Heteroatom Chemistry
Volume 10, Number 2, 1999

Synthesis of Biologically Active Phosphorus Heterocycles via Cyclization Reactions of Lawesson's Reagent

Liang-Nian He,[1,2] Ren-Xi Zhuo,[1] Ru-Yu Chen,[3] Kai Li,[2] and You-Jie Zhang[2]

[1]*Department of Chemistry, Wuhan University, Wuhan, 430072, P. R. China*

[2]*Institute of Organic Synthesis, Central China Normal University, Wuhan, 430079, P. R. China*

[3]*Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, 300071*

Received 19 December 1997; revised 23 March 1998

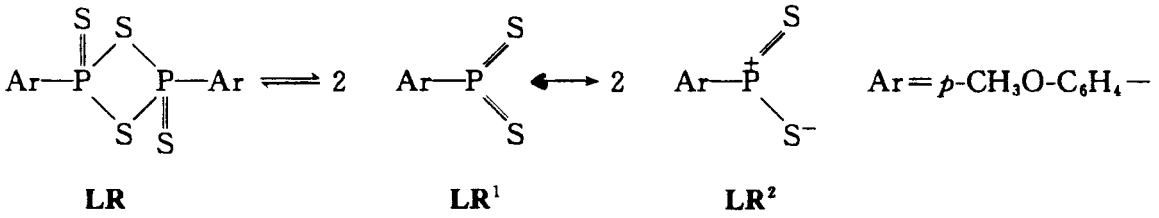
ABSTRACT: *2,4-Bis(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulfide (Lawesson's reagent,* **LR***) reacted with certain bifunctional compounds, such as glycinamides* **1***, 1-glyceryl monoesters* **4***, and 3-mercapto-4-amino-5-substituted-1,2,4-triazoles* **6***, to yield five-membered phosphorus heterocycles* **3**,**5***, and* **7***, respectively. The cycloaddition reaction of* **LR** *with 1,3-butadiene* **8** *was investigated to form the six-membered heterocycle* **9***. The results of preliminary bioassays showed that these heterocycles obtained via cyclization reactions of* **LR** *possess significant selective herbicidal activity. The QSAR of* **3** *was also made. In conclusion, the cyclization reactions of* **LR** *with bifunctional substrates and 1,3-dienes afford novel routes to the syntheses of biologically active phosphorus heterocycles*. © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 105–111, 1999

INTRODUCTION

One of the best known thiation reagents is 2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulfide, known as Lawesson's reagent (**LR**) [1]. It also undergoes cyclization reactions with substrates containing two functional groups to form phosphorus or sulfur-containing heterocycles, which incorporate the (4-methoxyphenyl) phosphinothioylidene moiety [2–4]. These heterocyclic compounds are of potential interest as herbicides, insecticides, and fungicides [5,6]. Recently, we became interested in syntheses of phosphorus heterocycles by cyclization reactions of Lawesson's reagent, some of them having shown significant selective herbicidal activity [7–9].

