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Synthesis of Novel Chiral 2-Oxo- and 2-Thio-1,3,2-oxazaphospholidines via Asymmetric Cyclization of L-methionol with (Thio)Phosphoryl Dichlorides

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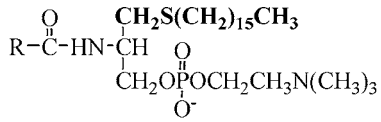
ABSTRACT: *In order to search for novel antitumor and antiviral agents with high activity and low toxicity, a series of chiral 2-thio(oxo)-1,3,2-oxazaphospholidines were synthesized via the reaction of L-methionol with all kinds of (thio)phosphoryl dichlorides in THF in the presence of triethylamine at room temperature. The structures of all of the new compounds were confirmed by elemental analyses, ^{1}H, ^{31}P NMR, and IR spectra.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:33–38, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20060

INTRODUCTION

Organophosphorus compounds are ubiquitous in nature, and they have broad applications in the fields of agriculture and medicine [1–4]. It has been found that 4-*H*-1,3,2-oxazaphosphoridines derived from 2-amino acids possess fairly high insecticidal activity [5,6]. 4-*H*-1,3,2-Benzoxaphosphorins also show some insecticidal activity [7]. Synthesis and structural assignments of 1,3,2-oxazaphospholidines derived from (−)-ephedrine have been reported [8]. Incorporating amino acid esters in some drugs lowered their toxicity and enhanced their cellular uptake [9], which can be attributed to producing nontoxic products upon in vivo hydrolysis of these drugs. Coupling this information, Ali and Mohamed synthesized a series of analogues from L-cysteine [10]. Then, He et al. also reported the synthesis of chiral 2-oxo- and 2-thio-1,3,2-oxazaphospholidines via the asymmetric cyclization of L-serinoates with (thio) phosphoryl dichlorides [11,12].

Despite of the well-recognized biological importance and synthetic methods of 2-oxo- and 2-thio-1,3,2-oxazaphospholidines, relatively a little progress has been made toward the sulfur-containing phosphorus heterocycles. Furthermore, because of recent developments in the area of antitumor of active phospholipids, demonstrating that introduction of an *sn*-1 thioether substituent in place of the *O*-alkyl group significantly improves the chemotherapeutic properties of these derivatives [13,14], Bhatia et al. [15] synthesized thioether analogues of phospholipids.



(R=CH_3 , $CH_3(CH_2)_{14}$, $CH_3(CH_2)_{13}O$)

Guided by these features, we also intended to induce the thioether group into the 2-oxo- and 2-thio-1,3,2-oxazaphospholidines. Herein, some new

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results have been obtained from the asymmetric cyclizations of various (thio) phosphoryl dichlorides using L-methionine as the starting material. Products **1–9** were obtained with diastereomeric preference (Scheme 1). Their de% values were determined on the basis of ^{31}P NMR data. Compounds **7** were successfully isolated as pure diastereomers by column chromatography. Other compounds were diastereomeric mixtures.

Compounds **1–9** were synthesized by one step, but compounds **10–12** by two steps in relatively low yields (Scheme 2). Nevertheless, because of the instability of the intermediate 2-chloro-2-thio-1,3,2-oxazaphospholidine, the reaction was traced by ^{31}P NMR spectra that showed only two peaks in the low field. Then the intermediate was used directly in the next step.

