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### SYNTHESIS OF NEW $\beta$ -(1,3-BENZOTHAZOLE-2-)PHOSPHORIC HYDRAZIDES

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## SYNTHESIS OF NEW $\beta$ -(1,3-BENZOTHAZOLE-2-)PHOSPHORIC HYDRAZIDES

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$\beta$ -(1,3-Benzothiazole-2-)phosphoric hydrazides **2** were synthesized by Atherton-Tod method using potassium carbonate as a base in the presence of tetrabutylammonium bromide as a phase transfer catalyst or by the intermolecular condensation of the hydrazines **1** with phosphorochloridate,  $(R^3O)(R^4O)P(O)Cl$ , in the presence of a base, triethylamine. Thermal decomposition at 180°C of **2a** caused the intramolecular shift of ethyl group from oxygen to  $\beta$ -nitrogen atom, and formed the monoester phosphoric hydrazide **3**, whose structure was determined by elementary analysis, IR,  $^1H$  NMR,  $^{13}C$  NMR,  $^{31}P$  NMR, MS spectra. The biological activity of the title product was also tested and some of them showed good fungicidal activity.

**Keywords:** phosphoric hydrazide; Atherton-Tod method; thermal decomposition; intermolecular condensation; fungicidal activity

### INTRODUCTION

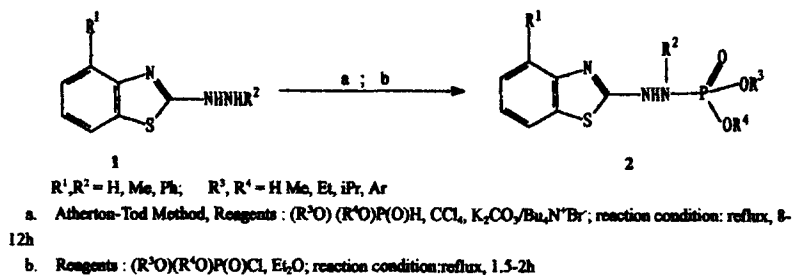
Thiadiazole derivatives are known as biologically active materials, which have exhibited the antitubercular, antiviral, antifungal, carcinostatic, hypoglycemic properties<sup>[1-6]</sup>. Functionalized organophosphorus acids and their derivatives are of great interest as bioactive substances with various properties<sup>[7]</sup>. It is reported that phosphoryl hydrazines possess good biological activity<sup>[8]</sup>. In our laboratory<sup>[9,10]</sup>, intermolecular cyclocondensation reactions of 2-hydrazono-2,3-dihydro-1,3-benzothiazole with tris(diakylamino)phosphines was carried out, and we found that their products and some phosphoryl hydrazines possess good inhibitory effects

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against tobacco mosaic. In recent years, a focus of our research has been the development of new synthetic methodology centered around biologically active phosphorus compounds. In this paper, we wish to report the synthesis of new  $\beta$ -(1,3-benzothiazole-2-) phosphoric hydrazides (**2**), which may serve as plant virucides.

## RESULTS AND DISCUSSION

Synthesis of  $\beta$ -(1,3-benzothiazole-2-)phosphoric hydrazides **2** was easily accomplished by phase transfer Atherton-Tod reaction (Route a, Scheme 1) of **1** with dialkyl phosphite in the of potassium carbonate. The yields are higher than 65%, except for **2c**, **2f**, **2i** ( $R^2=\text{ph}$ ). We also prepared **2** by employing intermolecular condensation (Route b, Scheme 1) of O,O-disubstituents phosphorochloridate in the presence of triethylamine. Route b is suitable for the preparation of **2c**, **2f**, **2i**, and **2j-2q** ( $R^3/R^4=\text{aryl}$ ) in significant yield.



