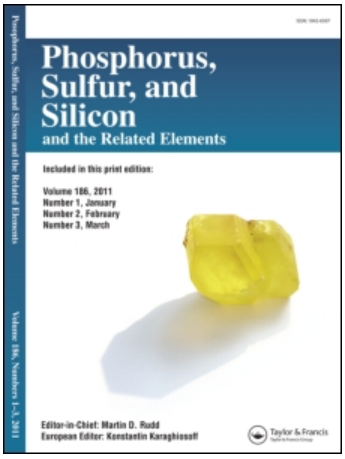

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

HYDRAZONES OF PHOSPHOROHYDRAZIDIC ACID DIETHYL ESTER

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To cite this Article Kraicheva, I.(2001) 'HYDRAZONES OF PHOSPHOROHYDRAZIDIC ACID DIETHYL ESTER', Phosphorus, Sulfur, and Silicon and the Related Elements, 173: 1, 243 — 252
To link to this Article: DOI: 10.1080/10426500108045273
URL: http://dx.doi.org/10.1080/10426500108045273

Phosphorus, Sulfur and Silicon, 2001, Vol. 173, pp. 243-252
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HYDRAZONES OF PHOSPHOROHYDRAZIDIC ACID DIETHYL ESTER

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(Received December 19, 2000)

Novel phosphorus-containing hydrazones: (2-furfurylidene)phosphorohydrazidic acid diethyl ester (**1**), [1-(2-furyl)ethylidene]phosphorohydrazidic acid diethyl ester (**2**), salicylidene phosphorohydrazidic acid diethyl ester (**3**), (4-hydroxybenzylidene) phosphorohydrazidic acid diethyl ester (**4**), [1-(1-naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester (**5**) and [1-(2-naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester (**6**) have been synthesized by condensing phosphorohydrazidic acid diethyl ester with the corresponding aldehydes or ketones. The compounds have been characterized by elemental analysis, TLC, IR, ^{1}H and ^{13}C (1D and COSY) NMR spectroscopy. The IR spectra show the presence of intramolecular and intermolecular hydrogen bonding in **3** and **4**, respectively. The ^{13}C NMR spectra indicate the presence of geometric isomers for compounds **1–6**.

Keywords: phosphorohydrazidic acid diethyl ester; phosphorus-containing hydrazones; TLC; IR spectra; ^{1}H and ^{13}C NMR spectra

INTRODUCTION

There are several reports on the synthesis of organophosphorus derivatives of hydrazine by condensation of phosphorohydrazidic acid dialkyl or diphenyl esters with versatile carbonyl compounds [1–7]. These compounds appear to be attractive products useful for different utilizations [8]. Thus, phosphorus-containing hydrazones participate as intermediates in organic synthesis, especially for the preparation of functionalized heterocyclic derivatives and azines [9–12]. Some organophosphorus hydrazones are starting compounds in the synthesis of pesticides and herbicides [13,

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[14]. Phosphorylated hydrazone derivatives have been explored as fluorescent dyes for polymer fibers [15]. Compounds of this type have also been found to act as potent accelerators for anaerobic adhesive compositions [16]. On the other hand, it is noteworthy that the phosphonate function frequently confers pharmaceutical and phytopharmaceutical activity; in particular, Schiff bases containing hydrazone fragment exhibit a cytostatic effect [8,17,18].

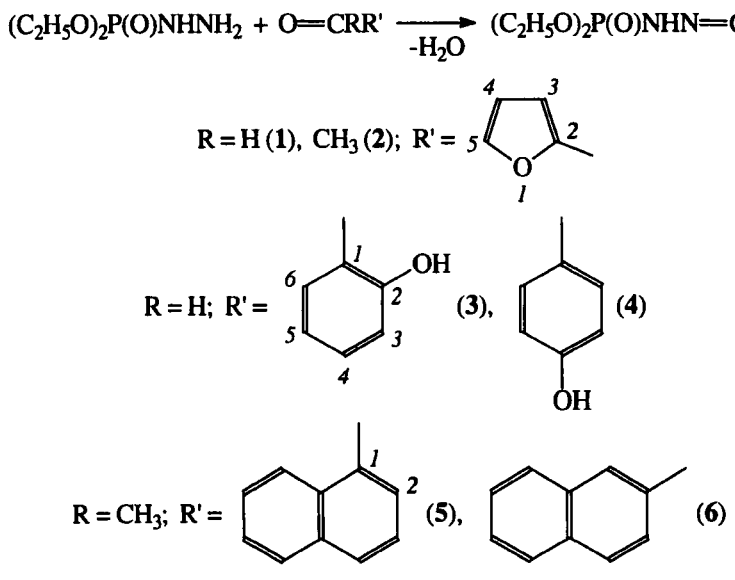
It is of interest to synthesize organophosphorus derivatives of hydrazine as potential candidates for cytostatic activity assays. In the present work the synthesis of novel phosphorus-containing hydrazones is described and their IR and NMR spectra are discussed.