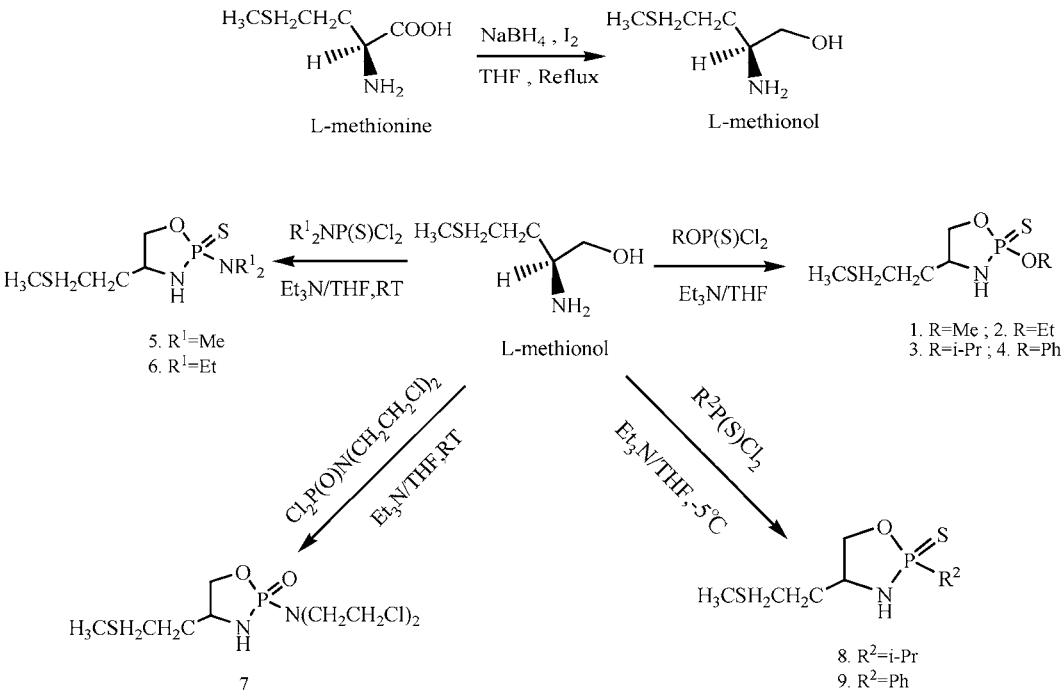
RESULTS AND DISCUSSION

All of the new compounds were characterized by spectral data and elemental analyses. The physical constants of the title compounds (**1–12**) are listed in Table 1, and data of the ^{1}H NMR, ^{31}P NMR, and IR spectra are listed in Tables 2 and 3. In Scheme 1, phenyl (alkyl) thiophosphonyl dichlorides reacted with L-methionol faster than do other thiophosphoryl dichlorides, and this reaction was performed in an ice-salt bath (−8–−5°). It was not necessary to be warmed to room temperature. The product **9** (R=Ph), which did not need separation by column chromatography, was redissolved in ethyl acetate and kept in a refrigerator. It caused precipitation of a great amount of white solid.

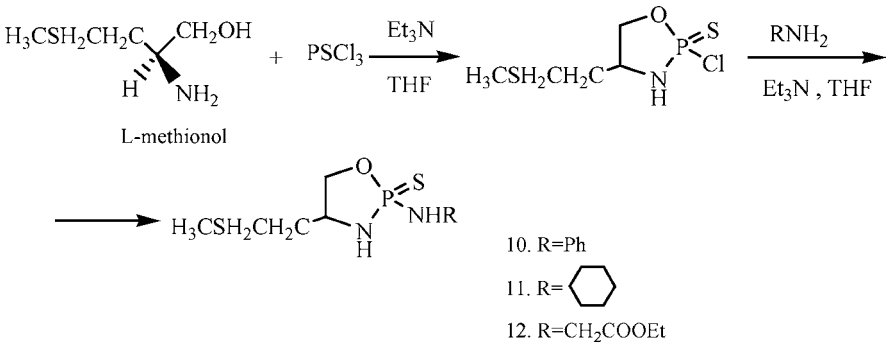
2-Oxo (thio)-1,3,2-oxazaphosphorinane derivatives are not stable. Their P—N bonds are easier to cleavage by opening the cycle, they were purified by passing through chromatography column on silica gel, and TLC showed that there were several new spots. Thus it caused that the yields were relatively low, particularly in multistep reactions.