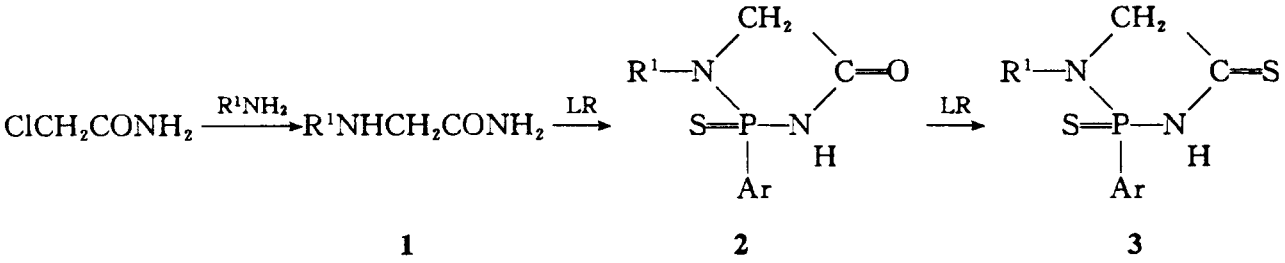
The possible existence of the monomeric form of **LR**[1] in solution led us to start investigating the potential of **LR** as a dienenophile for [2 + 4] cycloaddition reactions (Scheme 1) [1,10]. It has been reported that the stable monomeric dithioxo(tri-tert-butylphenyl)phosphorane reacts with 2,3-dimethyl-1,3-butadiene in a [4 + 2] cycloaddition process [11–13], and **LR**, as a dipolarophile, undergoes [2 + 3] cycloaddition reactions with stable 1,3-dipolar compounds [10]. However, to our knowledge, there have been no attempts to utilize **LR** as a dienenophile or as an electrophile in syntheses of biologically active phosphorus heterocycles with P–S bond incorporation, and no report on biological activity of phosphorus-containing heterocycles obtained by employing Lawesson's reagent. Herein, we wish to report in detail the preparation of biologically active phospho-

Correspondence to: Liang-Nian He.
Contract Grant Sponsor: National Natural Science Foundation of China.
Contract Grant Sponsor: NSF of Hubei Province.
Contract Grant Sponsor: Dawn Plan of Science and Technology for Young Scientists of Wuhan City.
 CCC 1042-7163/99/020105-07



LR **LR¹** **LR²**

SCHEME 1



1 **2** **3**

SCHEME 2

rus heterocycles **3**,**5**,**7**, and **9** by the cyclization reactions of **LR** with certain bifunctional substrates and of [2 + 4] cycloaddition reactions of **LR** with 1,3-dienes, respectively.

RESULTS AND DISCUSSION

Cyclization Reactions of **LR** *with Bifunctional Substrates* **1**, **4**, *and* **6**

LR reacted in benzene at 55–60°C with 3-substituted glycinamides **1**, which were obtained in satisfactory yields by amination of α-chloroacetamide in benzene at 80°C, to give 1,3,2-diazaphospholidin-4-thione-2-sulfide **3** in moderate yield, as depicted in Scheme 2. In the reaction, 1 mole of **LR** was sufficient for the conversion of 1 mole of the substrate to the phosphorus heterocyclic compounds **3**. When the reaction was carried out in benzene at room temperature, the ring-closure product **2** was formed, but **3** and the thionation product of **1** were not found by gas chromatography–mass spectra (GC-MS) detection. Therefore, the carbonyl group of glycinamide is thionated after the ring-closure stage.

The structures of **3** were established by elemental analysis, IR, ^{1}H-NMR, and ^{31}P-NMR spectroscopy, MS, and X-ray diffraction, as shown in Table 1. Figure 1 is the molecular structure of compound **3b** R^1 = *o*-$CH_3C_6H_4$) showing the atomic numbering scheme. The X-ray diffraction analysis indicated the coplanar structure of the five-membered phosphorus heterocycle and the existence of the dπ-pπ bond between P and N(1) atom.

The reaction of long-aliphatic-chain 1-glyceryl esters **4** with 0.5 equivalence of **LR** in toluene at 100°C under anhydrous nitrogen for 3 hours led to five-membered cyclic phospholipid analogs **5** (Scheme 3) in moderate yields. The results and spectral data are listed in Table 2. The structure of **5a** (R^2 = tridecyl), taken as a representative example, was confirmed by elemental analysis, ^{1}H-NMR and ^{31}P-NMR spectroscopy, and MS. Compound **5a** showed signals at δ 3.84 (s, 3H, OCH_3, para to the phosphorus atom), 7.07–7.12 (dd, 2H, meta protons to the phosphorus atom), 7.78–7.84 (dd, 2H, ortho protons to the phosphorus atom in the anisole ring), 4.89(m,1H), 4.72(m,2H), 4.27(m,2H), 3.34 (m, 2H), 1.52 (m, 2H), 1.21 (m, 20H), 0.90 (t, 3H, methyl group at the end of the long chain ester group). Compound **5a** under electron impact gave the molecular ion peak *m*/*z* (%), 470(2.65), and other conspicuous peaks: 228, 188, and 171. The IR spectrum of **5a** showed normal stretch absorption bands, indicating the existence of the C=O, P=S, and P–O–C groups. This cyclization reaction of the 1-glyceryl ester with **LR** supplies another convenient route to five-membered cyclic phospholipid analogs.

