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A CONVENIENT ONE-POT SYNTHESIS OF NOVEL 1,4-DIHYDRO-1,2-BENZOXAPHOSPHININO[4,3-*c*]PYRAZOLES

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Abstract – We have synthesized novel examples of 1,2-benzoxaphosphinino-[4,3-*c*]pyrazoles in one-pot. The methodology depends on the cyclization of 5-(2-hydroxyphenyl)-1-phenyl-1*H*-pyrazole with different examples of phosphorus halides and sulfides. The possible reaction mechanisms of the formation of these products were displayed and discussed. The isolated products were established by elemental analyses and spectral tools.

Heterocyclic compounds play an important role in the development of pharmaceuticals, natural resources, agriculture products and dyes. Therefore, the researchers of heterocycles still do their best to designing the novel synthetic methodologies for the advancement of new generation of heterocycles.[1,2] The pyrazoles are interesting group of heterocyclic compounds due to their important functions in nature and their pharmacological applications.[3] Their derivatives are most valuable pharmacologic compounds possessing a range of significant biological properties such as anti-inflammatory,[4] antimicrobial,[5] anticancer[6] and antiviral activities.[7] On the other hand, 2-oxido(sulfido)-1,2-benzoxaphosphinines (phosphacoumarin) are phosphorus analogues of 2-oxo-2*H*-1-benzopyran (coumarin) compounds, that have recently an important attention in synthetic strategies, structural modification, and relative reactivity.[8-10] The introduction of the phosphorus atom into coumarin (structure **A**) to form phosphacoumarin (structures **B** and **C**) resulted in the development of new molecules with biological activities (Figure 1).[11] Some phosphacoumarins possess important bioactivities such as anti-inflammatory, antioxidant properties and inhibiting histone deacetylase.[12,13] In view of the importance of phosphacoumarins and pyrazoles as two categories of molecular frameworks, and as a continuation of our efforts in the development of new strategies for the efficient synthesis of phosphorus heterocycles fused heterocyclic skeletons,[14-18] herein, we describes a new simple method for the synthesis of phosphacoumarins fused with a pyrazole moiety.

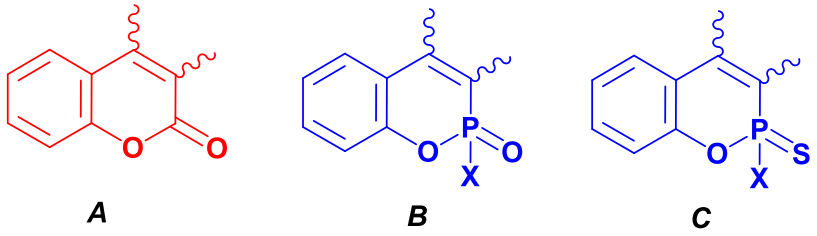
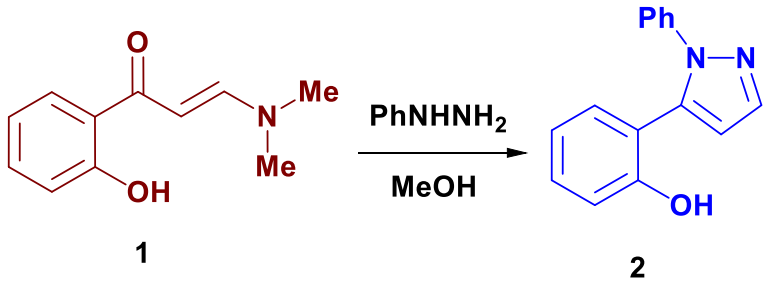