RESULTS AND DISCUSSION

Condensation of phosphorohydrazidic acid diethyl ester was performed with the following carbonyl compounds: 2-furylaldehyde, 2-furyl methyl ketone, salicylaldehyde, 4-hydroxybenzaldehyde, 1-acetonaphthone and 2-acetyl-naphthalene. The reaction proceeds according to Scheme 1. Six novel phosphorus-containing hydrazones were obtained: (2-furfurylidene)phosphorohydrazidic acid diethyl ester (**1**), [(1-(2-furyl)ethylidene]phosphorohydrazidic acid diethyl ester (**2**), salicylidenephosphorohydrazidic acid diethyl ester (**3**), (4-hydroxybenzylidene)phosphorohydrazidic acid diethyl ester (**4**), [1-(1-naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester (**5**) and [1-(2-naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester (**6**).

The reaction was performed with equimolar ratios of the reagents. The phosphorus-containing hydrazones **2–6** were synthesized by refluxing the reaction mixture in ethanol. The reaction of furfural with the hydrazide was carried out under softer conditions, but this did not prevent the formation of resinous by-products.

The hydrazones obtained (**1–6**) are colourless crystalline solids, stable at normal conditions, and soluble in methanol, ethanol, chloroform, dimethylsulfoxide (DMSO) *etc.* The purity of the substances was controlled by thin layer chromatography (TLC). All of them gave single spots in each eluting system.



SCHEME 1

In the infrared spectra of compounds **1–6** taken as KBr disks, the absorption bands of the stretching vibrations of the NH groups were observed in the range of 3185–3098 cm^{-1}. In the spectra of **1–3** and **5** these bands are split into doublets (see Experimental). Similar splitting of the ν(NH) band in the solid state has been registered for other phosphorus-containing hydrazones[1, 5,19] and has been attributed to the presence of rotamers[1]. In the IR spectra of **3** and **4** there are no bands above 3300 cm^{-1}, which suggests the absence of free (non-associated) hydroxyl groups. The band at 2836 cm^{-1} in the spectrum of the salicylaldehyde derivative **3** should be assigned to the stretching vibration of the OH group. The remarkable low-frequency shift of this band might be explained by the association of the OH group with the imine nitrogen atom. The formation of such a stable intramolecular hydrogen bond accompanied with a drastic lowering of the OH stretching is well known among salicylaldimine derivatives [17, 20–22]. It should be noted, however, that in the same range (around 2840 cm^{-1}) the band of the symmetric CH_2 stretching is also expected. The OH stretching band of the *para*-hydroxybenzaldehyde derivative **4** appears at 3253 cm^{-1}. The low-frequency shift of this band is less pronounced as compared to the

ortho-derivative, and this should be attributed to the existence of intermolecular hydrogen bonding [23].

TABLE I ^{1}H-NMR parameters of compounds **1–6** in DMSO-d_6

Compd. No	*Signal assignment*	*Chemical shift (δ, ppm), multiplicity, relative intensity*	*Coupling constants (J, Hz)*
1	CH_3	1.23 and 1.24, 2t, 6H	^{3}J(H,H)=7.06
	OCH_2	4.01, m, 4H	
	H-4	6.55, dd, 1H	^{3}J(H-4,H-3)=3.39; ^{3}J(H-4,H-5)=1.81
	H-3	6.69, d, 1H	^{3}J(H-3,H-4)-3.39
	H-5	7.72, dd, 1H	^{3}J(H-5,H-4)=1.82, ^{4}J(H-5,H-3)=0.60
	HC=N	7.80, s, 1H	
	NH	9.50, d, 1H	^{2}J(P,H)=27.98
2	CH_3	1.23, 2t[a], 6H	^{3}J(H,H)=7.06, 7.07
	CH_3C=N	2.08 s, 3H	
	OCH_2	4.02, m, 4H	
	H-4	6.52, dd, 1H	^{3}J(H-4,H-3)=3.40; ^{3}J(H-4,H-5)=1.79
	H-3	6.71, dd, 1H	^{3}J(H-3,H-4)=3.40; ^{4}J(H-3,H-5)=0.71
	H-5	7.68, dd, 1H	^{3}J(H-5,H-4)=1.75; ^{4}J(H-5,H-3)=0.70
	NH	8.48, d, 1H	^{2}J(P,H)=25.79
3	CH_3	1.23 and 1.24, 2t, 6H	^{3}J(H,H)=7.04, 7.05
	OCH_2	4.03, m, 4H	
	H-3, H-5	6.85, m, 2H	
	H-4	7.21, m, 1H	^{3}J(H-4,H-3)=^{3}J(H-4,H-5)=7.92
	H-6	7.41, m, 1H	^{3}J(H-6,H-5)=7.39
	HC=N	8.15, s, 1H	
	NH	9.66, d, 1H	^{2}J(P,H)=26.82
	OH	10.53, s, 1H	
4	CH_3	1.23, 2t[a], 6H	^{3}J(H,H)=6.98
	OCH_2	4.01, m, 4H	
	H-arom.	7.07, m, 4H	
	HC=N	7.81, s, 1H	
	NH	9.27, d, 1H	^{2}J(P,H)=27.54
	OH	9.81, s, 1H	