1,2,4-Triazole derivatives are known as biologically active materials besides having other uses and applications [14,15]. In order to look for potent biologically active materials, the reactions of **LR** with 3-mercapto-4-amino-5-substituted-1,2,4-triazoles **6** were investigated. The treatment of 1 mole of each **6** with 0.5 equivalent of **LR** at 100°C in dry toluene resulted in a new fused phosphorus heterocycle **7**, as shown in Scheme 4. The results and spectral data are listed in Table 2.

TABLE 1 The Physical and Chemical Data of Compounds **3**

No.	R[c] R^1	Yield (%)	MP (°C)	Elemental Analysis Found (Calcd.) C (%)	H (%)	N (%)	MS (M^+)	IR (cm^{-1})	NMR[b] ($CDCl_2$, δ)
3a	H	51	161–162	53.87 (53.89)	4.68 (4.49)	8.45 (8.38)	334	3396 633	8.22 (d, 1H, NH), 6.93–7.83 (m, 9H, Ar-H), 4.80 (d, 2H, CH_2), 3.84 (s, 3H, OCH_2), ^{31}P-NMR: 71.57.
3b	*o*-Me	57	149–150	55.36 (55.17)	4.67 (4.89)	8.28 (8.05)	348	3207 637	8.48 (d, 1H, NH), 6.94–6.99 (dd, 4H), 7.75–7.87 (m, 4H), 4.74 (d, 2H, CH_3), 3.84 (s, 3H, OCH_3), 2.17 (s, 3H, CH_3), ^{31}P-NMR: 74.75.
3c	*m*-Me	53	168–170	55.29 (55.17)	4.73 (4.89)	8.22 (8.05)	—[a]	—[a]	8.32 (d, 1H, NH), 6.77–7.86 (m, 8H, Ar-H), 4.81 (d, 2H, CH_2), 3.84 (s, 3H, OCH_3), 2.21 (s, 3H, CH_3), ^{31}P-NMR: 71.84.
3d	*p*-Me	57	162–163	55.35 (55.17)	4.78 (4.89)	8.17 (8.05)	—	3283 639	8.42 (d, 1H, NH), 6.88–7.90 (m, 8H, Ar-H), 4.79 (d, 2H, CH_2), 3.84 (s, 3H, OCH_3), 2.21 (s, 3H, CH_3), ^{31}P-NMR: 71.84.
3e	*o*-Cl	43	154–155	48.96 (48.85)	3.92 (3.80)	7.73 (7.06)	—	—	8.40 (d, 1H, NH), 6.97–7.88 (m, 8H, Ar-H), 4.68 (d, 2H, CH_2), 3.85 (s, 3H, OCH_3), ^{31}P-NMR: 71.68.
