New 2-aryloxy macroheterocyclic phosphates

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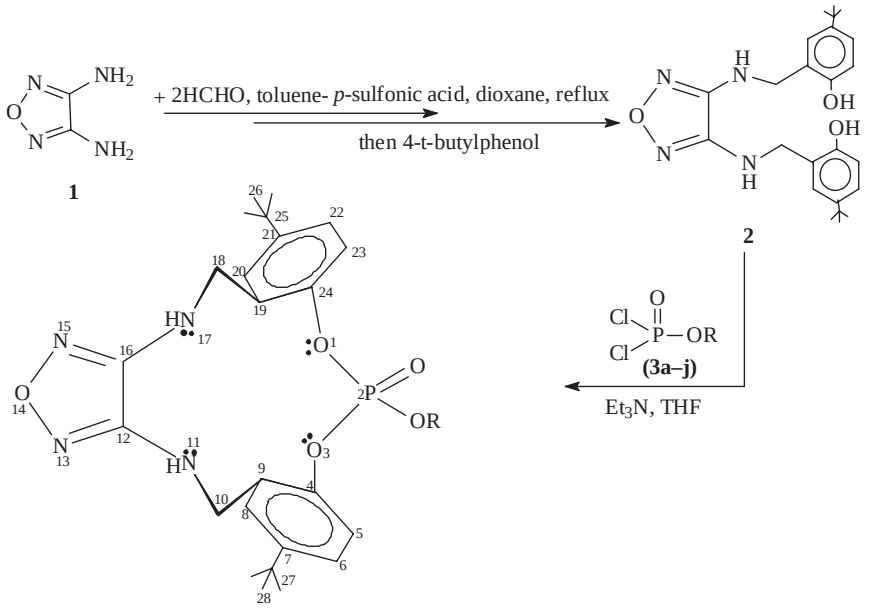
A new class of phosphorus macroheterocycles (**4a–j**) has been synthesised in two steps. The reaction of 1 mole of 3,4-diamino-1,2,5-oxadiazole (**1**) with 2 moles of aqueous formaldehyde gave a Schiff's base which subsequently underwent addition with 2 moles of 4-*t*-butylphenol in 1,4-dioxane, afforded *N*,*N*-bis-(5-*t*-butyl-2-hydroxybenzyl)furazan-3,4-diamine (**2**). Cyclisation of **2** *in situ* with various substituted aryl phosphorodichloridates (**3a–j**) in the presence of Et_3N in THF afforded 13-membered macroheterocycles (**4a–j**) containing P, N, O and C in good yields.

Keywords: phosphorus macroheterocycles, 3,4-diaminofurazan, Schiff's base, arylphosphorodichloridates

Investigations on the synthesis of new phosphorus macroheterocycles for applications in pharmacy and industry have attracted great attention in recent years.[1-7] Previously, novel Schiff-base ligands derived from *N*-functionalised amines and salicylaldehyde have been developed[8-11] and used for the conjugation of proteins to radionucleides for labelling. *N*-functionalised diaminofurazan derivatives have enjoyed increasing chemical and medicinal applications. As part of our efforts to develop multifunctional macrocyclic chelating agents, the synthesis of a new class of 2-aryloxy macroheterocyclic phosphoranes with 13 membered rings containing P, N, O and C atoms has been accomplished.

The synthetic route (Scheme 1) involves two steps. 3,4-Diaminofurazan (**1**)[12] as reacted with 2 moles of aqueous formaldehyde in the presence of *p*-toluenesulfonic acid to form a Schiff's base which subsequently underwent the Mannich reaction[13] with 2 moles of 4-*t*-butylphenol at reflux temperature in THF to afford *N*,*N*-bis-(5-*t*-butyl-2-hydroxybenzyl)furazan-3,4-diamine (**2**). On cyclocondensation of **2** with equimolar quantities of various aryl phosphorodichloridates (**3a–j**)[14] in the presence of triethylamine at 50–60°C in THF the title compounds (**4a–j**) were obtained in good yields. Thin layer chromatography was employed to follow the progress of the reactions. Separation of the insoluble triethylamine hydrochloride by filtration followed by evaporation of the solvent from the filtrate afforded crude products which on recrystallisation from ethanol gave analytically pure samples. Compounds **4a–j** are readily soluble in polar solvents and melt between 214 and 296°C. Their chemical structures were established by analytical and spectroscopic (IR, ^1H, ^{13}C and ^{31}P NMR and mass spectroscopic) data.