Compd. No	*Signal assignment*	*Chemical shift (δ, ppm), multiplicity, relative intensity*	*Coupling constants (J, Hz)*
5	CH_3	1.23 and 1.24, 2t, 6H	$^3J(H,H)=7.05$
	$CH_3C=N$	2.30, s, 3H	
	OCH_2	4.04, m, 4H	
	H-arom.	7.49, m, 4H; 7.93, m, 2H; 8.10, m, 1H	
	NH	8.63, d, 1H	$^2J(P,H)=25.63$
6	CH_3	1.25 and 1.26, 2t, 6H	$^3J(H,H)=7.06$
	$CH_3C=N$	2.29, s, 3H	
	OCH_2	4.08, m, 4H	
	H-arom.	7.51, m, 2H; 7.91, m, 4H; 8.12 br. s, 1H	
	NH	8.64, d, 1H	$^2J(P,H)=26.10$

a. The difference between the centers of the two triplet signals is less than 0.01 ppm.

Proton NMR data of DMSO-d_6 solutions of the compounds **1–6** are collected in Table I. For the aldehyde derivatives (**1, 3** and **4**), the doublet signal of the proton of the NH group appears at a weaker field and has a higher value of the coupling constant $^2J(P,H)$ as compared to the corresponding signal of the ketone derivatives (**2, 5** and **6**). The deuterium exchange of these protons is slow and the intensity of the NH signal decreases considerably, but the signal does not disappear within one day and night. Addition of D_2O causes complete exchange of the OH protons in compounds **3** and **4**. The signals of the CH_3 protons of the ethoxy groups in **1–6** appear as two triplets with very close centers. Two very close triplets ($\Delta\delta<0.01$ ppm) were also observed for the methyl protons of phosphorohydrazidic acid diethyl ester in DMSO-d_6. The multiplet signals of the methylene protons of the two ethoxy groups partially overlap, making impossible to measure their coupling constants with the ^{31}P nucleus after decoupling of the CH_3 protons. The signals of the aromatic protons in the hydrazone **3** were identified with the aid of spin-decoupling and literature data for salicylidene derivatives [17]. Data for ^{13}C chemical shifts of the compounds **1–6** and phosphorohydrazidic acid diethyl ester are given in Table II and Experimental, respectively. The assignment of the signals is based on the analysis of the decoupled ^{13}C NMR, DEPT and CH-COSY spectra of the compounds. A couple of signals was observed for each of

the two carbons of the ethoxy group. The same was registered for the two carbons of the ethyl group in phosphorohydrazidic acid diethyl ester. The C=N fragment also gives a pair of signals ($\Delta\delta$=0.3–0.4 ppm) in the spectra of hydrazones **1–6**. This indicates the existence of geometrical isomers (*syn* and *anti*) of hydrozones **1–6**, similarly to other C=N containing compounds (imines, imino ethers, oximes, semicarbazones and phenylhydrazones) [24–28]. The appearance of a pair of signals for each of the two carbon atoms in the spectrum of phosphorohydrazidic acid diethyl ester is probably due to the high rotational barrier around the P-N bond [29,30].