3f	*p*-Cl	50	148–149	48.64 (48.85)	3.92 (3.80)	7.43 (7.06)	368	3374 640	8.48 (d, 1H, NH), 6.88–7.88 (m, 3H, Ar-H), 4.74 (d, 2H, CH_2), 3.84 (s, 3H, OCH_3), ^{31}P-NMR: 71.66.
3g	2,4-2Cl	31	170–172	48.34 (44.55)	3.28 (3.32)	6.85 (6.93)	—	—	8.46 (d, 1H, NH), 6.93–7.80 (m, 7H, Ar-H), 4.72 (d, 2H, CH_2), 3.85 (s, 3H, OCH_3)
3h	*p*-Br	55	156–157	43.57 (43.58)	3.31 (3.39)	6.58 (6.78)	413	3386 638	8.48 (d, 1H, NH), 6.93–7.89 (m, 8H, Ar-H), 4.61 (d, 2H), 3.86 (s, 3H, OCH_3), ^{31}P-NMR: 71.60.
3c	*o*-Br	48	157–158	43.41 (43.58)	3.42 (3.39)	6.93 (6.78)	—	—	8.46 (d, 1H, NH), 6.95–7.83 (m, 8H, Ar-H), 4.70 (d, 2H, CH_2), 3.84 (s, 3H, OCH_3).
3j	*o*-NO_2	40	124–126	47.62 (47.49)	3.54 (3.69)	11.23 (11.08)	379	3342 642	8.54 (d, 1H, NH), 6.98–7.90 (m, 8H, Ar-H), 4.72 (d, 2H, CH_2), 3.85 (s, 3H, OCH_3).
3k	*m*-NO_2	36	101–102	47.55 (47.49)	3.56 (3.69)	11.26 (11.08)	—	3360 634	8.46 (d, 1H, NH), 6.98–7.88 (m, 8H, Ar-H), 4.72 (d, 2H, CH_2), 3.85 (s, 3H, OCH_3).
3l	*p*-NO_2	31	142–143	47.38 (47.49)	3.52 (3.69)	11.25 (11.08)	—	3380 634	8.72 (d, 1H, NH), 6.95–7.92 (m, 8H, Ar-H), 4.77 (d, 2H, CH_2), 3.88 (s, 3H, OCH_3).
3m*	Me	58	141–142	44.25 (44.12)	4.59 (4.78)	10.12 (10.29)	272	—	8.92 (s, 1H, NH), 6.74–8.05 (m, 4H, C_6H_4) 4.70 (d, 2H, CH_2), 3.84 (s, 3H, OCH_3), 3.26 (d, 3H, CH_3).
3n*	Et	54	145–146	46.42 (46.15)	5.08 (5.24)	9.45 (9.79)	—	3214 639	8.90 (s, 1H, NH), 6.82–8.10 (m, 4H, C_6H_4), 4.72 (d, 2H, CH_2), 3.82 (s, 3H, OCH_3), 3.24 (m, $2H_2$ CH_2 CH_3), 1.36 (t, 3H, CH_3 CH_2).
3o*	ipr	46	151–152	48.16 (48.00)	5.32 (5.67)	9.62 (9.33)	300	3210 640	8.94 (s, 1H, NH), 6.79–8.00 (m, 4H, C_6H_4), 4.72 (d, 2H, CH_2), 3.82 (s, 3H, OCH_3), 3.28 (m, 1H, CH), 1.24 (d, 6H, CH_3).

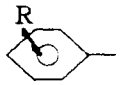
[a] Data were not recorded.