The methylene protons of the cyclic ring are diastereotopic, and hence they are chemically nonequivalent. In their proton NMR spectra, they split each other and are split further by a neighboring methyne proton and the phosphorus atom to give a multiplet at 3.7–4.5 ppm. The methyne proton is also split by each of the methylene protons, by NH and the phosphorus atom to give a multiplet at 3.7–4.5 ppm. CH_3S group appears as a single peak. SCH_2CH_2 in which α-methylene protons are split by a neighboring β-methylene to give a multiplet at ∼2.48 ppm, and β-methylene appears as a multiplet. Detailed assignments are given in Table 2.

The ^{31}P NMR signals appeared in the range of 27.65–121.12 ppm. Moreover, there were two peaks in the ^{31}P NMR spectra, as verified that the title compounds were diastereomeric mixtures.



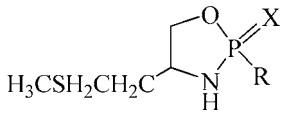
SCHEME 1



SCHEME 2

Thompson et al. [16] have described the cyclization of methyl *N*-benzyl L-serinonate with phosphorus oxychloride to provide diastereomeric 2-chloro-1,3,2-oxazaphospholidin-2-ones, which were isolated in crude form, followed by reaction with the appropriate alcohol or phenol to give the chiral cyclic phosphoramidates in a near 1:1 diastereomeric ratio. In such cyclization reaction, no asymmertric induction effect had previously been observed. In our study, L-methionol did cyclize directly with an appropriate thiophosphoryl dichloride with some degree of diastereomeric preference to afford the corresponding chiral cyclization products **1–9**. In the cyclization, the de% value of the product depends

TABLE 1 Experimental Data of Compounds **1–12**

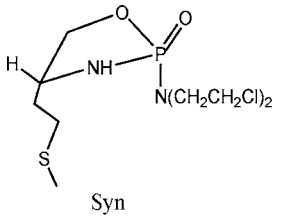


						Found/(Calcd)%		
No.	*R*	*X*	*Molecular Formula*	*Yield (%)*	*mp (°)*	*C*	*H*	*N*
1	OCH_3	S	$C_6H_{14}PO_2NS_2$	61.24	Slight yellow thick liquid	31.72 (32.16)	6.17 (5.96)	6.17 (5.68)
2	OC_2H_5	S	$C_7H_{16}PO_2NS_2$	50.52	Slight yellow thick liquid	34.85 (34.79)	6.63 (6.14)	5.80 (5.82)
3	OPr-i	S	$C_8H_{18}PO_2NS_2$	79.12	Slight yellow thick liquid	37.60 (37.70)	7.06 (6.66)	5.48 (5.52)
4	OPh	S	$C_{11}H_{16}PO_2NS_2$	49.10	Slight yellow thick liquid	45.64 (45.12)	4.84 (4.94)	5.54 (5.54)
5	Me_2N	S	$C_7H_{17}PON_2S_2$	60.78	Colorless thick liquid	35.00 (35.05)	7.08 (7.18)	11.67 (11.70)
6	Et_2N	S	$C_9H_{21}PON_2S_2$	55.92	Colorless thick liquid	40.26 (40.36)	7.83 (7.89)	10.44 (10.55)
7a	$N(CH_2CH_2Cl)_2$	O	$C_9H_{19}PO_2N_2SCl_2$	22.50	Slight yellow thick liquid	33.64 (33.83)	5.92 (6.46)	8.71 (8.66)
7b	$N(CH_2CH_2Cl)_2$	O	$C_9H_{19}PO_2N_2SCl_2$	29.38	White solid mp 102–104	33.64 (33.46)	5.92 (6.40)	8.71 (8.26)
8	i-Pr	S	$C_8H_{18}PONS_2$	52.68	Slight yellow thick liquid	40.13 (40.15)	7.53 (7.54)	5.86 (6.01)
9	Ph	S	$C_{11}H_{16}PONS_2$	82.00	White solid mp 83–89	48.31 (48.15)	5.85 (6.05)	5.12 (5.20)
10	PhNH	S	$C_{11}H_{17}PON_2S_2$	38.19	Slight yellow thick liquid	44.86 (44.87)	7.82 (7.61)	9.52 (9.40)
11		S	$C_{11}H_{23}PON_2S_2$	40.14	Colorless wax solid	45.78 (45.75)	5.90 (5.99)	9.71 (9.75)
12	$NHCH_2COOC_2H_5$	S	$C_9H_{19}PO_3N_2S_2$	36.24	Slight yellow thick liquid	36.42 (36.22)	6.68 (6.37)	9.43 (9.39)