Compd.	R	Compd.	R
5a	4-CH_3-C_6H_4	**5f**	C_6H_5
5b	4-Cl-C_6H_4	**5g**	2,3-$(CH_3)_2C_6H_3$
5c	2-COOEt-C_6H_4	**5h**	2,4-$(CH_3)_2C_6H_3$
5d	$C_{10}H_7$	**5i**	4-(CH_3O)-C_6H_4
5e	4-NO_2-C_6H_4	**5j**	2-Cl-C_6H_4

Scheme 1

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The presence of characteristic bands[15-16] for NH (3325–3339 cm^{-1}), P=O (1226–1256 cm^{-1}) and P–O–C_{ar} (1167–1176, 978–996 cm^{-1}) in the IR spectra of **4a–j** proved that cyclisation of **2** with **3a–j** occurred to form the phosphorus macroheterocycles **4a–j**. In their ^{1}H NMR spectra, the multiplets in the δ 6.23–7.83 region[12] are attributed to the aromatic protons. The triplet at δ 6.29–6.71 accounts for the NH protons of the macrocyclic ring.[12] This assignment was confirmed by D_2O exchange experiments and the methylene protons resonated as doublet at δ 4.27–4.56. The proton-decoupled ^{13}C NMR chemical shifts of **4a–j** are given in the experimental section.[12-14] The oxygen bonded carbons C-4 and C-24 of the macrocyclic ring resonated as doublets in the downfield region at δ 147.6–148.6 (^2J = 7.2–7.6 Hz). The doublet in the upfield region at 117.7–119.6 (^3J = 8.0–8.8) is assigned to C-5 and C-23. Another doublet at δ 121.5–123.0 (J = 7.0–7.6 Hz) is ascribed to C-9 and C-19. The chemical shifts at δ 125.0–126.8, 145.8–147.2 and 121.3–124.7 are attributed to C-6 and C-22, C-7 and C-20, and C-8 and C-20, respectively. The signals observed in the δ 153.2–155.9 range are attributed to C-12 and C-16. The carbons C-10 and C-18 resonated at 47.6–49.7. The signals of low intensity at δ 34.0–34.8 are for C-25 and C-27 and signals at δ 30.9–31.8 are for C-26 and C-28, respectively. The carbons of the exocyclic phenoxy moiety resonated in the expected range. ^{31}P NMR signals appeared in the range of –2.97 to –5.47 ppm.

Fragmentation values in the mass spectra for **4a–j** are given in the experimental data. The appearance of M^+ at appropriate molecular weight numbers, [M-(OR, C_4H_8)]$^+$ at *m/z* 357, [M-(OR, PO_2)]$^+$ at *m/z* 423, [M-(OR)]$^+$ at *m/z* 469 and [M-(R-H)]$^+$ at *m/z* 486 with the macrocyclic phosphorane 2-oxides moiety, conclusively establishes a 1: 1 molecular ratio of moieties derived from **2** and **3a–j** in these molecules. Further, these ions may serve as diagnostic daughter ions of these compounds for their monitoring in bio and eco systems.

In summary, we have reported an effective and simple method for the synthesis of novel 13-membered phosphorus macroheterocyclic compounds bearing the heteroatoms P, N and O, and fused with an oxadiazole ring.

Experimental

The melting points were determined on a Mel-Temp apparatus and are uncorrected. Microanalyses were performed at the Central Drug Research Institute, Lucknow, India. IR spectra were recorded on KBr pellets using a Perkin-Elmer 283 unit. The ^{1}H, ^{13}C and ^{31}P NMR spectra were taken on a Bruker AMX-400 MHz spectrometer operating at 400 MHz for ^{1}H, 100 MHz for ^{13}C and 161.9 MHz for ^{31}P. Compounds were dissolved in $CDCl_3$ or DMSO-d_6, and chemical shifts were referenced to TMS (^{1}H and ^{13}C) and 85% H_3PO_4 (^{31}P). Mass spectral data were collected on a GC MS instrument at 70 eV. 3,4-Diaminofurazan (**2**) and arylphosphorus dichloridates (**3a–j**) were prepared according to the reported procedures. 4-*t*-butylphenol was procured from the Aldrich Chemical Company, USA and used without further purification.

Synthesis of N,N-bis-(5-tert-butyl-2-hydroxybenzyl)furazan-3,4-diamine **(2):** To a stirred and cooled (10–20°C) solution of 3,4-diaminofurazan (2.0 g, 0.02 mol) and toluene-4-sulfonic acid hemihydrate (700 mg) in 30 ml of dioxane, was slowly added a solution of 37% aqueous formaldehyde (3.5 ml, 0.04 mol) in 20 ml of dioxane at 20–25°C with stirring. The mixture was later kept under reflux for 1 h then cooled to room temperature. A solution of 4-*t*-butylphenol (3.0 g, 0.02 mol) in 20 ml of dioxane was added dropwise to the above solution with simultaneous stirring and the resulting solution was heated under gentle reflux with stirring for 2 h. A sticky mass, obtained upon removal of dioxane with a flash evaporator, was recrystallised from 95% ethanol to yield **2**.

