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A CONVENIENT SYNTHESIS OF NOVEL 6-ARYL-5-PHENYL-5-OXIDO-6,7-DIHYDRO[1,3,4,2]THIADIAZAPHOSPHOLO[5,4-*c*][1,2,4,5]-TRIAZAPHOSPHININES

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Abstract – A convenient method for design of novel 6-aryl-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]triazaphosphinines was achieved. This method depended on three-component reaction of 5-hydrazino-1,3,4,2-thiadiaza-phosphole with aromatic aldehydes and phenyldichlorophosphine in dry THF. The methodology was easy, efficient, catalyst-free and good synthetic procedure.

Phosphorus-nitrogen compounds have gained considerable attention over the past three decades due to their biological and pharmacological effects such as anticancer,[1] insecticidal,[2] and herbicidal properties.[3] The nitrogen- and sulfur-containing heterophospholes, especially including N-P-S unit, have been less studied, primarily due to difficult synthesis and intrinsically due to the weakness of the P-S bond.[4] 1,3,4,2-Thiadiazaphospholes are useful in the syntheses of insecticides and acaricides.[5] However, they can be formed by reactions of substituted thiohydrazides with PCl_3, $PSCl_3$, and $P(NR_2)_3$.[6] On the other hand, the triazaphosphinine systems as an important class of phosphorus-nitrogen heterocycles are rare in the literature.[7] The triazaphosphinine class has possible six isomers as shown in Figure 1. To our best knowledge, the isomers of 1,2,3,4-triazaphosphinine (**I**),[8-10] 1,2,4,3-triazaphosphinine (**IV**)[11,12] and 1,3,5,2-triazaphosphinine (**VI**)[13-21] have been reported. Previously, we constructed some isomers of 1,2,4,5-triazaphosphinine (**III**) fused with pyridine, pyrimidine and 1,2,4-triazine moieties.[22-24] In continuation of our work for design of novel phosphorus compounds,[25-27] we herein report an efficient

approach for synthesis of some novel 1,3,4,2-thiadiazaphospholo[5,4-*c*][1,2,4,5]triazaphosphinines. The methodology depends on using 5-hydrazino-1,3,4,2-thiadiazaphosphole with aromatic aldehydes and phenyldichlorophosphine in dry THF in one-pot under mild reaction conditions.

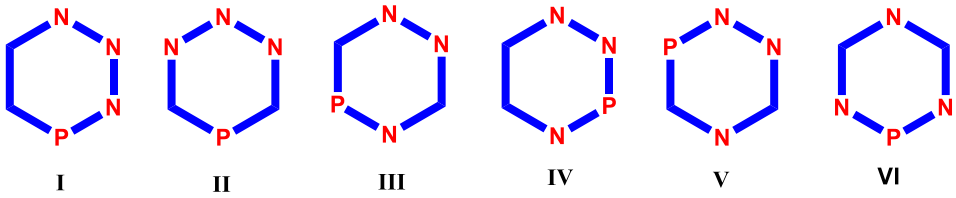
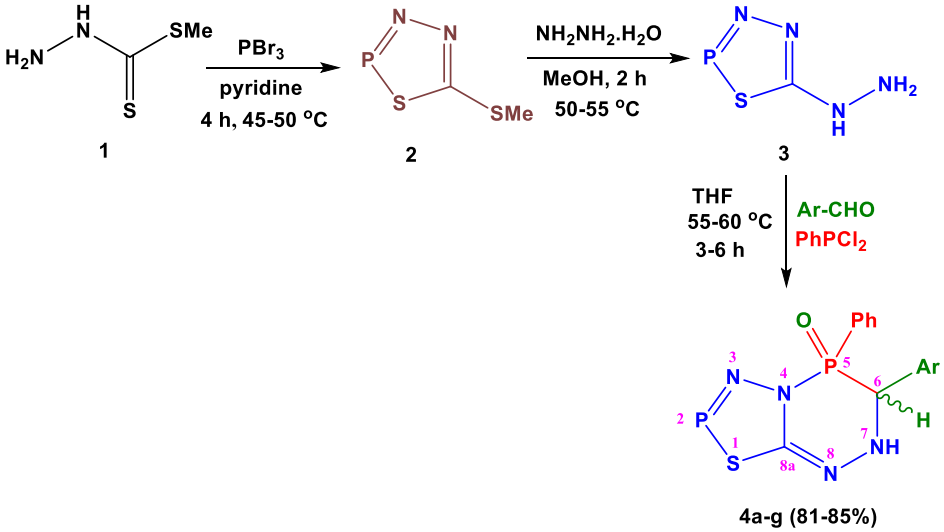