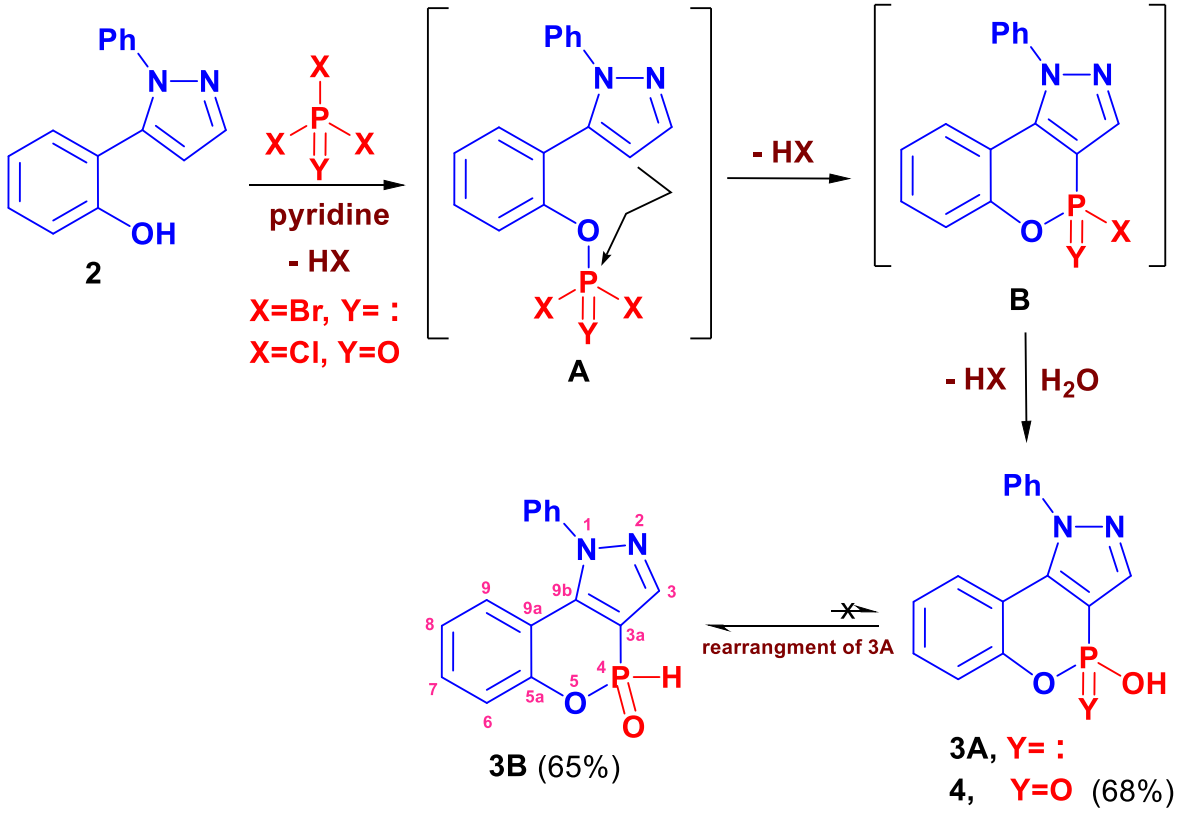
Figure 1

The synthetic strategy to construct the target compounds depends on treatment of 5-(2-hydroxyphenyl)-1-phenyl-1*H*-pyrazole (**2**) as starting material with different examples of phosphorus reagents in dry pyridine. The substrate **2** was synthesized by reaction of 3-(dimethylamino)-1-(2-hydroxy-phenyl)prop-2-en-1-one (**1**) with phenylhydrazine in methanol (Scheme 1).[19]



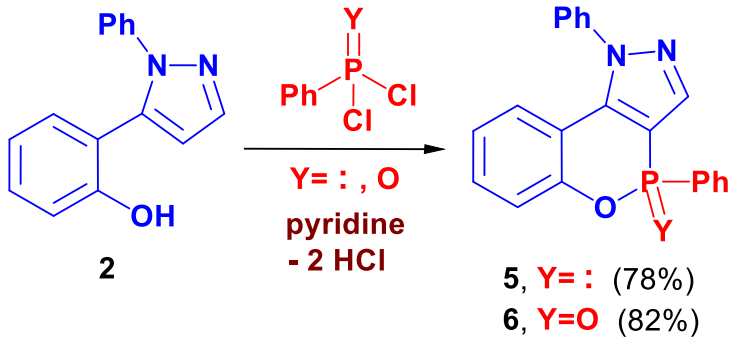
Scheme 1

It is known that pyrazole ring with unsubstituted 4-position undergoes easily electrophilic reactions in this position.[20] This property can be used in the synthesis of a series of phosphorylated pyrazoles at position 4 which may undergo an intramolecular cyclization with active neighboring hydroxyl group.[21] Thus, readily available 5-(2-hydroxyphenyl)-1-phenyl-1*H*-pyrazole (**2**) was subjected to the phosphorylation by using phosphorus halides such as phosphorus tribromide and phosphorus oxychloride in dry pyridine to produce the intermediates **A** which underwent cyclization processes to form the nonisolable intermediates **B** as shown in Scheme 2. These intermediates underwent hydrolysis to give the isolated products 1-phenyl-4-oxido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazoles **3** and **4**, respectively, in good yields (Scheme 2). The spectral data of product **3** supported its existence in the form **3B** and not **3A** due to the phosphite and phosphonate tautomerism.[22]