SCHEME 1

The structure of **2a**, taken as a representative example, was determined by elemental analysis, IR, NMR, and MS spectra. The physical data and the spectral data of new compounds **2** are tabulated in Table I, Table II, Table III, and Table IV, respectively. The IR spectrum of **2a** showed absorption bands at  $3380\text{cm}^{-1}$ ,  $3430\text{cm}^{-1}$  due to N-H, at  $1250\text{cm}^{-1}$  due to  $\text{P}=\text{O}$ , at  $1615\text{cm}^{-1}$  due to  $\text{C}=\text{N}$  (cyclic conjugated  $\text{C}=\text{N}$ ) vibration. In the  $^1\text{H}$  NMR, compound **2a** ( $\text{CDCl}_3$ ) showed signals at  $\delta = 7.20-7.50(\text{m}, 4\text{H})$ , of the ring benzene),  $4.20(\text{m}, 4\text{H}, \text{POCH}_2)$ ,  $1.34(\text{t}, 6\text{H}, \text{CH}_3)$ , splittd by the adjacent hydrogen atom). The chemical shift of the mobile proton in N-H of **2a** was 4.81, which disappeared when deuterated. Compound **2a** gave  $^{31}\text{P}$  NMR chemical shift at  $-2.01\text{ppm}$ . Its  $^{13}\text{C}$  NMR spectra were listed in Table III.

TABLE I The Physical Data and IR Data of Compounds **2**

No.	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup> /R <sup>4</sup>	State	M.P. (°C)	Yield (%)	Elemental analysis <sup>b</sup> , P(%) / Found (Calcd.)	IR (cm <sup>-1</sup> )
<b>2a</b>	H	H	Et/Et	Pale red solid	84–85	84	10.20(10.30)	3430, 3380(N-H) 1615(C=N) 1250(P=O)
<b>2b</b>	Me	H	Et/Et	White cryst.	89–90	85	10.18(9.84)	3450, 3350(N-H) 1614(C=N) 1240(P=O)
<b>2c</b>	H	Ph	Et/Et	Yellow solid	112–113	36.89 <sup>a</sup>	8.04(8.22)	3450(N-H) 1615(C=N) 1265(P=O)
<b>2d</b>	H	H	i-Pr/i-Pr	White cryst.	93–94	74	9.61(9.42)	3550, 3300(N-H) 1620(C=N) 1250(P=O)
<b>2e</b>	Me	H	i-Pr/i-Pr	White cryst.	99–100	86	8.81(9.04)	3450, 3250(N-H) 1610(C=N) 1255(P=O)
<b>2f</b>	H	Ph	i-Pr/i-Pr	Yellow solid	109–111	31.83 <sup>a</sup>	7.57(7.65)	3500(N-H) 1610(C=N) 1250(P=O)
<b>2g</b>	H	H	n-Bu/n-Bu	White cryst.	142–143	65	8.81(8.68)	3200, 3450(N-H) 1640(C=N) 1240(P=O)
<b>2h</b>	Me	H	n-Bu/n-Bu	White cryst.	107–108	67	8.53(8.36)	3450, 3200(N-H) 1610(C=N) 1250(P=O)
<b>2i</b>	H	Ph	n-Bu/n-Bu	White cryst.	85–86	18.76 <sup>a</sup>	7.01(7.18)	3480(NH) 1620(C=N) 1260(P=O)