Figure 1. The possible six isomers of triazaphosphinine systems

The known methyl hydrazinecarbodithioate (**1**)[28] was cyclized with phosphorus tribromide in dry pyridine to give 5-metylthio-1,3,4,2-thiadiazaphosphole (**2**) in good yield (Scheme 1). Hydrazinolysis of 5-methylthio-1,3,4,2-thiadiazaphosphole (**2**) with an equimolar amount of hydrazine hydrate in methanol under reflux gave the novel 5-hydrazino-1,3,4,2-thiadiazaphosphole (**3**) (Scheme 1). The IR spectrum of compound **3** recorded the absorption bands for NH_2 and NH groups at 3325, 3194 and 3132 cm^{-1}, respectively. Its ^{1}H-NMR spectrum displayed the protons of NH_2 and NH as two singlets at δ 5.02 and 10.13 ppm, respectively. Furthermore, its ^{13}C-NMR spectrum recorded the only one carbon atom at δ 151.9 (C−5) ppm. The mass spectrum of compound **3** showed its molecular ion peak at *m/z* 134 (M^+, 15%).



Scheme 1

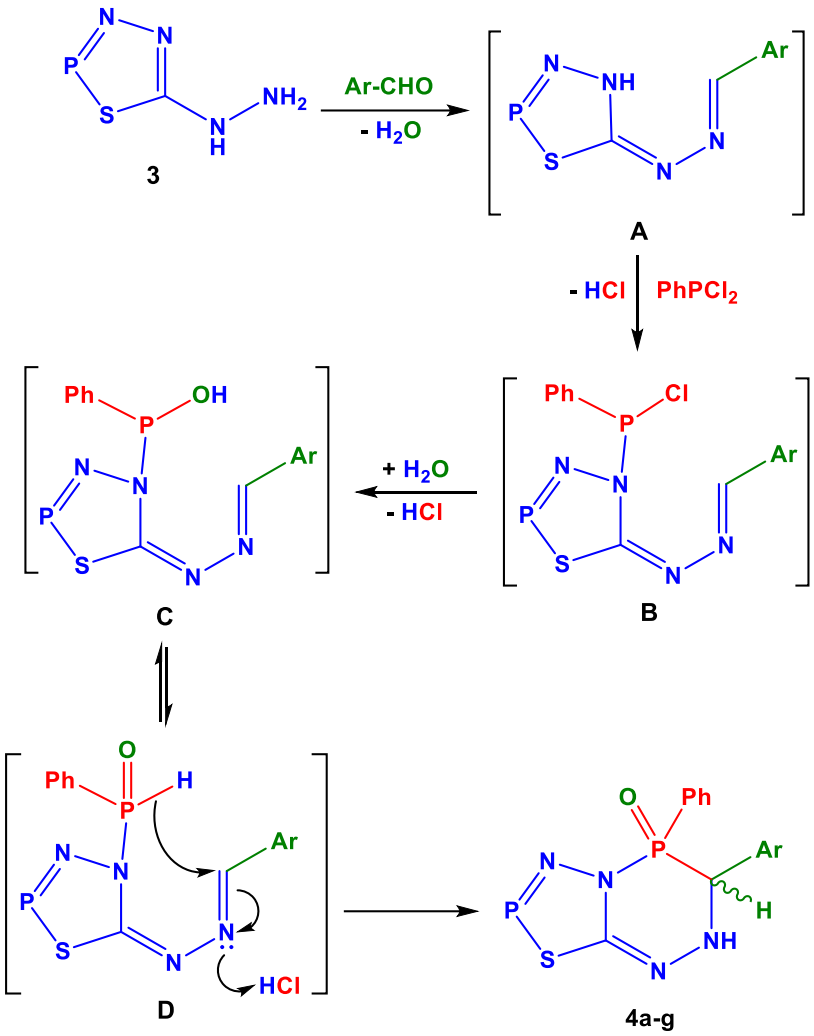
The presence of two active nucleophilic sites such as NH_2 and NH in the substrate **3** offers alternative reaction possibilities with electrophilic carbon and phosphorus reagents.[29,30] Thus, a mixture of compound