Scheme 2

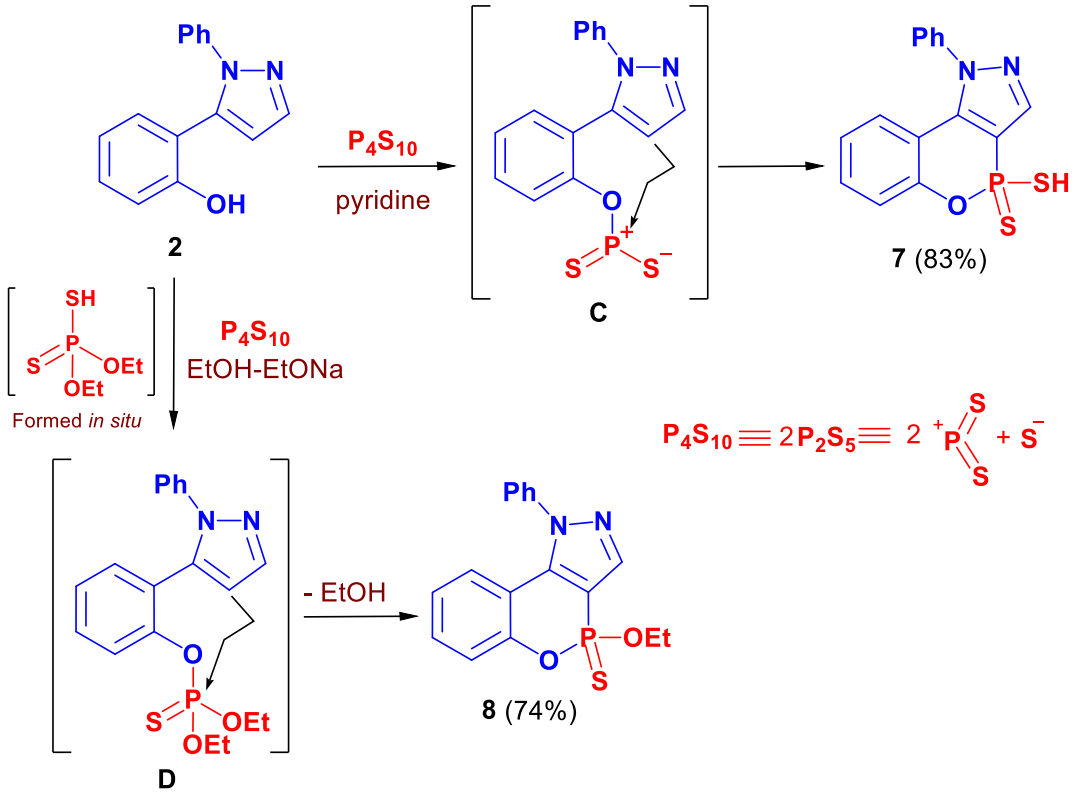
In the same way, the 1,4-diphenyl-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazoles **5** and **6** were obtained in high yield by reaction of compound **2** with P,P-dichlorophenylphosphine and phenylphosphonic dichloride, respectively (Scheme 3).[23]



