 cm^{-1} m, 1465 m, 1275 m, 1005 s, 940 m, 855 m , 790 s, 650 m, 630 s. NMR ($CDCl_3$); δ 1.5 (dd, 6H, CH_3, J_{PH} = 22 Hz, J_{HH} = 7 Hz), 2.5 (m, 1H, CH), 3.5 (m, 2H, SCH_2), 4.6 (m, 2H, OCH_2). Mass spec.; *182*(49.1, M^+), 140(43.1), 107(100.0), 63(12.3), 43(8.8), 41(11.5).

2-isopropyl-1,3,2-dithiaphospholane-2-oxide (V). The yield was 1.5 g (54%) with the side product (VI), 0.4 g (13%). IR (NaCl); 2980 cm^{-1} m, 1465 m, 1200 s, 940 m, 880 m, 670 s. NMR ($CDCl_3$); δ 1.5 (dd, 6H, CH_3, J_{PH} = 21 Hz, J_{HH} = 7 Hz), 2.5 (m, 1H, CH), 3.6 (m, 4H, CH_2). Mass spec.; *182*(23.3, M^+), 140(100.0), 47(33.3), 43(41.5), 41(36.0).

2-isopropyl-1,3,2-dithiaphospholane-2-sulfide (VI). The resulting solid was recrystallized from chloroform and petroleum ether solvent system. The yield was 1.0 g (33%). IR (KBr); 2950 cm^{-1} m, 1465 m, 1420 m, 1280 m, 1245 m, 1035 m, 940 s, 700 s, 615 s. NMR ($CDCl_3$); δ 1.5 (dd, 6H, CH_3, J_{PH} = 23 Hz, J_{HH} = 7 Hz), 2.6 (m, 1H, CH), 3.7 (m, 4H, CH_2). Mass spec.; *198*(46.7, M^+), 156(57.5), 123(100.0), 63(24.6), 43(16.7), 41(18.2).

4,5-dimethyl-2-isopropyl-1,3,2-dioxaphospholane-2-oxide (VII). The yield was 1.8 g (67%). IR (NaCl); 2970 cm^{-1} m, 1460 m, 1380 m, 1250 s, 1050 s, 950 vs, 890 m, 820 m, 695 m. NMR ($CDCl_3$); δ 1.4 (m, 12H, CH_3), 2.2 (m, 1H, CH), 4.5 (m, 2H, CH). Mass spec.; *178*(12.6, M^+), 136(60.9), 125(89.5), 119(28.0), 91(100.0), 65(35.1), 55(28.2), 43(59.5), 41(48.9).

4,5-dimethyl-2-isopropyl-1,3,2-dioxaphospholane-2-sulfide (VIII). The yield was 0.9 g (31%). IR (NaCl); 2980 cm^{-1} m, 1470 m, 1385 m, 1040 s, 940 s, 800 m, 750 m, 650 m. NMR ($CDCl_3$); δ 1.6 (m, 12H, CH_3), 2.8 (m, 1H, CH), 4.5 (m, 2H, CH). Mass spec.; *194*(49.0, M^+), 179(12.8), 142(63.8), 119(100.0), 115(44.6), 91(22.5), 63(13.2), 55(25.5), 43(20.2), 41(19.1).

2-isopropyl-4-methyl-1,3,2-dioxaphospholane-2-oxide (IX). The yield was 1.5 g (61%). IR (NaCl); 2950 cm^{-1} m, 1470 m, 1390 m, 1250 s, 1015 vs, 945 m, 825 s, 695 m, NMR ($CDCl_3$); δ 1.5 (m, 9H, CH_3), 2.15 (m, 1H, PCH), 4.15 (m, 3H, OCH_2, OCH). Mass spec.; *164*(5.3, M^+), 149(20.2), 122(100.0), 96(37.9), 91(21.5), 65(11.5), 47(5.0), 43(9.4).