3, aromatic aldehydes and phenyldichlorophosphine in dry THF was heated under reflux at 55−60 °C for 3−6 h to furnish 6-aryl-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]-triazaphosphinines (**4a-g**) in excellent yields (Scheme 1 and Table 1). The reactions were carried out using one-pot procedure. All the products were isolated from the reaction mixture by crystallization process and checked by TLC. The structure of compounds **4a-g** was deduced from their IR, NMR, MS spectra and elemental analyses. The IR spectra of the title products **4a-g** exhibited the characteristic absorption bands for NH and P=O functions at 3123−3211 and 1206−1219 cm^{-1}, respectively. Also, their ^{1}H-NMR spectra displayed the specific NH as singlets at δ 9.02−9.50 ppm. The P-CH protons resonated as doublets at δ 4.01−4.22 (J_{PCH}=20.4−22.6 Hz). Furthermore, the carbon chemical shifts for compounds **4a-g** exhibited the carbon atoms of C−8a at δ 150.4−152.8 ppm. There are two carbon atoms appeared as doublets at regions δ 51.6−55.3 ppm (C−6, J_{PC}=133.6−144.5 Hz) and 136.1−139.1 ppm (C−1_{phenyl}, J_{PC}=156.0−162.3 Hz). The ^{31}P-NMR spectra of these products were resonated in region δ 23.94−27.46 (triazaphosphinine) and 78.34−81.64 (thiadiazaphosphole) ppm.[23,31]

Table 1. Synthesis of 6-aryl-5-phenyl-5-oxido-6,7-dihydro-[1,3,4,2]thiadiazaphospholo[5,4-c][1,2,4,5]-triazaphosphinines (**4a-g**)

Entry	Ar	Product	Time (h)	Yield (%)
1	C_6H_5	**4a**	6	81
2	2-ClC_6H_4	**4b**	5	84
3	4-MeC_6H_4	**4c**	5	81
4	2,4-$Cl_2C_6H_3$	**4d**	3	85
5	2,4-$(MeO)_2C_6H_3$	**4e**	6	82
6	3-Br-4-ClC_6H_3	**4f**	5	82
7	3-Cl-4-HOC_6H_3	**4g**	5	81

Based on our observation and knowledge found in the literature, we proposed a likely reaction mechanism for the formation of 6-aryl-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*]-[1,2,4,5]triazaphosphinines (**4a-g**) (Scheme 2). The substrate **3** was easily condensed with aromatic aldehydes forming the corresponding Schiff bases **A**. These Schiff bases underwent another condensation process of phenyldichlorophosphine with the *endo* imino group through removal of one molecule of hydrogen chloride to give the intermediates **B**. The labelled chlorine atoms in the intermediates **B** were hydrolyzed into the nonisolable intermediates **C** by water molecules that resulted from first condensation process. These intermediates **C** could be existed in another forms **D** as a result of hydroxyphosphane-phosphine oxide tautomerism.[32] The phosphorus atom in the intermediates **C** or **D** could attack the azomethine bond according to Pudovik reaction to yield the target products **4a-g** (Scheme 2).[33]



Scheme 2

EXPERIMENTAL

The melting points were measured on a digital Stuart SMP-3 apparatus in an open capillary tube. Infrared spectra were measured on FT-IR spectrophotometer (Nicolet iS10) using KBr disks. The NMR spectra were recorded on a Bruker 600 MHz instrument in DMSO using TMS as an internal standard. Mass spectra were recorded on direct probe controller inlet part to single quadropole mass analyzer in (Thermo Scientific GCMS). Elemental microanalysis was performed on Perkin-Elmer 2400II at the Chemical War department, Ministry of Defense, Egypt. The purity of the synthesized compounds was checked by thin layer chromatography (TLC) and elemental microanalysis.

Synthesis of 5-hydrazino-1,3,4,2-thiadiazaphosphole (**3**).

Phosphorus tribromide (0.25 mL, 2.5 mmol) was added to a solution of methyl hydrazinecarbodithioate (**1**) (0.30 mL, 2.5 mmol) in dry pyridine at 5–10 ºC for 1 h then heated at 45–50 ºC for 3 h. The mixture was cooled and poured into cold ice-water. The mixture was neutralized with diluted HCl (30%) under stirring for 1 h. The formed solid was filtered off, washed with water, and crystallized from MeOH to