EXPERIMENTAL

Starting compounds

2-Furaldehyde, 2-furyl methyl ketone, salicylaldehyde, 4-hydroxybenzaldehyde, 1-acetonaphthone and 2-acetyl-naphthalene were commercial products (*purum*). 2-Furaldehyde, salicylaldehyde and the solvents used were purified by distillation. 4-Hydroxybenzaldehyde and 2-acetyl-naphthalene were recrystallized from benzene and petroleum ether, respectively. Phosphorohydrazidic acid diethyl ester was prepared according to Ref. 31. ^{1}H NMR (DMSO-d_6), δ (ppm), J (Hz): 1.21, 2t, 6H, ^{3}J(H,H)=7.07, CH_3; 3.92, m, 4H, ^{3}J(P,H)=7.78, OCH_2; 5.98, d, 1H, ^{2}J(P,H)=31.40, NH. ^{13}C NMR (DMSO-d_6), δ (ppm): 16.4, 16.5, CH_3; 61.9, 62.0, OCH_2.

Apparatus and conditions

The melting points were measured on a Kofler microscope and are uncorrected.

The IR spectra were taken on a Bruker Vector 22 spectrometer as KBr disks. NMR spectra (^{1}H, ^{13}C{^{1}H}, DEPT, H,H-COSY and CH-COSY) were recorded on a Bruker DRX-250 spectrometer (250 MHz) at room temperature; DMSO-d_6 as solvent and TMS as internal standard were used. D_2O exchange was applied to confirm the assignment of NH and OH protons.

TABLE II ^{13}C NMR chemical shifts of compounds **1–6** in DMSO-d_6, δ(ppm)

No	*Signal assignment*								
	$CH_3C{=}N$	CH_3	OCH_2	$C{=}N$	*C-fur.; C-arom.*				
					C-1	*C-2*	*C-3*	*C-4*	*C-5*
	–	15.9	62.2	134.0	–	149.6	111.1	111.7	144.0
		16.0	62.3	134.4					
	13.1	16.5	63.4	141.9	–	152.4	110.4	112.4	144.2
		16.6	63.5	142.2					
	–	16.1	62.6	144.6	116.1	156.3	116.2	130.6	119.5
		16.2	62.7	144.9	119.4	156.4			
	–	16.4	62.6	144.4	126.2	128.1	115.9	158.8	115.9
		16.5	62.7	144.7					
	18.1	16.2	62.5	150.3	125.5[a], 128.5, 128.5[b]; 125.6, 125.9, 126.1, 126.5[c]; 130.4, 133.6, 138.0[d]				
		16.3	62.6	150.6					
	12.9	16.2	62.6	148.1	125.3[a]; 126.6, 126.6[b]; 123.4, 127.6, 127.8, 128.5[c]; 133.0, 133.0, 136.0[d]				
		16.3	62.7	148.4					

ıe signals correlate with the multiplet signals for one, two and four aromatic protons, respectively.
of non-protonated aromatic carbon atoms.

The thin layer chromatograms were performed on Kieselgel-60 F_{254}plastic sheets (Merck) at room temperature. The samples were applied as methanolic solutions. The chromatograms were developed ascendingly using the following solvent systems: a) benzene/methanol (3:1), b) benzene/methanol (10:1), c) ethylacetate saturated with water, d) benzene/acetonitrile (3:2) saturated with water. The spots were detected under UV light and in iodine vapour atmosphere.

Preparation of compounds 1–6

The compounds **2–6** were prepared by the following procedure. A mixture of 2.20 g (13.11 mmol) of phosphorohydrazidic acid diethyl ester, a stoichiometric amount of the corresponding carbonyl compound (2-furyl methyl ketone, salicylaldehyde, 4-hydroxybenzaldehyde, 1-acetonaphthone and 2-acetyl-naphthalene) and 15 ml ethanol was stirred for two hours at room temperature, then further two hours at 70–75°C. In the case of **2** the reaction was carried out under a nitrogen atmosphere. Evaporation of the solvent gave crude solids (**2–6**) which were recrystallized from cyclohexane (**2, 3, 5** and **6**) or water (**4**) to give colourless crystalline powders.

The compound **1** was prepared by reacting stoichiometric amounts (13.11 mmol) of phosphorohydrazidic acid diethyl ester and 2-furaldehyde in diethyl ether at room temperature under nitrogen. Diethyl ether was removed, the residue was dissolved in hot benzene and the solution was filtered through Al_2O_3. After evaporation of benzene, the crude product was recrystallized from cyclohexane.

(2-Furfurylidene)phosphorohydrazidic acid diethyl ester, 1

Yield: 1.26 g (39%); m. p.: 106–107°C.; R_f=0.68 (a), 0.50 (b), 0.51 (c), 0.34 (d).