2-isopropyl-4-methyl-1,3,2-dioxaphospholane-2-sulfide (X). The yield was 0.9 g (33%). IR (NaCl); 2900 cm^{-1} m, 1460 m, 1385 m, 1005 s, 945 m, 910 m, 870 m, 800 m, 755 m, 720 m, 645 m. NMR ($CDCl_3$); δ 1.5 (m, 9H, CH_3), 2.2 (m, 1H, PCH), 4.3 (m, 3H, OCH_2, OCH). Mass spec.; *180*(34.5 M^+), 138(27.0), 105(100.0), 65(10.8), 63(4.9), 43(3.5), 41(7.8).

2-isopropyl-4-phenyl-1,3,2-dioxaphospholane-2-oxide (XI). Ethyl ether and benzene (1:1 v/v) were used for solvent. The yield was 1.4 g (41%). IR (NaCl); 3040 cm^{-1} w, 2960 m, 1460 m, 1260 s, 1020 vs, 930 s, 835 s, 755 m, 700 m. NMR ($CDCl_3$); δ 1.4 (m, 6H, CH_3), 2.25 (m, 1H, PCH), 4.4 (m, 2H, OCH_2), 5.5 (m, 1H, OCH), 7.35 (m, 5H, C_6H_5). Mass spec.; *226*(26.0, M^+), 184(2.5), 167(1.4), 153(100.0), 105(52.3), 104(42.3), 91(32.4), 77(29.6), 65(9.7), 47(2.6), 43(6.6), 41(5.5).

2-isopropyl-4-phenyl-1,3,2-dioxaphospholane-2-sulfide (XII). Ethyl ether and benzene (1:1 v/v) were used for solvent. The yield was 1.3 g (36%). IR (NaCl); 3035 cm^{-1}, w, 2965 m, 1460 m, 1250 s, 1015 s, 930 m, 855 m, 750 m, 730 m, 700 m, 635 m. NMR ($CDCl_3$); 1.4 (m, 6H, CH_3), 2.4 (m, 1H, PCH), 4.45 (m, 2H, OCH_2), 5.5 (m, 1H, OCH), 7.4 (m, 5H, C_6H_5). Mass spec.; *242*(28.7, M^+), 200(3.9), 167(9.2), 153(14.6), 120(23.5), 104(100.0), 91(30.5), 77(17.1), 65(6.0), 63(5.3), 43(3.7).

REFERENCES

1. (a) Guy Lacroix, Jean C. Debourge, and Jacques Ducret, *Ger. Offen.*, 2,602,804 (1976). (b) Richard Sallmann, *Swiss Patent*, 323,228 (1957). (c) Gerhard Schrader, *Ger.*, 1,104,520 (1963).
2. R. S. Edmundson and A. J. Lambie, *J. Chem. Soc., C*, 1997 (1966).
3. R. S. Edmundson and A. J. Lambie, *J. Chem. Soc., C*, 2001 (1966).
4. D. Y. Oh and B. M. Kwon, *Bull. Kor. Chem. Soc.*, **1**, 58 (1980).
5. D. Y. Oh and B. M. Kwon, *Phosphorus and Sulfur*, **11**, 177 (1981).
6. A. M. Kinnear and E. A. Perren, *J. Chem. Soc.*, 3437 (1952).
7. L. Maier, *Helv. Chim. Acta.*, **48**, 133 (1965).
8. Mahabir P. Kausik and R. Viadyanathaswamy, *J. Org. Chem.*, **45**, 2270 (1980).
9. W. Richard Griffiths and John C. Tebby, *Phosphorus and Sulfur*, **4**, 341 (1978).
10. W. Richard Griffiths and John C. Tebby, *Phosphorus and Sulfur*, **5**, 101 (1978).
11. C. F. Poole, S. Singhawangcha, L-E. Chen Hu, and A. Zlatkis, *J. Chromatogr.*, **178**, 495 (1979).