give 5-methylthio-1,3,4,2-thiadiazaphosphole (**2**) as buff solid in 72% yield, mp 111−113 ºC. Then a mixture of 5-methylthio-1,3,4,2-thiadiazaphosphole (**2**) (0.37 g, 2.5 mmol) and hydrazine hydrate (0.13 mL, 2.5 mmol) in MeOH, was heated under reflux for 2 h. The separated solid was filtered, washed with Et_2O and recrystallized from MeOH to give 5-hydrazino-1,3,4,2-thiadiazaphosphole (**3**) as beige solid in 65% yield, mp 158−159 ºC. IR (KBr), (*v* max, cm^{-1}): 3325, 3194, 3132 (NH_2, NH), 1610 (C=N). ^{1}H-NMR (600 MHz, DMSO-d_6): 5.02 (s, 2H, NH_2), 10.13 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 151.9 (C–5). ^{31}P-NMR (242 MHz, DMSO-d_6): 78.02. MS (*m/z*, I%): 134 (M^+, 15%). Anal. Calcd for CH_3N_4PS (134.10): C, 8.96%; H, 2.26%; N, 41.78%; S, 23.91%. Found: C, 8.80%; H, 2.09%; N, 41.13%; S, 23.72%.

General procedure for the synthesis of the target products 4a-g.

A mixture of 5-hydrazino-1,3,4,2-thiadiazaphosphole (**3**) (0.33 g, 2.5 mmol) and aromatic aldehyde (2.5 mmol) in dry THF (20 mL) was heated under reflux at 55−60 ºC for 1 h. A solution of phenyldichloro-phosphine (0.35 mL, 2.5 mmol) in THF (3 mL) was added and the mixture was further heated at 55−60 ºC for 2−5 h. After completion of the reaction, the solvent was removed under pressure. The residue was recrystallized from diluted EtOH to obtain the final compounds.

5,6-Diphenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]triazaphosphinine (**4a**): Yield 81%, pale brown solid, mp 194−196 ºC. IR (KBr), (*v* max, cm^{-1}): 3211 (NH), 1598 (C=N), 1219 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 4.13 (d, 1H, , J_{PCH}=22.6 Hz, H–6), 7.25–7.30 (m, 4H, Ar–H), 7.59–7.63 (m, 2H, Ar–H), 7.70–7.74 (m, 2H, Ar–H), 8.10–8.14 (m, 2H, Ar–H), 9.02 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 55.0 (d, J_{PC}=136 Hz, C–6), 123.9 (C–4_{phenyl}), 124.6 (C–4_{aryl}), 125.9 (C–$3,5_{phenyl}$), 126.7 (C–$2,6_{aryl}$), 128.6 (C–$3,5_{aryl}$), 129.0 (C–$2,6_{phenyl}$), 134.5 (C–1_{aryl}), 136.1 (d, J_{PC}=162.3 Hz, C–1_{phenyl}), 152.4 (C–8a). ^{31}P-NMR (242 MHz, DMSO-d_6): 25.43 and 78.34. MS (*m/z*, I%): 346 (M^+, 13%). Anal. Calcd for $C_{14}H_{12}N_4OP_2S$ (346.28): C, 48.56%; H, 3.49%; N, 16.18%; S, 9.26%. Found: C, 48.39%; H, 3.38%; N, 16.03%; S, 9.08%.

6-(2-Chlorophenyl)-5-phenyl-5-oxido-6,7-dihydro-[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]-triazaphosphinines (**4b**): Yield 84%, beige solid, mp 168−170 ºC. IR (KBr), (*v* max, cm^{-1}): 3198 (NH), 1603 (C=N), 1219 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 4.22 (d, 1H, H–6, J_{PCH}=21.5 Hz), 6.89–6.93 (m, 2H, Ar–H), 7.27–7.32 (m, 3H, Ar–H), 7.60 (t, 2H, *J*=7.4 Hz, Ar–H), 7.71 (t, 1H, *J*=7.4 Hz, Ar–H), 8.13 (d, 1H, *J*=8.4 Hz, Ar–H), 9.50 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 52.6 (d, J_{PC}=133.6 Hz, C–6), 120.6 (C–4_{phenyl}), 123.9 (C–5_{aryl}), 125.7 (C–$3,5_{phenyl}$), 127.2 (C–$2,6_{phenyl}$), 128.4 (C–3_{aryl}), 129.2 (C–6_{aryl}), 129.8 (C–4_{aryl}), 131.8 (C–2_{aryl}), 140.1 (C–1_{aryl}), 136.3 (d, J_{PC}=161.0 Hz, C–1_{phenyl}), 150.4 (C–8a). ^{31}P-NMR (242 MHz, DMSO-d_6): 27.01 and 79.12. MS (*m/z*, I%): 382 (M+2, 9%), 380 (M^+, 28%). Anal. Calcd for $C_{14}H_{11}ClN_4OP_2S$ (380.73): C, 44.17%; H, 2.91%; N, 14.72%; S, 8.42%. Found: C, 44.02%; H, 2.73%; N, 14.59%; S, 8.24%.