[b] The solvent marked by asterisk is DMSO-d_6

[c] R^1 = 

TABLE 2 Physical Data and Analytical Data of **5, 7,** and **9**

No.	R^a R^2	R^4/R^5	M. P. /n_D^{20}	Yield (%)	Elemental Analysis Found (Calcd) C (%)	H (%)	N (%)	MS (M^+)	IR (cm^{-1})	NMR[c] ($CDCl_3$, ppm)
5a	$C_{13}H_{27}$		47–48	46	61.43 (61.28)	8.15 (8.30)		470	650 1130 1740	7.07–7.84 (m, 4H, C_6H_4), 3.84 (s, 3H, OCH_3), 4.89 (m, 1H, CH), 4.72 (m, 2H, CH_2), 4.27 (m, 2H, CH_3), 3.34 (m, 2H, CH_3), 1.52 (m, 2H, CH_2), 1.21 (m, 20H, 10 × CH_3), 0.90 (t, 3H, CH_3), ^{31}P-NMR 39.68.
5b	$C_{15}H_{31}$		43–44	44	62.78 (62.65)	8.45 (8.63)		—[b]	645 1736 1145	7.08–7.81 (m, 4H, C_6H_4), 3.85 (s, 3H, OCH_3), 5.07 (m, 1H, CH), 4.74 (m, 2H, CH_3), 4.74 (m, 2H, CH_3), 3.30 (m, 2H, CH_3), 1.49 (m, 2H, CH_2), 1.24 (m, 24H, 12 × CH_3), 0.92 (t, 3H, CH_3).
5c	$C_{17}H_{35}$		35–36	37	63.65 (63.88)	8.99 (8.94)		—	648 1745 1150	6.96–7.84 (m, 4H, C_6H_4), 3.86 (a, 3H, OCH_3), 5.10 (m, 1H, CH), 4.75 (m, 2H, CH_2), 4.23 (m, 2H, CH_2), 3.31 (m, 2H, CH_2), 1.46 (m, 2H, CH_2), 1.20 (m, 28H, 14 × CH_2), 0.90 (t, 3H, CH_3).
7a*	H		165–167	45	50.38 (50.00)	3.35 (3.61)	15.19 (15.56)	360	640 3150	6.96–7.60 (m, 9H, Ar–H), 5.40 (s, 1H, NH), 3.79 (a, 3H, OCH_3). ^{31}P-NMR 14.49.
7b*	*p*-Me		162–163	50	51.58 (51.34)	3.88 (4.01)	14.75 (14.97)	—	645 3205	6.98–7.64 (m, 8H, A–H), 5.41 (s, 1H, NH), 3.82 (s, 3H, OCH_3), 2.34 (s, 3H, CH_3).
7c*	*p*-Cl		154–155	45	45.43 (45.63)	2.96 (3.04)	14.41 (14.20)	—	635 3155	6.90–7.61 (m, 8H, Ar–H), 5.37 (s, 1H, NH), 3.80 (s, 3H, OCH_3).
9a		H/Me	1.6165	63	53.07 (53.33)	5.81 (5.56)		270	1099 1404 1665 1090	6.92–7.93 (m, 4H, Ar–H), 5.82 (m, 1H, CH), 3.82 (s, 3H, OCH_3), 2.70–3.60 (m, 4H, CH_3), 1.80 (s, 3H, CH_3), ^{31}P-NMR 52.39.
9b		Me/Me	1.6372	66	54.72 (54.93)	6.14 (5.99)		284	1400 1660	6.90–7.95 (m, 4H, Ar–H), 3.84 (s, 3H, OCH_3), 2.72–3.64 (m, 4H, CH_3), 1.78 (s, 6H, CH_3).

[a] R^3 = 

[b] Data were not recorded.

[c] The solvent marked by asterisk is DMSO−d_6.