No.	$R^1$	$R^2$	$R^3/R^4$	State	M.P. (°C)	Yield (%)	Elemental analysis <sup>b</sup> , P(%) / Found (Calcd.)	IR (cm <sup>-1</sup> )
<b>2j</b>	H	H	Et/p-MeC <sub>6</sub> H <sub>4</sub>	White cryst.	108–109	78 <sup>a</sup>	8.25(8.54)	3500(N-H) 1620(C=N) 1270(P=O)
<b>2k</b>	Me	H	Et/p-MeC <sub>6</sub> H <sub>4</sub>	Color solid	67–68	61 <sup>a</sup>	8.35(8.22)	3400, 3240(N-H) 1620(C=N) 1275(P=O)
<b>2l</b>	H	H	Et/C <sub>6</sub> H <sub>5</sub>	Pale red solid	70–71	88 <sup>a</sup>	9.01(8.88)	3450, 3200(N-H) 1615(C=N) 1275(P=O)
<b>2m</b>	Me	H	Et/C <sub>6</sub> H <sub>5</sub>	White cryst.	75–76	66 <sup>a</sup>	8.81(8.54)	3400, 3300(N-H) 1615(C=N) 1270(P=O)
<b>2n</b>	H	H	Et/m-OMeC <sub>6</sub> H <sub>4</sub>	Yellow solid	101–103	70 <sup>a</sup>	8.50(8.18)	3350, 3200(N-H) 1610(C=N) 1250(P=O)
<b>2o</b>	Me	H	Et/m-OMeC <sub>6</sub> H <sub>4</sub>	Yellow solid	98–99	63 <sup>a</sup>	8.10(7.89)	3380, 3250(N-H) 1615(C=N) 1260(P=O)
<b>2p</b>	H	H	Et/m-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Red solid	127–128	76 <sup>a</sup>	7.95(7.79)	3400, 3350(N-H) 1615(C=N) 1250(P=O)
<b>2q</b>	Me	H	Et/m-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	Color solid	55–56	62 <sup>a</sup>	7.45(7.52)	3430, 3380(NH) 1615(C=N) 1250(P=O)

a: obtained by b route.

b: The maximum analysis deviations of C and H are C, 0.35; H, 0.10.

TABLE II  $^1\text{H}$  NMR and  $^{31}\text{P}$  NMR Data for Compounds **2**

NO	$^1\text{H}$ NMR $\delta$ ( $\text{CDCl}_3$ , J/Hz)					$^{31}\text{P}$ NMR $\delta$ ( $\text{CDCl}_3$ )
	N-H	Ar-H	Alkyl-CH <sub>3</sub>	Alkyl-CH <sub>2</sub>	Ar-CH <sub>3</sub>	OCH <sub>2</sub> (OCH)
<b>2a</b>	4.81(br,2H)	7.20-7.50(m,4H <sub>s</sub> )	1.34(t,6H,J=7.8)			4.20(m,4H)
<b>2b</b>	4.9(br,2H)	7.10-7.30(m,3H <sub>s</sub> )	1.31(t,6H,J=7.5)		2.40(s,3H)	4.45(m,4H)
<b>2c</b>	5.12(br,2H)	6.80-7.70(m,9H <sub>s</sub> )	1.25(t,6H,J=7.6)			4.19(m,4H)
<b>2d</b>	4.90(br,2H)	7.00-7.72(m,4H <sub>s</sub> )	1.20(m,12H)			4.60(m,2H)
<b>2e</b>	4.90(br,2H)	7.00-7.77(m,3H <sub>s</sub> )	1.21(m,12H)		2.35(s,3H)	4.70(m,2H)
<b>2f</b>	5.02(br,2H)	6.80-7.90(m,9H <sub>s</sub> )	1.25(m,12H)			4.50(m,2H)
<b>2g</b>	4.60(br,2H)	7.08-7.80(m,4H <sub>s</sub> )	0.90(t,6H,J=7.6)	1.25-1.35(m,4H <sub>s</sub> ), 1.57-1.70(m,4H <sub>s</sub> )		4.15(m,4H)
<b>2h</b>	4.70(br,2H)	7.10-7.73(m,3H <sub>s</sub> )	0.91(t,6H,J=7.6)	1.20-1.35(m,4H <sub>s</sub> ), 1.67-1.75(m,4H <sub>s</sub> )	2.41(s,3H)	4.35(m,4H)
<b>2i</b>	5.01(br,2H)	7.30-7.90(m,9H <sub>s</sub> )	0.92(t,6H,J=7.5)	1.12-1.43(m,4H <sub>s</sub> ), 1.58-1.69(m,4H <sub>s</sub> )		4.42(m,4H)
<b>2j</b>	4.80(br,2H)	7.00-7.80(m,8H <sub>s</sub> )	1.20(t,3H,J=7.5)		2.48(s,3H)	4.30(m,2H)
<b>2k</b>	4.80(br,2H)	7.00-7.60(m,7H <sub>s</sub> )	1.25(t,3H,J=7.5)		2.43(s,3H) 2.53(s,3H)	4.05(m,2H)
<b>2l</b>	4.75(br,2H)	7.50-8.2(m,9H <sub>s</sub> )	1.25(t,3H,J=7.6)			4.35(m,2H)
<b>2m</b>	4.65(br,2H)	7.10-7.80(m,8H <sub>s</sub> )	1.26(t,3H,J=7.3)		2.40(s,3H)	4.25(m,2H)
<b>2n</b>	4.80(br,2H)	6.80-7.80(m,8H <sub>s</sub> )	1.25(t,3H,J=7.4)		3.80(s,3H,Ar-OCH <sub>3</sub> )	4.31(m,2H)
<b>2o</b>	4.95(br,2H)	6.90-7.70(m,7H <sub>s</sub> )	1.26(t,3H,J=7.3)		2.25(s,3H) 3.85(s,3H,Ar-OCH <sub>3</sub> )	4.20(m,2H)
<b>2p</b>	5.10(br,2H)	6.80-8.02(m,8H <sub>s</sub> )	1.25(t,3H,J=7.4)			4.22(m,2H)
<b>2q</b>	4.95(br,2H)	6.80-8.10(m,7H <sub>s</sub> )	1.30(t,3H,J=7.2)		2.20(s,3H)	4.35(m,2H)