6-(4-Methylphenyl)-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]-triazaphosphinine (**4c**): Yield 81%, pale brown solid, mp 181−182 ºC. IR (KBr), (*v* max, cm^{-1}): 3201 (NH), 1605 (C=N), 1214 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 2.10 (s, 3H, Me), 4.16 (d, 1H, J_{PC}=21.0 Hz, H–6), 6.73–6.83 (m, 3H, Ar–H), 7.06–7.10 (m, 2H, Ar–H), 7.60–7.65 (m, 3H, Ar–H), 8.12 (d, 1H, *J*=8.0 Hz, Ar–H), 9.43 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 20.1 (Me), 55.3 (d, J_{PC}=138.0 Hz, C–6), 121.4 (C–4_{phenyl}), 125.6 (C–$2,6_{aryl}$), 126.7 (C–$3,5_{phenyl}$), 127.0 (C–$3,5_{aryl}$), 128.6 (C–$2,6_{phenyl}$), 132.4 (C–1_{aryl}), 136.8 (C–4_{aryl}), 138.1 (d, C–1_{phenyl}, J_{PC}=156.0 Hz), 151.5 (C–8a). ^{31}P-NMR (242 MHz, DMSO-d_6): 26.02 and 79.62. MS (*m/z*, I%): 360 (M^+, 12%). Anal. Calcd for $C_{15}H_{14}N_4OP_2S$ (360.31): C, 50.00%; H, 3.92%; N, 15.55%; S, 8.92%. Found: C, 49.83%; H, 3.74%; N, 15.37%; S, 8.79%.

6-(2,4-Dichlorophenyl)-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]-triazaphosphinine (**4d**): Yield 85%, pale brown solid, mp 193−194 ºC. IR (KBr), (*v* max, cm^{-1}): 3186 (NH), 1601 (C=N), 1206 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 4.02 (d, 1H, J_{PCH}=21.2 Hz, H–6), 6.78 (s, 1H, Ar–H), 7.47–7.55 (m, 3H, Ar–H), 7.66–7.78 (m, 3H, Ar–H), 8.08 (d, 1H, *J*=8.0 Hz, Ar–H), 9.13 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 52.6 (d, J_{PC}=144.0 Hz, C–6), 122.8 (C–4_{phenyl}), 124.6 (C–5_{aryl}), 126.7 (C–$2,6_{phenyl}$), 128.2 (C–$3,5_{phenyl}$), 129.4 (C–6_{aryl}), 131.2 (C–3_{aryl}), 134.6 (C–4_{aryl}), 136.2 (C–2_{aryl}), 139.1 (d, J_{PC}=160.5 Hz, C–1_{phenyl}), 141.0 (C–1_{aryl}), 151.8 (C–8a). ^{31}P-NMR (242 MHz, DMSO-d_6): 27.46 and 80.05. MS (*m/z*, I%): 416 (M+2, 3%), 414 (M^+, 10%). Anal. Calcd for $C_{14}H_{10}ClN_4OP_2S$ (415.17): C, 40.50%; H, 2.43%; N, 13.50%; S, 7.72%. Found: C, 40.34%; H, 2.31%; N, 13.31%; S, 7.58%.

6-(2,4-Dimethoxyphenyl)-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,4,5]-triazaphosphinine (**4e**): Yield 82%, brown solid, mp 138−140 ºC. IR (KBr), (*v* max, cm^{-1}): 3123 (NH), 1600 (C=N), 1206 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 3.75 (s, 3H, OMe), 3.78 (s, 3H, OMe), 4.29 (d, 1H, J_{PCH}=20.4 Hz, H–6), 6.52 (s, 1H, Ar–H), 7.33–7.38 (m, 3H, Ar–H), 7.55–7.58 (m, 3H, Ar–H), 8.11 (d, 1H, *J*=8.0 Hz, Ar–H), 9.03 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 51.6 (d, J_{PC}=144.5 Hz, C–6), 54.8 (MeO), 55.1 (MeO), 104.6 (C–3_{aryl}), 108.4 (C–5_{aryl}), 115.4 (C–1_{aryl}), 122.6 (C–4_{phenyl}), 126.6 (C–$3,5_{phenyl}$), 128.3 (C–$2,6_{phenyl}$), 130.2 (C–6_{aryl}), 135.3 (d, J_{PC}=156.5 Hz, C–1_{phenyl}), 152.8 (C–8a), 160.3 (C–4_{aryl}), 162.0 (C–2_{aryl}). ^{31}P-NMR (242 MHz, DMSO-d_6): 23.94 and 80.98. MS (*m/z*, I%): 406 (M^+, 22%). Anal. Calcd for $C_{16}H_{16}N_4O_3P_2S$ (406.34): C, 47.29%; H, 3.97%; N, 13.79%; S, 7.89%. Found: C, 47.11%; H, 3.82%; N, 13.60%; S, 7.73%.