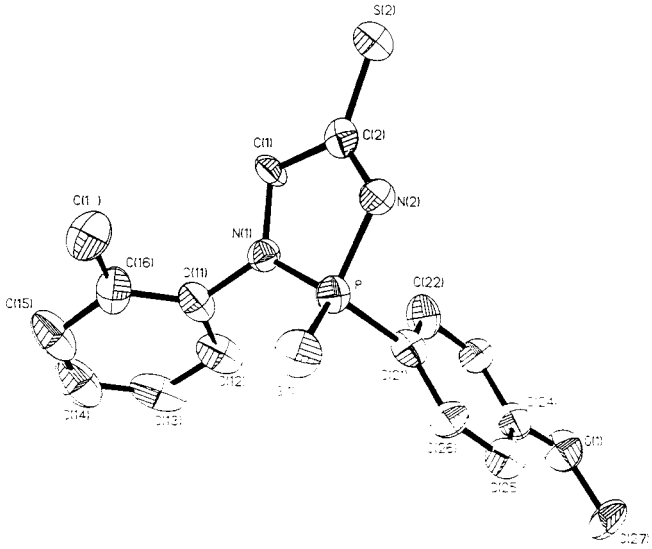
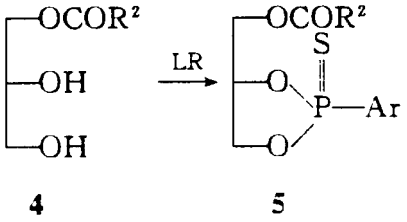
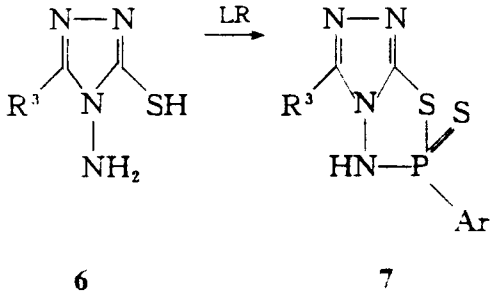


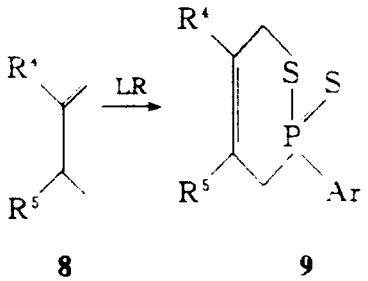
FIGURE 1 The molecular structure of **3b**.



SCHEME 3

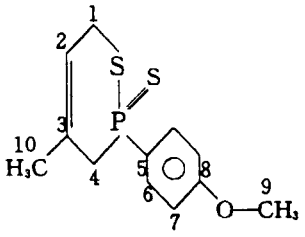


SCHEME 4



SCHEME 5

TABLE 3 ^{13}C-NMR Data of **9** ($CDCl_3$)



C	δ	J_{PC} (*Hz*)	*C*	δ	J_{PC} (*Hz*)
1	28.35	5.03	6	132.60	13.03
2	120.26	12.42	7	113.90	14.13
3	133.35	9.31	8	162.45	0
4	41.15	51.76	9	55.30	0
5	124.31	88.23	10	26.03	9.81

Cycloaddition Reactions of **LR** *with 1,3-dienes* **8**

The opportunity exists for [2 + 4] cycloaddition reactions with unsaturated substrates having 4π-electrons to yield six-membered phosphorus- and sulfur-containing heterocycles. 1,3-Dienes **8** were chosen as the unsaturated substrates for this purpose. 2-Methyl-1,3-butadiene(**8a**) reacted with 0.5 equivalent of **LR** in anhydrous toluene at 100°C under dry nitrogen for 4 hours, leading to a six-membered ring compound, **9a** (R^4 = H, R^5 = Me), in a significant yield (Scheme 5). The results and spectral data are listed in Table 2. The ^{13}C-NMR chemical shift and assignments of **9a** are listed in Table 3. The chemical shifts appearing at 28.35 and 26.03 are assigned to the carbon of C-1 and C-10, which was confirmed by a DEPT ^{13}C-NMR technique.

Herbicidal Activity

The herbicidal activities of heterocycles **3,5,7,** and **9** were tested. A set amount of each sample was dissolved in acetone to which a drop of an emulsifier was added. Then, the solution was diluted with water until it reached the concentration required. Rape and barnyard grass were subjected to the leaf treatment. Preliminary bioassays indicated that some of these compounds display significant selective herbicidal activity at 3.0 kg/ha.