TABLE 2 ^{1}H NMR Data for Compounds **1–12**

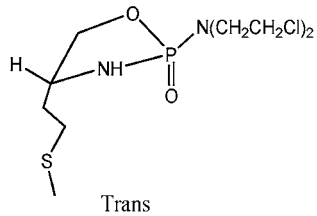
Compound	δ_H *(ppm)*
1	1.69–1.91 (m,2H,$SCH_2C\mathbf{H}_2$) ;2.05 (s,3H,$C\mathbf{H}_3S$); 2.46–2.56 (t,2H,$SC\mathbf{H}_2$); 3.69 (s,3H,$OC\mathbf{H}_3$); 3.73–4.44 (b,3H,cycle)
2	1.20–1.27 (t,3H,$OCH_2C\mathbf{H}_3$); 1.67–1.89 (m,2H,$SCH_2C\mathbf{H}_2$); 2.02 (s,3H,$C\mathbf{H}_3S$); 2.45–2.55 (t,2H,$SC\mathbf{H}_2$); 3.62–3.91 (m,2H, $OC\mathbf{H}_2CH_3$); 3.94–4.43 (b,3H,cycle)
3	1.18–1.29 (d,6H,$CH(C\mathbf{H}_3)_2$); 1.74–1.90 (m,2H,$SCH_2C\mathbf{H}_2$); 2.08 (s,3H,$C\mathbf{H}_3S$); 2.49–2.59 (t,2H,$SC\mathbf{H}_2$); 3.83–4.45 (m,3H,cycle); 4.69–4.75 (m,1H,$C\mathbf{H}(CH_3)_2$)
4	1.59–1.92 (m,2H,$SCH_2C\mathbf{H}_2$); 2.14 (s,3H,$C\mathbf{H}_3S$); 2.45–2.57 (t,2H,$SC\mathbf{H}_2$); 3.81–4.08 (b,3H,cycle); 7.13–7.36 (m,5H,**Ph**O)
5	1.72–1.82 (m,2H,$SCH_2C\mathbf{H}_2$); 2.09 (s,3H,$C\mathbf{H}_3S$); 2.48–2.60 (t,2H,$SC\mathbf{H}_2$); 2.74–2.82 (s,6H,$N(C\mathbf{H}_3)_2$); 3.00 (s,1H,N**H**); 3.70–4.42 (m,3H,cycle)
6	1.03–1.23 (t,6H,$N(CH_2C\mathbf{H}_3)_2$); 1.70–1.80 (m,2H,$SCH_2C\mathbf{H}_2$); 2.07 (s,3H,$C\mathbf{H}_3S$); 2.46–2.55 (t,2H,$SC\mathbf{H}_2$); 2.98 (s,1H,NH); 3.04–3.36 (m,4H,$N(C\mathbf{H}_2CH_3)_2$); 3.70–4.43 (b,3H,cycle)
7a	1.80–1.95 (m,2H,$SCH_2C\mathbf{H}_2$); 2.09 (s,3H,$C\mathbf{H}_3S$); 2.53–2.60 (t,2H,$SC\mathbf{H}_2$); 3.15–3.64 (m,8H,$N(C\mathbf{H}_2C\mathbf{H}_2Cl)_2$); 3.75–4.33(m,3H,cycle)
7b	1.80 (m,2H,$SCH_2C\mathbf{H}_2$); 2.10 (s,3H,$C\mathbf{H}_3S$); 2.55 (t,2H,$SC\mathbf{H}_2$); 3.43–3.61 (m,8H,$C\mathbf{H}_2C\mathbf{H}_2Cl)_2$); 3.74–4.52 (m,3H,cycle)
8	1.11–1.16 (d,6H,$CH(C\mathbf{H}_3)_2$); 1.70–1.80 (m,2H,$SCH_2C\mathbf{H}_2$); 1.81–2.01 (m,1H,$C\mathbf{H}(CH_3)_2$); 2.07 (s,3H,$C\mathbf{H}_3S$); 2.46–2.53 (t,2H,$SC\mathbf{H}_2$); 3.0 (s,1H,N**H**); 3.87–4.49 (m,3H,cycle)
9	1.92–2.04 (m,2H,$SCH_2C\mathbf{H}_2$); 2.12 (s,3H,$C\mathbf{H}_3S$); 2.57–2.65 (m,2H,$SC\mathbf{H}_2$); 3.38 (s,1H,N**H**); 3.79–4.48 (b,3H,cycle); 7.38–7.49 (m,3H,**Ph**); 7.83–7.95 (m,2H,**Ph**)
10	1.74–1.83 (m,2H,$SCH_2C\mathbf{H}_2$); 2.04 (s,3H,$C\mathbf{H}_3S$); 2.47–2.54 (m,2H,$SC\mathbf{H}_2$); 3.5 (s,1H,N**H**); 3.97–4.42 (m,3H,cycle); 6.97–7.23 (m,5H,**Ph**)
11	1.48–1.87 (m,13H,$SCH_2C\mathbf{H}_2$, cyclohexyl); 2.07 (s,3H,$C\mathbf{H}_3S$); 2.47–2.58 (m,2H,$SC\mathbf{H}_2$); 3.10 (s,1H,N**H**) 3.63–4.43 (m,3H,cycle)
12	1.19–1.36 (t,3H,$COOCH_2C\mathbf{H}_3$); 1.72–1.89 (m,2H,$SCH_2C\mathbf{H}_2$); 2.06 (s,3H,$C\mathbf{H}_3S$); 2.46–2.54 (m,2H,$SC\mathbf{H}_2$); 3.27 (s,1H,N**H**); 3.62–3.67 (m,2H,$NHC\mathbf{H}_2COOEt$); 3.70-3.78(m,3H, cycle); 4.13–4.41 (m,2H,$COOC\mathbf{H}_2$)