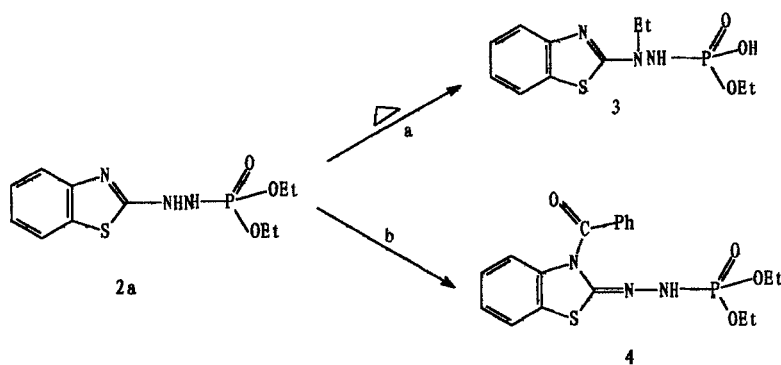
TABLE III <sup>13</sup>C NMR Data for some of Compounds **2**

NO.	<sup>13</sup> C NMR δ (CDCl <sub>3</sub> , ppm)					
	Ar-C	C=N	CH <sub>2</sub>	CH <sub>3</sub>	OCH <sub>2</sub> (OCH)	Ar-CH <sub>2</sub> /Ar-OCH <sub>3</sub>
<b>2a</b>	116.50,126.31,128.80,130.61,139.70,157.82	176.80		17.80	60.13	
<b>2d</b>	118.30,123.91,124.10,126.31,136.30,154.70	172.50		23.40	73.90	
<b>2g</b>	114.70,119.20,125.31,129.85,135.79,155.82	175.81	20.12,35.60	13.21	70.63	
<b>2k</b>	130.85,130.30,126.45,123.20,119.82,118.36; 150.70,135.90,129.73,123.30,120.40,114.38	175.30		17.71	65.91	16.21,20.89
<b>2n</b>	121.01,121.16,120.88,120.60,112.60,123.10; 125.65,126.29,133.20,150.32,121.45,116.30	177.01		16.01	65.50	56.02
<b>2q</b>	140.70,130.65,129.84,127.42,126.40,126.82; 123.80,121.92,120.94,120.80,120.60,115.1	174.30		16.10	66.10	22.50