QSAR of 1,3,2-Diazaphospholidin-4-thione-2-sulfide **3**

The quantitative relationship between the structure of **3** and their herbicidal activity on rape was ana-

lyzed, using the physicochemical parameters of the substituents in the benzene ring by regression analysis. The following regression equation could be established:

$$D = 2.980\pi + 2.035\sigma - 0.150\text{Es} + 2.947$$

$$n = 13, \gamma = 0.872, s = 0.211$$

Herbicidal activity is indicated in terms of the activity indicator (D)[16]: $D = \lg[a/(100 - a)] + \lg \text{MW}$, where a refers to inhibition percentage against rape at 3.0 kg/ha and MW is the molecular weight. Determination of each a was repeated for at least three runs and averaged.

The equation indicates that there exists a relationship between the hydrophobic parameter (π)[17], steric parameter (Taft constant: Es), and electronic effect parameter (σ) of a substituent with herbicidal activity, with a significant regression coefficient. Although the steric parameter also affected herbicidal activity, the contribution was smaller than that of the hydrophobic property and the electronic parameter. The results were used as a guide to the synthesis of more highly active compounds.

In conclusion, cyclization reactions of **LR** with certain bifunctional substrates and the cycloaddition reactions of **LR** with 1,3-dienes are novel routes leading to biologically active phosphorus heterocycles.

EXPERIMENTAL

Melting points were uncorrected, ^{1}H-NMR and ^{31}P-NMR spectra were recorded on a varian XL-200 MHz spectrometer. Mass spectra were measured on an HP 5988A spectrometer and a VG-7070E spectrometer. The IR spectra were measured by using a SHIMADZU-435 instrument. Elemental analyses were performed with a CHN CORDERD MT-3 elementary analyzer. Column chromatography was performed on silica gel I (10–40μ, Hai Yang Chemical Factory of Qingdao). All solvents and materials were reagent grade and purified as required. Lawesson's reagent was prepared in a yield of 75% according to a published procedure [18].

A single crystal of **3b** was cultured from a mixture of petroleum ether and dry ethyl ether. The reflections in the range of $4 < 2\theta < 46°$ were collected on an ENRAF-NONIUS CAD_4 X-ray diffractiometer with MoKα radiation ($\lambda = 0.071073$ nm). All calculations were performed on a PDP 11/44 computer using the SDP-PLUS program system. The crystal is monoclinic with space group $P2_1/n$, $a = 9.495$ (2) Å, $b = 7.954$ (5) Å, $c = 23.039$ (8) Å, $\beta = 96.30$ (2)°, $V = 1739$ (2) Å^3, $Z = 4$, Mr = 348, F(000) = 728, μ (MoKα) = 0.386 mm^{-1}, Dx = 1.331 Mg/m^3. The final R factor is 0.044 and Rw is 0.047 for 1002 observed reflections [$1 \geqslant 3\sigma(I)$].

1,2,4-Triazoles **6** were synthesized according to Ref. [19].

Preparation of Glycinamides **1**

At 20°C, trimethylamine (25 mmol) was added dropwise to a mixture of α-chloroacetamide (25 mmol), the reactant amine (25 mmol), and anhydrous benzene (20 mL). The resulting mixture was refluxed for 5–6 hours. The reaction mixture was cooled to room temperature and filtered. The product obtained was suspended in 100 mL of water and subsequently collected by filtration and crystallized from a mixture of water and ethanol.

General Procedure for the Reaction of **LR** *with Each Glycinamide* **1**, *Preparation of 1,3,2-Diazaphospholidin-4-thione-2-sulfides* **3**

A mixture of each glycinamide **1**(0.01 mol), Lawesson's reagent (0.01 mol), and 100 mL of dry benzene was stirred at 55–60°C for 3–4 hours until no more of the starting material could be detected by TLC. The solvent was evaporated under reduced pressure and the residue purified by chromatography on a silica gel column using petroleum ether and dry ethyl ether mixtures as eluent: The physical and chemical data are summarized in Table 1.