on the different exocyclic R group on the phosphorus atom center. We found that the products **6** and **9** (R=Et_2N, Ph) have higher diastereoselectivity than other compounds. Their de% value reached 81.82%. However, the highest de% was a little more than 60% in the previous literature. The configuration correlations of compound **7** (see Scheme 3) prepared as diastereomerically pure isomers with some spectra data are shown in Table 3. Bentrude et al. [17] have found that the ^{31}P NMR chemical shift correlates with the orientation of an exocyclic group on a phosphorus atom (not P=O) in 2-oxo-1,3,2-oxazaphosphorinanes; namely, when this group resides in the axial position, the chemical shift δ is less than when it resides in the equatorial position (δ_p (axial) < δ_p (equatorial)). Later, Thompson et al. [16] extended this finding to a series of 2-oxo-1,3,2-oxazaphospholidines derived from L-serine and also obtained reasonable results. In our study, this configuration correlation with the ^{31}P NMR chemical shift is also observed (Table 3). Our preliminary configuration assignments are as follows: the slow eluting isomer **7b** corresponds to the exocyclic R group trans to the thioether moiety, and the fast band **7a** corresponds to the ligand cis to the thioether group.



Syn

7a (δ_p 27.65 ppm)



Trans

7b (δ_p 29.47 ppm)

The IR spectra of **1–12** (Table 3) showed bands 3206–3427 cm^{-1} (P—NH) and 772.8–812.3 cm^{-1} (P=S). The products **7a** and **7b** showed bands 1229.0, 1233.9 cm^{-1} (P=O), respectively.

The biological activities of the title compounds are being tested.