TABLE IV MS Data of some compounds **2**

NO.	MS(EI) m/e (%)
<b>2a</b>	301(M <sup>+</sup> ,20.68),177(5.00),164(100.00),158(14.97),99(9.64)
<b>2e</b>	343(M <sup>+</sup> ,7.96),205(22.68),178(100.00),161(20.54),125(26.34),99(14.82)
<b>2n</b>	379(M <sup>+</sup> ,25.95),338(25.43),271(100.00),233(14.64),215(10.79),165(20.68),150(72.58),124(10.18)

Compound **2a** under electron impact gave the molecular ion peak  $m/z$  301 ( $M^+$ , 20.68). The base peak corresponded to a fragment at  $m/z$  164, probably was due to the rearrangement of ethyl group shift in the molecular ion. This observation led us to investigate the thermal decomposition of **2a**. Its thermal decomposition at  $180^\circ\text{C}$  caused the shift of ethyl group from oxygen atom to  $\beta$ -nitrogen atom in the same molecule, resulting in formation of the monoester phosphoric hydrazide **3** (Scheme 2), whose structure was characterized by elemental analysis, IR, MS, and  $^1\text{H}$  NMR,  $^{31}\text{P}$  NMR,  $^{13}\text{C}$  NMR spectra (Experimental). The IR spectrum of **3** showed one of absorption band  $1610\text{cm}^{-1}$  due to cyclic conjugated  $\text{C}=\text{N}$  vibration<sup>[11]</sup>, which is different from  $1650\text{cm}^{-1}$  due to acyclic nonconjugated  $\text{C}=\text{N}$  vibration in the product (**4**) of the reaction of **2a** with benzoyl chloride, as shown in Scheme 2.



SCHEME 2

The biological activity of the title products **2** was also tested and some of them showed good inhibitory effects against tobacco mosaic in vitro at 100ppm.

## EXPERIMENTAL

Melting points were uncorrected,  $^1\text{H}$  NMR spectra and  $^{31}\text{P}$  NMR, and  $^{13}\text{C}$  NMR spectra were recorded on a varian XL-200 MHz spectrometer. TMS



was used as an internal standard for NMR, and 85%  $\text{H}_3\text{PO}_4$  was used as an external standard for the  $^{31}\text{P}$  NMR. Mass spectra were measured on a HP 5988A spectrometer. The IR spectra were measured by using a SHIMADZU-408 instrument. Elemental analysis was performed with a PE-2400 elementary analyzer. Column chromatography was performed on silica gel II (10–40  $\mu$ , Hai Yang Chemical Factory of Qingdao). All solvents and materials were reagent grade and purified as required. Dialkyl phosphite, O,O-dialkyl phosphorochloridate, and O-alkyl-O-aryl phosphorochloridate were synthesized according to Ref. 12, 13, 14, respectively.  $\beta$ -(1,3-benzothiazole-2-)-hydrazine **1** was prepared as described in published procedure<sup>[15]</sup>.

### General procedure of the synthesis of $\beta$ -(1,3-benzothiazole-2-)-phosphoric hydrazides **2**

#### Route a

A 150ml round-bottomed flask, fitted with a reflux condenser ( $\text{CaCl}_2$ , tube), and a magnetic stirrer, was charged with 10 mmol of **1**, 40ml of dichloromethane, 15ml of tetrachloromethane, potassium carbonate (4g), and tetrabutyl ammonium bromide (0.5g). The mixture was heated under reflux. After 0.5 h, a solution of 10mmol of dialkyl phosphite and 20ml dichloromethane was added dropwise to the flask. The reactants were refluxed for 8–12 h, then cooled to room temperature, filtered. The filtrate was subsequently concentrated in vacuo. The residual crude product **2** was purified by short column chromatography on silica gel using anhydrous ethyl ether/petroleum ether as eluent. The physical data for compounds **2** are listed in Table I.