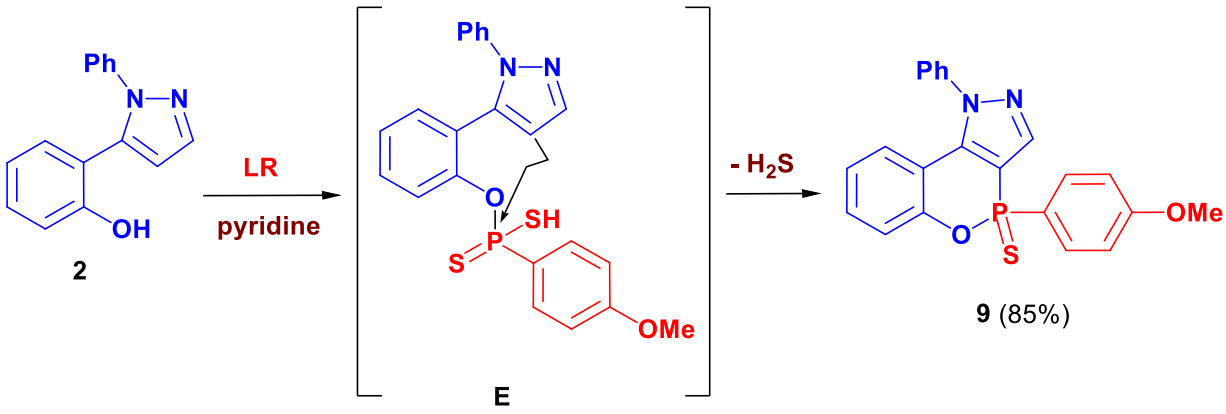
Scheme 3

At this point, it was of interest to investigate the reactivity of the pyrazole **2** with three examples of phosphorus sulfides. Thus, tetraphosphorus decasulfide (P_4S_{10}), known as phosphorylating agent of less reactivity, was shown to react with the parent compound **2** in dry pyridine to furnish the novel 1-phenyl-4-sulfanyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**7**), but when this was carried out

in ethanolic sodium ethoxide, the interesting 4-ethoxy-1-phenyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**8**)[24] was yielded as depicted in Scheme 4. Moreover, the reaction of Lawesson`s reagent (LR) with an equimolar amount of compound **2** in dry pyridine was carried out successfully to give 4-(4-methoxyphenyl)-1-phenyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**9**) (Scheme 5). The latter reaction apparently involved the attack of the oxygen nucleophile on the polar phosphorus atom and gave the intermediate **E** which readily cyclized under the reaction conditions through removal of H_2S molecule to form the isolated product **9** (Scheme 5).



Scheme 4



Scheme 5

The structures **3B**–**9** were characterized by their elemental analysis, mass spectrometry, IR, ^{1}H-, ^{13}C- and ^{31}P-NMR spectral data. The ^{1}H-NMR spectra of the synthesized compounds showed complete signals corresponding to all aromatic protons. The phenolic and C_4–H of pyrazole ring protons of compound **2** were absent in all the isolated products which confirmed the cyclization processes. The characteristic C_3–H proton of pyrazole ring appeared as singlets at δ 8.87–8.73 ppm which confirmed the absence of C_4–H proton. In the ^{1}H-NMR spectrum of compound **3B**, a doublet signal was observed at δ 8.22 ppm due to P–H fragment with coupling constant 479 Hz[25] while a singlet at δ 2.90 ppm was assigned to acidic OH in compound **4**. Also, the protons of the SH, EtO and MeO groups in compounds **7**–**9** were displayed at δ 5.35 (SH), 1.19 (Me), 3.82 (CH_2O) and 3.80 (MeO) ppm. The ^{13}C-NMR spectra of all compounds showed the exact signals of all aromatic carbon atoms between δ 110.9 and 157.2 ppm. The carbon atoms of the EtO and MeO fragments in compounds **8** and **9** were displayed at δ 15.6 (Me), 60.1 (CH_2O) and 55.0 (OMe) ppm. In addition, the C_{3a}–P atoms were appeared as doublets in region δ 110.9–112.6 ppm with coupling constants in range 70–80 Hz. The ^{31}P-NMR spectrum of compound **3B** recorded a singlet at δ 8.2 ppm,[25] while the products **4**–**6** displayed singlets at 26.2, 19.9 and 29.8 ppm, respectively. In addition, the remaining products **7**–**9** recorded their ^{31}P-NMR peaks at δ 44.1, 46.8 and 52.1 ppm, respectively.[26] The mass spectral data of all isolated products showed their molecular ion peaks at the expected values in moderate intensities.

EXPERIMENTAL

The melting point was determined in an open capillary tube on a digital Stuart SMP-3 apparatus. Infrared spectra were measured on FT-IR (Nicolet IS10) spectrophotometer using KBr disks. The ^{1}H- and ^{13}C-NMR spectra were measured on a Bruker spectrometer (400 and 100 MHz), using DMSO-d_6 as a solvent and TMS (δ) as an internal standard. The ^{31}P-NMR spectra were measured on a Bruker (162 MHz) spectrophotometer using DMSO-d_6 as a solvent and 85% H_3PO_4 as an external reference. Mass spectra were recorded on direct probe controller inlet part to single quadropole mass analyzer in ionization method (thermo scientific GCMS). Elemental microanalysis were performed Perkin-Elmer 2400II at the Chemical War department, Ministry of Defense. The purity of the synthesized compounds was checked by thin layer chromatography (TLC) and elemental microanalysis.