EXPERIMENTAL

Instruments and Reagents

Melting points were determined on a model YANACO MP-500 apparatus and are uncorrected. All temperatures and pressures are uncorrected. ^{1}HNMR spectra were taken in deuterated chloroform ($CDCl_3$) on

TABLE 3 ^{31}P NMR Data and IR Data for Compounds **1–12**

			IR γ (cm^{-1}) film or KBr			
Compound	*^{31}PNMR δ (ppm)*	*de (%)*	*N–H*	*C–O*	*P–N*	*P=S(O)*
1	88.89,86.37	49.53	3206.0	1221.8	1035.0	794.0
2	58.60,56.12	10.24	–	–	–	–
3	57.50,55.00	5.26	–	–	–	–
4	82.61,79.92	16.18	3387.0	1212.3	1066.5	772.8
5	88.65,85.86	45.05	3290.0	1291.8	1030.0	799.1
6	87.13,84.29	81.82	3278.0	1207.4	1019.0	786.7
7a	27.65	13.26	3230.0	1229.0	1029.0	1229.0
7b	29.47		3427.0	1233.9	1060.7	1233.9
8	118.97,121.12	7.45	3298.0	1278.2	1073.0	812.3
9	95.02,97.78	81.82	3327.0	1248.5	1109.0	808.1
10	74.85,78.49	3.93	–	–	–	–
11	87.68,85.52	2.38	3289.0	1233.9	1097.0	807.4
12	82.74,80.35	35.26	3340.0	1742.4(C=O)	1027.4	805.3

AC-P300 or AC-P200 instruments using TMS as an internal standard. ^{31}PNMR chemical shifts are relative to phosphoric acid (H_3PO_4 in $CDCl_3$). Elemental analyses were conducted on a YANAMT-3 instrument. Analytical thin layer chromatography (TLC) was conducted with glasses-backed silica plates (GF-254). Visualization was accomplished with an ultraviolet lamp. α-amino acids used were BCS grade and of the L-configuration. All solvents and reagents were purified when necessary by standard literature methods. Air or water-sensitive reactions were conducted under a positive nitrogen atmosphere by utilizing standard techniques.

Preparation of L-Methionol

L-methionol [18] was prepared from L-methionine 29.84 g by the $NaBH_4$-I_2 procedure to afford 62.02% of 16.77 g as a colorless oil: bp 104–108°C/0.37 mmHg. The oil obtained from a second experiment spontaneously solidified to give colorless crystals: mp 33–34°C.

Preparations of Thiophosphoryl Dichlorides

O-Alkyl Thiophosphorodichloridate [19]. Acoording to an ordinary method, these products were obtained by the reaction of thiophosphoryl chloride with excess absolute alcohol at 5–10°C. The correlated data are shown in the following:

(MeOP(S)Cl_2: bp 108–112°C/26.67 kPa, n_D^{27} 1.5110, yield 61.33%. (lit: bp 42°C/1.33 kPa, n_D^{25} 1.5124, yield 76.68%); EtOP(S)Cl_2: bp 118–120°C/23–24 kPa, n_D^{27} 1.5008, yield 64.57%. (lit: bp 60–61°C/1.06 kPa, n_D^{25} 1.5026, yield 87.60%); *i*-PrOP(S)Cl_2: bp 78–80°C/1.325 kPa, yield 52.35%. (lit: bp 62–64°C/0.931 kPa, yield 70.50%.)

O-Phenyl Thiophosphorodichloridate [20]. This compound was obtained by the reaction of phenol with excess thiophosphoryl chloride at 8–10°C in 56.56% yield, bp 102–104°C/60–80 Pa, n_D^{30} 1.5720 (lit: bp 129°C/1.60 kPa, yield 66%, n_D^{20} 1.5730).

N,N-Dimethyl Thiophosphorodichloridate [21]. This compound was prepared by the reaction of dimethylamine hydrochloride, thiophosphoryl chloride, and triethylamine in chloroform at about −5–+5°C in 46.79% yield, bp 53–56°C/120 Pa, n_D^{12} 1.5450. (lit: bp 66–67°C/133 Pa, yield 43.5%, n_D^{20} 1.5404.)