General Procedure for the Cyclization Reaction of Lawesson's Reagent with Each 1-Glyceryl Monoester **4**, *Preparation of Cyclic Phospholipid Analogs* **5**

A suspension of 0.01 mole of each starting material **4**, 0.005 mole of **LR**, and 15 mL of dry toluene was stirred magnetically at 100°C for 3–4 hours until no more of the starting material could be detected by TLC. The solvent was evaporated under reduced pressure and the residue applied to a silica gel column using dry ethyl ether/petroleum ether mixture as eluent (starting from 10% up to 15%) to give each cyclic phospholipid analog **5**.

General Procedure for the Reaction of 1,2,4-Triazoles **6** *with* **LR**, *Preparation of* **7**

A mixture of 0.01 mole of each starting compound **6** and 0.005 mole of **LR** was refluxed in 15 mL of anhydrous toluene with stirring until no more of the starting material could be detected by TLC. Evaporation of the solvent under reduced pressure followed by purifying the residue on a silica gel column using dry ethyl ether/petroleum ether mixture as eluent gave each fused ring **7.**

General Procedure for Cycloaddition Reaction of Lawesson's Reagent with 1,3-Dienes **8**, *Preparation of each* **9**

A suspension of 0.01 mole of **LR**, 0.05 mol of each 1,3-diene **8**, and 5 mL of anhydrous toluene was stirred at reflux for 4–5 hours. The solvent was evaporated under reduced pressure and the residue applied to a silica gel column using petroleum ether/ethyl ether mixture (10,1) as eluent to give each product **9**.

REFERENCES

[1] M. P. Cava, M. I. Levinson, *Tetrahedron*, *41*, 1985, 5061.
[2] R. A. Cherkasov, G. A. Kutyrev, A. N. Pudovik, *Tetrahedron*, *41*, 1985, 2588.
[3] R. Shabana, F. H. Osman, S. S. Atrees, *Tetrahedron*, *50*(23), 1994, 6975.
[4] R. Shahana, F. H. Osman, S. S. Atrees, *Tetrahedron*, *49*(6), 1993, 1271.
[5] R. Shabana, S. S. Atrees, *Phosphorus, Sulfur, and Silicon*, *105*, 1995, 57.
[6] A. A. Fahmy, *Phosphorus, Sulfur, and Silicon*, *68*, 1992, 139.
[7] L.-N. He, R.-X. Zhuo, R.-Y. Chen, J. Zhou, *Synth. Commun.*, *27*, 1997, 2853.
[8] L.-N. He, R.-Y. Chen, *Heterocycl Commun.*, *3*, 1997, 461.
[9] L.-N. He, R.-Y. Chen, *Phosphorus, Sulfur, and Silicon*, in press.
[10] N. Dubau-Assibat, A. Baceiredo, G. Bertrand, *J. Org. Chem.*, *60*, 1995, 3904.
[11] J. Navech, J. P. Majoral, R. Kraemer, *Tetrahedron Lett.*, *24*, 1983, 5885
[12] J. Navech, J. P. Majoral, A. Meriem, R. Kraemer, *Phosphorus Sulfur*, *18*, 1983, 27.
[13] J. Navech, M. Revel, R. Kraemer, *Phosphorus Sulfur*, *21*, 1984, 105.
[14] G. F. Duffin, J. D. Kendall, H. R. J. Waddington, *J. Chem. Soc.*, 1959, 3799.
[15] A. K. Bhat, *Indian. J. Chem.*, *5*, 1967, 397.
[16] H. L. Wang, J. Zhou, Y. G. Qiu, K. S. Feng, *Phosphorus, Sulfur, and Silicon*, *104*, 1995, 35.
[17] T. Fujita, J. Iwasa, C. Hanach, *J. Am. Chem. Soc.*, *84*, 1964, 5157.
[18] I. Thomsen, K. Clausen, S. Scheibye, S.-O. Lawesson, *Org. Synth.*, *62*, 1984, 158.
[19] R. J. Reid, D. N. Heindel, *J. Heterocycl. Chem.*, *13*, 1976, 925.