Analysis. Calcd. for $C_9H_{15}N_2O_4P$: N: 11.38 %, P: 12.60 %; found: N: 11.14 %, P: 12.79 %.

IR (KBr disk), $\tilde{\nu}$(cm^{-1}): 3146, 3120 (ν_{NH}); 1630 ($\nu_{C=N}$); 1579, 1554, 1497 ($\nu_{C=C(furan)}$); 1239 ($\nu_{P=O}$); 1023 ($\nu_{P\text{-}OEt}$).

[1-(2-Furyl)ethylidene]phosphorohydrazidic acid diethyl ester, 2

Yield: 2.04 g (60 %); m. p.: 97–98°C; R_f=0.71 (a), 0.54 (b), 0.52 (c), 0.38 (d).

Analysis. Calcd. for $C_{10}H_{17}N_2O_4P$: N: 10.77 %, P: 11.92 %; found: N: 10.91 %, P: 11.90 %.

IR (KBr disk), $\nu^{-}(cm^{-1})$: 3173, 3117 (ν_{NH}); 1637 ($\nu_{C=N}$); 1603, 1569, 1493 ($\nu_{C=C(furan)}$); 1245 ($\nu_{P=O}$); 1040 ($\nu_{P\text{-}OEt}$).

Salicylidenephosphorohydrazidic acid diethyl ester, 3

Yield: 2.53 g (71 %); m. p.: 84–85 °C; R_f=0.73 (a), 0.54 (b), 0.56 (c), 0.42 (d).

Analysis. Calcd. for $C_{11}H_{17}N_2O_4P$: N: 10.29 %, P: 11.40 %; found: N: 10.06 %, P: 11.22 %.

IR (KBr disk), $\nu^{-}(cm^{-1})$: 3123, 3098 (ν_{NH}); 2836 (ν_{OH}); 1620 ($\nu_{C=N}$), 1606, 1580, 1500, 1490 ($\nu_{C=C(arom.)}$); 1230 ($\nu_{P=O}$); 1043 ($\nu_{P\text{-}OEt}$).

(4-Hydroxybenzylidene)phosphorohydrazidic acid diethyl ester, 4

Yield: 0.96 g (27 %); m. p.: 129–130 °C; R_f=0.67 (a), 0.34 (b), 0.50 (c), 0.24 (d).

Analysis. Calcd. for $C_{11}H_{17}N_2O_4P$: N: 10.29 %, P: 11.40 %; found: N: 10.11 %, P: 11.31 %.

IR (KBr disk), $\nu^{-}(cm^{-1})$: 3253 (ν_{OH}); 3185 (ν_{NH}); 1610 ($\nu_{C=N}$); 1584, 1519, 1465 ($\nu_{C=C(arom.)}$); 1225, 1208 ($\nu_{P=O}$); 1030 ($\nu_{P\text{-}OEt}$).

[1-(1-Naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester, 5

Yield: 1.89 g (45 %); m. p.: 124–125 °C; R_f=0.74 (a), 0.58 (b), 0.55 (c), 0.42 (d).

Analysis. Calcd. for $C_{16}H_{21}N_2O_3P$: N: 8.75 %, P: 9.69 %; found: N: 9.12 %, P: 9.65 %.

IR (KBr disk), $\nu^{-}(cm^{-1})$: 3160, 3146 (ν_{NH}); 1611 ($\nu_{C=N}$); 1508, 1441 ($\nu_{C=C(arom.)}$); 1247 ($\nu_{P=O}$); 1045 ($\nu_{P\text{-}OEt}$).

[1-(2-Naphthyl)ethylidene]phosphorohydrazidic acid diethyl ester, 6

Yield: 2.85 g (68 %); m.p.: 117–118°C; R_f=0.73 (a), 0.59 (b), 0.55 (c), 0.43 (d).

Analysis. Calcd. for $C_{16}H_{21}N_2O_3P$: N: 8.75 %, P: 9.69%; found: N: 9.15 %, P: 9.51 %.

IR (KBr disk), $\nu^{-}(cm^{-1})$: 3172 (ν_{NH}); 1604 ($\nu_{C=N}$); 1500, 1476, 1437 ($\nu_{C=C(arom.)}$); 1240 ($\nu_{P=O}$); 1034 ($\nu_{P\text{-}OEt}$).

^{1}H NMR data of compounds **1–6** are presented in Table I and their ^{13}C NMR chemical shifts are given in Table II.

Acknowledgements

The author thanks the UNESCO Division of Life Sciences for the financial support (Contract SC 2065790).

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