General procedure for synthesis of the target compounds 3B-9.

A mixture of 5-(2-hydroxyphenyl)-1-phenyl-1*H*-pyrazole (**2**) (1.18 g, 5 mmol) and the phosphorus reagent (5 mmol) in dry pyridine (35 mL) was heated under reflux for 6–12 h. In case of formation of product **8**, the reaction was carried out in ethanolic sodium ethoxide (0.15 g of Na in 30 mL of absolute EtOH). After completion of reaction (monitored by TLC), the mixture was poured into cold water and neutralized with

diluted hydrochloric acid (5%) under stirring for 1 h. The formed solid was filtered off and crystallized from the proper solvent.

1-Phenyl-4-oxido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**3B**): Yield 65%, pale yellow solid (MeOH), mp 192–194 ºC. IR (KBr), (*v* max, cm^{-1}): 3101 ($C–H_{arom}$), 1627 (C=N), 1595 (C=C), 1273 (P=O), 1036 (P–O–C). ^{1}H-NMR (400 MHz, DMSO-d_6): 6.98 (d, 2H, *J*=8.0 Hz, Ar–H), 7.14 (t, 2H, *J*=7.2 Hz, Ar–H), 7.43 (t, 2H, *J*=8.0 Hz, Ar–H), 7.79–7.81 (m, 3H, Ar–H), 8.22 (d, 1H, J_{PH}=479 Hz, P–H), 8.87 (s, 1H, $C_3–H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 110.9 (d, J_{PC}=70 Hz, C–3a), 117.6 (C–6), 119.6 (C–9a), 121.2 (C–4`$_{phenyl}$), 122.8 (C–2`,6`$_{phenyl}$), 125.0 (C–9), 130.0 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 139.2 (C–7), 141.3 (C–1`$_{phenyl}$), 142.0 (C–3), 142.7 (C–9b), 150.8 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 8.2 ppm. MS (EI, *m/z*): 282 (M^+, 25%). Anal. Calcd for $C_{15}H_{11}N_2O_2P$ (282.24): C, 63.83; H, 3.93; N, 9.93%. Found: C, 63.51; H, 3.78; N, 9.75%.

4-Hydroxy-1-phenyl-4-oxido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**4**): Yield 68%, beige solid (EtOH), mp 262–264 ºC. IR (KBr), (*v* max, cm^{-1}): 3277 (OH), 3090 ($C–H_{arom}$), 1613 (C=N), 1598 (C=C), 1270 (P=O), 1075 (P–O–C). ^{1}H-NMR (400 MHz, DMSO-d_6): 2.90 (s, 1H, P–OH), 6.99 (d, 1H, *J*=7.6 Hz, Ar–H), 7.22–7.48 (m, 5H, Ar–H), 7.95–7.99 (m, 2H, Ar–H), 8.05 (t, 1H, *J*=7.6 Hz, Ar–H), 8.73 (s, 1H, $C_3–H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 110.9 (d, J_{PC}=80 Hz, C–3a), 119.6 (C–6), 121.2 (C–9a), 121.8 (C–4`$_{phenyl}$), 122.8 (C–2`,6`$_{phenyl}$), 126.9 (C–9), 130.0 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 139.2 (C–7), 141.0 (C–1`$_{phenyl}$), 142.7 (C–3), 143.2 (C–9b), 150.8 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 26.2 ppm. MS (EI, *m/z*): 298 (M^+, 5%). Anal. Calcd for $C_{15}H_{11}N_2O_3P$ (298.24): C, 60.41; H, 3.72; N, 9.39%. Found: C, 60.21; H, 3.61; N, 9.09%.