6-(3-Bromo-4-chlorophenyl)-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*]-[1,2,4,5]triazaphosphinine (**4f**)**:** Yield 82%, cumin solid, mp 162−164 ºC. IR (KBr), (*v* max, cm^{-1}): 3202 (NH), 1597 (C=N), 1208 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6): 4.01 (d, 1H, J_{PCH}=21.0 Hz, H–6), 7.24 (d, 2H, *J*=8.4 Hz, Ar–H), 7.33–7.40 (m, 4H, Ar–H), 7.49 (t, 1H, *J*=7.2 Hz, Ar–H), 8.11 (d, 1H, *J*=8.4 Hz, Ar–H), 9.08 (s, 1H, NH). ^{13}C-NMR (150 MHz, DMSO-d_6): 52.2 (d, J_{PC}=140 Hz, C–6), 117.2

(C–3_{aryl}), 121.8 (C–4_{phenyl}), 125.8 (C–6_{aryl}), 129.3 (C–$3,5_{phenyl}$), 129.9 (C–$2,6_{phenyl}$), 131.4 (C–4_{aryl}), 133.5 (C–5_{aryl}), 135.1 (C–2_{aryl}), 135.9 (C–1_{aryl}), 136.8 (d, J_{PC}=158.0 Hz, C–1_{phenyl}), 150.8 (C–8a). ^{31}P-NMR (242 MHz, DMSO-d_6): 26.03 and 81.23. MS (*m/z*, I%): 462 (M+4, 20%), 460 (M+2, 34%), 458 (M^+, 14%). Anal. Calcd for $C_{14}H_{10}BrClN_4OP_2S$ (459.62): C, 36.59%; H, 2.19%; N, 12.19%; S, 6.98%. Found: C, 36.42%; H, 2.10%; N, 12.02%; S, 6.82%.

6-(3-Chloro-5-hydroxyphenyl)-5-phenyl-5-oxido-6,7-dihydro[1,3,4,2]thiadiazaphospholo[5,4-*c*][1,2,-4,5]triazaphosphinine (**4g**): Yield 81%, pale brown solid, mp 165−166 °C. IR (KBr), (*v* max, cm^{-1}): 3204 (NH), 1603 (C=N), 1212 (P=O). ^{1}H-NMR (600 MHz, DMSO-d_6: 4.09 (d, 1H, J_{PCH}=21.4 Hz, H–6), 6.74 (s, 1H, Ar–H), 7.14 (t, 2H, *J*=8.4 Hz, Ar–H), 7.55–7.59 (m, 2H, Ar–H), 7.64–7.67 (m, 2H, Ar–H), 8.10 (d, 1H, *J*=8.4 Hz, Ar–H), 9.07 (s, 1H, NH), 10.37 (s, 1H, OH). ^{13}C-NMR (150 MHz, DMSO-d_6): 51.6 (d, J_{PC}=139 Hz, C–6), 116.2 (C–4_{aryl}), 117.5 (C–6_{aryl}), 119.3 (C–2_{aryl}), 123.6 (C–4_{phenyl}), 128.4 (C–$3,5_{phenyl}$), 129.8 (C–$2,6_{phenyl}$), 135.2 (C–3_{aryl}), 137.2 (d, J_{PC}=161.2 Hz, C–1_{phenyl}), 140.2 (C–1_{aryl}), 151.4 (C–8a), 160.8 (C–5_{aryl}). ^{31}P-NMR (242 MHz, DMSO-d_6): 25.87 and 81.64. MS (*m/z*, I%): 398 (M+2, 4%), 396 (M^+, 13%). Anal. Calcd for $C_{14}H_{11}ClN_4O_2P_2S$ (396.73): C, 42.39%; H, 2.79%; N, 14.12%; S, 8.08%. Found: C, 42.21%; H, 2.69%; N, 13.96%; S, 7.92%.

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