#### Route b

50ml anhydrous ethyl ether, 10mmol of **1**, and triethylamine (0.8g) was placed in a 150ml round-bottom flask. After the mixture had been stirred for 10 minutes, a solution of phosphorochloridate (10mmol) in 25ml of anhydrous ethyl ether was added dropwise slowly to the stirred contents. The reaction mixture was warmed and stirred at reflux for 2 h. The ammonium salt was filtered off. The filtrate was washed with water ( $3 \times 40\text{ml}$ ). The separated organic phase was dried with anhydrous magnesium sulfate. The solvent was removed and the residue was chromatographed on silica

gel to give the product **2**. The physical data for compounds **2** are listed in Table I and their spectra data is tabulated in Table II, Table III, Table IV, respectively.

### Procedure for the thermal decomposition of **2a**

A mixture of 3.3mmol of **2a** and phenyl ether was stirred at 180°C under a nitrogen atmosphere for 0.5 h. The mixture was then allowed to cool and product **3** was isolated by chromatograph on silica gel column under reduced pressure using petroleum ether/dry ethyl ether as eluent. **3**, colorless oil,  $n_D^{20}$  1.53894, yield 81%. Anal Calcd for  $C_{11}H_{16}N_3O_3PS$ : C, 43.85; H, 5.32; N, 13.95; P, 10.30. Found: C, 43.72; H, 5.58; N, 13.83; P, 10.57. IR  $\nu$  ( $cm^{-1}$ ): 3480(N - H), 1610(C = N), 2680(P - OH), 1240(P = O).  $^1H$  NMR  $\delta$  ( $CDCl_3$ ): 6.90 ~ 7.82(m, 4H, Ar - H), 6.68(br, 1H, OH), 5.20(br, 1H, N - H), 4.15(m, 2H,  $OCH_2$ ), 2.90(m, 2H,  $CH_2N$ ), 1.25(t, 3H,  $CH_3$ ,  $^3J_{PH} = 7.4Hz$ ), 1.15(t, 3H,  $CH_3$ ,  $^3J_{PH} = 7.2Hz$ ).  $^{31}P$  NMR  $\delta$  ( $CDCl_3$ ): -0.1ppm.  $^{13}C$  NMR  $\delta$  ( $CDCl_3$ ): 178.52, 142.78, 125.90, 122.26, 121.90, 120.64, 108.12, 53.20, 38.81, 17.92, 12.08. MS  $m/z$  (%): 302(M + 1, 31.90).

### Procedure for the reaction of **2a** with benzoyl chloride

A solution of benzoyl chloride (5.5mmol) in 20ml anhydrous ethyl ether was added dropwise to a mixture of **2a** (5.5mmol) and triethylamine(5.5mmol) at room temperature. The reactants were refluxed with stirring for 2h. After filtrated, the filtrate was washed with dry ethyl ether (2  $\times$  10ml) and the organic phase was combined, dried over magnesium sulfate and evaporated under reduced pressure, followed by purifying the residue on silica gel column using dry ethyl ether/petroleum ether mixture (1:2) to yield compound **4**. Light yellow solid, m.p. 162–163, °C, yield 82%. Anal Calcd for  $C_{18}H_{20}N_3O_4PS$ : C, 53.33; H, 4.94; N, 10.37; P, 7.65. Found: C, 53.62; H, 4.75; N, 10.05; P, 7.80. IR  $\nu$  ( $cm^{-1}$ ): 3450(N - H), 1650(C = N), 1680(C = O), 1270(P = O).  $^1H$  NMR  $\delta$  ( $CDCl_3$ ): 6.90 ~ 7.84(m, 4H, ArH), 3.92 ~ 4.24(m, 4H,  $CH_2$ ), 1.26(t, 3H,  $CH_3$ ,  $^3J_{PH} = 7.34Hz$ ).  $^{31}P$  NMR  $\delta$  ( $CDCl_3$ ): -1.5 ppm. MS  $m/z$  (%): 405( $M^+$ , 25.96).

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