1,4-Diphenyl-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**5**): Yield 78%, yellow solid (EtOH), mp 254–255 ºC. IR (KBr), (*v* max, cm^{-1}): 3080 ($C–H_{arom}$), 1628 (C=N), 1604, 1589 (C=C), 1090 (P–O–C). ^{1}H-NMR (400 MHz, DMSO-d_6): 6.89 (d, 1H, *J*=7.6 Hz, Ar–H), 6.98 (d, 1H, *J*=7.2 Hz, Ar–H), 7.05 (t, 1H, *J*=6.8 Hz, Ar–H), 7.10–7.15 (m, 2H, Ar–H), 7.40–7.44 (m, 3H, Ar–H), 7.78–7.80 (m, 4H, Ar–H), 7.97 (d, 1H, *J*=6.2 Hz, Ar–H), 8.21 (d, 1H, *J*=7.0 Hz, Ar–H), 8.87 (s, 1H, $C_3–H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 112.6 (d, J_{PC}=80 Hz, C–3a), 115.9 (C–6), 117.8 (C–9a), 120.5 (d, J_{PC}=80 Hz, C–1``$_{phenyl}$), 122.0 (C–4`$_{phenyl}$), 125.0 (C–3``,5``$_{phenyl}$), 126.0 (C–2`,6`$_{phenyl}$), 128.3 (C–2``,6``$_{phenyl}$), 129.7 (C–9), 130.2 (C–3`,5`$_{phenyl}$), 132.0 (C–8), 137.4 (C–4``$_{phenyl}$), 138.4 (C–7), 140.5 (C–1`$_{phenyl}$), 142.0 (C–3), 143.5 (C–9b), 157.2 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 19.9 ppm. MS (EI, *m/z*): 342 (M^+, 6%). Anal. Calcd for $C_{21}H_{15}N_2OP$ (342.34): C, 73.68; H, 4.42; N, 8.18%. Found: C, 73.35; H, 4.19; N, 7.79%.

1,4-Diphenyl-4-oxido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**6**): Yield 82%, yellow solid (EtOH), mp 269–270 ºC. IR (KBr), (*v* max, cm^{-1}): 3080 ($C–H_{arom}$), 1625 (C=N), 1591 (C=C), 1241 (P=O), 1084 (P–O–C). ^{1}H-NMR (400 MHz, DMSO-d_6): 6.92 (d, 1H, *J*=7.6 Hz, Ar–H), 6.98 (d, 1H, *J*=7.6 Hz, Ar–H), 7.07–7.15 (m, 3H, Ar–H), 7.38–7.44 (m, 3H, Ar–H), 7.79–7.82 (m, 4H, Ar–H), 7.90 (d, 1H,

J=4.4 Hz, Ar–H), 8.04 (d, 1H, *J*=7.2 Hz, Ar–H), 8.80 (s, 1H, C_3–$H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 111.0 (d, J_{PC}=61 Hz, C–3a), 115.5 (C–6), 119.5 (C–9a), 121.5 (d, J_{PC}=64 Hz, C–1``$_{phenyl}$), 122.0 (C–4`$_{phenyl}$), 122.8 (C–2`,6`$_{phenyl}$), 125.0 (C–3``,5``$_{phenyl}$), 127.1 (C–9), 129.0 (C–2``,6``$_{phenyl}$), 130.0 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 133.2 (C–4``$_{phenyl}$), 139.2 (C–7), 140.1 (C–1`$_{phenyl}$), 142.7 (C–3), 144.2 (C–9b), 150.9 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 29.8 ppm. MS (EI, *m/z*): 358 (M^+, 31%). Anal. Calcd for $C_{21}H_{15}N_2O_2P$ (358.34): C, 70.39; H, 4.22; N, 7.82%. Found: C, 70.11; H, 4.03; N, 7.58%.

1-Phenyl-4-sulfanyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**7**): Yield 83%, orange solid (MeOH), mp 201–202 ºC. IR (KBr), (*v* max, cm^{-1}): 3067 (C–H_{arom}), 2750 (br, SH), 1619 (C=N), 1595 (C=C), 1075 (P–O–C), 761 (P=S). ^{1}H-NMR (400 MHz, DMSO-d_6): 5.35 (br, 1H, SH), 6.95–6.98 (m, 2H, Ar–H), 7.13 (t, 2H, *J*=7.2 Hz, Ar–H), 7.42 (t, 2H, *J*=7.6 Hz, Ar–H), 7.78–7.80 (m, 3H, Ar–H), 8.86 (s, 1H, C_3–$H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 110.9 (d, J_{PC}=82 Hz, C–3a), 119.5 (C–6), 121.2 (C–9a), 122.3 (C–4`$_{phenyl}$), 122.8 (C–2`,6`$_{phenyl}$), 126.0 (C–9), 130.0 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 139.2 (C–7), 140.0 (C–1`$_{phenyl}$), 141.0 (C–3), 142.7 (C–9b), 150.9 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 44.1 ppm. MS (EI, *m/z*): 330 (M^+, 9%). Anal. Calcd for $C_{15}H_{11}N_2OPS_2$ (330.37): C, 54.54; H, 3.36; N, 8.48; S, 19.41%. Found: C, 54.23; H, 3.24; N, 8.14; S, 19.09%.