N,N-Diethyl Thiophosphorodichloridate [22]. This compound was prepared by the reaction of diethylamine with thiophosphoryl chloride in benzene at about 50°C in 63.46% yield, bp 124–126°C/10 mmHg, $n_D^{17.5}$ 1.5280. (lit: bp 77°C/93 Pa, yield 60.80%, n_D^{25} 1.5240.)

Phenylphosphonodichloide [23]. This compound was prepared by the reaction of benzene with excess phosphorus trichloride catalyzed by anhydrous aluminum trichloride at about 78–80°C for 4–5 h, then the addition of sulfur powder into above mixture in small portions at 35–40°C in 77.13% yield, bp 97–98°/90 Pa, n_D^{12} 1.6280. (lit: bp 92–94°/0.04–0.067 kPa, yield 87.2%, n_D^{20} 1.6227.)

Isopropyl Phosphonidichloride [24]. This compound was synthesized by one-step synthesis of isopropylphosphonothioic dichlorides from chloroaluminate complex [*i*-$PrPCl_3$]$^+$[Al_2Cl_7]$^-$. The complex was treated with thiourea at room temperature followed by water to obtain pure isopropylphosphonothioic dichloride in one step in 33.4% yield,

bp 64–68°C/10 mmHg, n_D^{20} 1.5370. (lit: bp 70°C/12 mmHg, yield 33.39%, n_D^{17} 1.5391.)

Di-(2-chloroethyl)-phosphoramidic Dichloride [25]. This compound was obtained by the reaction of bis-(-chloroethyl)-amine hydrochloride [26] with excess phosphorus oxychloride under reflux for 12 h in 78.64% yield, bp 116–120°C/0.4 mmHg. (lit: bp 123–125°C/0.6 mmHg, yield 81.2%)

Preparation of Compounds **1–9**

To a solution of L-methionol (0.68 g, 5 mmol) in 10 mL of THF, triethylamine (1.01 g, 10 mmol) was added, and the mixture was cooled to 0°C in an ice-bath, then a solution of *O*-methyl thiophosphorodichloridate (0.83 g, 5 mmol) in 5 mL of THF was slowly added dropwise at 0°C. After the addition, the reaction mixture was stirred for 1 h, warmed to room temperature, and continued to stir for 6–7 h. After removal of triethylamine hydrochloride by filtration, the filtrate was concentrated under reduced pressure. The crude product 1 was purified and separated by column chromatography (petroleum ether: ethyl acetate as eluent) to afford diastereomeric mixture: 0.70 g , yield 61.24%.

Similarly, compounds **2–9** were prepared from the corresponding thiophosphoryl dichloride as a diastereomeric mixtures. The only difference was that the reactive time was prolonged with increasing R group. Data are shown in Tables 1–3.

Preparation of Compounds **10–12**

To a solution of L-methionol (0.68 g, 5 mmol) in 10 mL of THF, triethylamine (TEA) (1.01 g, 10 mmol) was added, and the mixture was cooled to −10°C in an ice-salt bath. Then a solution of thiophosphoryl chloride (0.85 g, 5 mmol) in 5 mL of THF was slowly added dropwise at −10°C. The reaction was monitored by TLC for loss of starting material. After completion, a sample for ^{31}P NMR examination was taken from the reaction mixture. Then the phenylamine (0.465 g, 5 mmol) was added at room temperature to a solution of 2-chloro-2-thio-1,3,2-oxazaphospholidine-4-methyl ethyl thioether in THF followed by the addition of TEA (100 mol%). The reaction mixture was stirred at room temperature overnight. After removal of triethylamine hydrochloride by filtration, the filtrate was concentrated under reduced pressure to obtain the crude product 1, which was purified and separated by column chromatography (petroleum ether: ethyl acetate as eluent) to afford a diastereomeric mixture 10: 0.55 g, yield 38.19%.

Similarly, compounds **11** and **12** were prepared from the corresponding thiophosphoryl dichloride as a diastereomeric mixtures. Data are shown in Tables 1–3.

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