4-Ethoxy-1-phenyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**8**): Yield 74%, orange solid (C_6H_6), mp 208–209 ºC. IR (KBr), (*v* max, cm^{-1}): 3069 (C–H_{arom}), 2912 (C–H_{aliph}), 1620 (C=N), 1595 (C=C), 1072 (P–O–C), 756 (P=S). ^{1}H-NMR (400 MHz, DMSO-d_6): 1.19 (t, 3H, *J*=7.6 Hz, CH_3), 3.82 (q, 2H, *J*=7.6 Hz, OCH_2), 6.99 (d, 1H, *J*=6.8 Hz, Ar–H), 7.16–7.23 (m, 3H, Ar–H), 7.27–7.30 (m, 2H, Ar–H), 7.41–7.45 (m, 2H, Ar–H), 7.61 (d, 1H, *J*=7.6 Hz, Ar–H), 8.81 (s, 1H, C_3–$H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 15.6 (CH_3), 60.1 (OCH_2), 110.9 (d, J_{PC}=80 Hz, C–3a), 119.5 (C–6), 120.5 (C–9a), 121.8 (C–4`phenyl), 122.7 (C–2`,6`$_{phenyl}$), 125.5 (C–9), 128.2 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 139.9 (C–7), 141.0 (C–1`$_{phenyl}$), 142.5 (C–3), 143.7 (C–9b), 150.0 (C–5a). ^{31}P-NMR (162 MHz, DMSO-d_6): 46.8 ppm. MS (EI, *m/z*): 342 (M^+, 5%). Anal. Calcd for $C_{17}H_{15}N_2O_2PS$ (342.36): C, 59.64; H, 4.42; N, 8.18; S, 9.37%. Found: C, 59.23; H, 4.16; N, 8.01; S, 9.11%.

4-(4-Methoxyphenyl)-1-phenyl-4-sulfido-1,4-dihydro[1,2]benzoxaphosphinino[4,3-*c*]pyrazole (**9**): Yield 85%, reddish orange solid (EtOH), mp 232–234 ºC. IR (KBr), (*v* max, cm^{-1}): 3065 (C–H_{arom}), 2920 (C–H_{aliph}), 1615 (C=N), 1598 (C=C), 1051 (P–O–C), 762 (P=S). ^{1}H-NMR (400 MHz, DMSO-d_6): 3.80 (s, 3H, OCH_3), 6.55 (d, 2H, *J*=8.8 Hz, H–3``,5``$_{aryl}$), 6.77 (d, 1H, *J*=2.4 Hz, Ar–H), 7.19 (d, 2H, *J*=8.8 Hz, H–2``,6``$_{aryl}$), 7.28–7.32 (m, 2H, Ar–H), 7.37–7.41 (m, 3H, Ar–H), 7.52–7.55 (m, 3H, Ar–H), 8.86 (s, 1H, C_3–$H_{pyrazole}$). ^{13}C-NMR (100 MHz, DMSO-d_6): 55.0 (OCH_3), 110.9 (d, J_{PC}=75 Hz, C–3a), 112.7 (C–3``,5``$_{aryl}$), 117.8 (C–6), 119.5 (C–9a), 121.2 (C–4`$_{phenyl}$), 122.8 (C–2`,6`$_{phenyl}$), 124.5 (C–9), 127.5 (C–2``,6``$_{aryl}$), 130.6 (C–3`,5`$_{phenyl}$), 132.2 (C–8), 133.6 (d, J_{PC}=81 Hz, C–1``$_{aryl}$), 139.2 (C–7), 142.7 (C–1`$_{phenyl}$), 144.8 (C–3), 146.0 (C–9b), 150.8 (C–5a), 152.9 (C–4``$_{aryl}$). ^{31}P-NMR (162 MHz, DMSO-d_6): 52.1 ppm. MS (EI, *m/z*):

404 (M^+, 100%). Anal. Calcd for $C_{22}H_{17}N_2O_2PS$ (404.43): C, 65.34; H, 4.24; N, 6.93; S, 7.93%. Found: C, 65.07; H, 3.99; N, 6.69; S, 7.71%.

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