Yield (5.9 g, 70%), m.p. 218°C. IR (KBr): ν_{max} 3320 (NH), 3382 (OH), 3031(C-H_{ar}), 1593 (C=N) cm^{-1}. ^{1}H NMR (DMSO-d_6): δ 10.67 (s, 2H, OH), 6.70–7.20 (m, 6H, Ar), 6.25 (t, J = 6.7 Hz, 2H, NH), 4.18 (d, J = 7.0 Hz, 4H, CH_2), 1.21 (s, 18H , *t*-butyl).^{13}C NMR (DMSO-d_6): δ 154.2 (C-1), 150.2 (C-9), 141.1 (C-4), 124.8 (C-3), 123.8 (C-5), 122.2 (C-6), 115.8 (C-2), 50.4 (C-7), 34.1 (C-10), 31.3 (3C, C-11). (MS) m/z(%): 424 (M^+, 25), 409 (48), 394 (51), 368 (63), 312 (74), 163 (32), 124 (42), 100 (69), 83 (80). found: C, 67.7; H, 7.4; N, 13.0. $C_{24}H_{32}O_3N_4$ requires: C, 67.9 ; H, 7.5; N, 13.2.

Synthesis of 2-(4'-methylphenoxy) macroheterocyclic phosphate **(4a):** A solution of 4-methylphenylphosphorodichloridate (1.13 g, 0.005 mol) in 25 ml of dry THF was added dropwise over a period of 30 min to a stirred and cooled (10–20°C) solution of **2** (2.1 g, 0.005 mol) and triethylamine (1.0g, 0.01 mol) in 30 ml of dry THF. After completion of the addition, the temperature of the reaction mixture was slowly raised to room temperature and the mixture was stirred for 2 h. Later the reaction mixture was heated to 50–60°C and maintained at this temperature for 4 h with stirring. The completion of the reaction was monitored by TLC analysis and a mixture of ethyl acetate and hexane (2:3) was used as mobile solvent and silica gel as adsorbent. The reaction product, being insoluble in THF, separated and was collected by filtration. The gummy solid obtained was washed with water to remove residual traces of triethylamine hydrochloride and dried. The dry solid obtained was recrystallised from aqueous propan-2-ol.

Yield 61%, m.p. 276–277°C. IR (KBr): ν_{max} 3336 (NH), 1256 (P=O), 1172, 996 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –3.22. ^{1}H NMR (DMSO-d_6): δ 6.35–6.80 (m, 10H, Ar-H), 6.29 (t, J= 9.1 Hz, 2H, NH), 4.27 (d, J = 6.6 Hz, 4H, CH_2), 2.25 (s, 3H, CH_3), 1.16 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 155.4 (C-12 and C-16), 150.8 (d, 8.8, C-1'), 148.2 (d, 7.6, C-4 and C-24), 145.9 (C-7 and C-21), 134.3 (C-4'), 129.6 (C-3' and 5'), 125.2 (C-6 and C-22), 123.3 (C-8 and C-20), 122.2 (d, 7.3, C-9 and C-19), 120.4 (d, 4.4, C-2' and 6'), 118.2 (d, 8.8, C-5 and C-23), 49.7 (C-10 and C-18), 34.2 (C-25 and C-27), 31.3 (C-26 and C-28), 20.9 (4'-CH_3). MS *m/z* (%): 576 (M^+, 16), 560 (26), 520 (32), 486 (57), 469 (34), 423 (46), 326 (11), 243 (30), 188 (21), 163 (11), 150 (43), 124 (100), 108 (59), 100 (63). $C_{31}H_{37}O_5N_4P$ requires : C, 64.6; H, 6.4; found: C, 64.3; H, 6.4.

This procedure was used for the preparation of the other compounds (**4b–j**), and their physical and spectroscopic data are given below.

2-(4'-Chlorophenoxy) macroheterocyclic phosphate **(4b):** Yield 65%, m.p. 230–232°C. IR(KBr): ν_{max} 3332 (NH), 1237 (P=O), 1175, 980 (POC_{ar}) cm^{-1}. ^{31}P NMR(DMSO-d_6): δ –4.28. ^{1}H NMR (DMSO d_6): δ 6.23–6.98 (m, 10H, Ar-H), 6.71 (t, 8.7, 2H, NH), 4.58 (d, 6.9, 4H, CH_2), 1.25 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 155.9 (C-12 and C-16), 149.7 (d, 8.9, C-1'), 148.1 (d, 7.6, C-4 and C-24), 146.6 (C-7 and C-21), 131.8 (C-4'), 129.5 (C-3' and C-5'), 126.5 (C-6 and C-22), 121.3 (C-8 and C-20), 122.1 (d, 7.4, C-9 and C-19), 121.0 (C-2' and 6'), 118.4 (d, 8.1, C-5 and C-23), 48.1 (C-10 and C-18), 34.3 (C-25 and C-27), 31.3 (C-26 and C-28). MS *m/z* (%): 598 (M^+ +2, 10), 596 (M^+, 24), 560 (56), 486 (54), 469 (80), 423 (34), 357 (17), 323 (8), 243 (31), 208 (28), 190 (13), 176 (23), 163 (11), 150 (48), 124 (100), 100 (43), 83 (78). found: C, 60.15, H, 5.7. $C_{30}H_{34}O_5N_4PCl$ requires: C, 60.4, H, 5.7.

2-(2-Ethylformate phenoxy)macroheterocyclic phosphate **(4c):** Yield 43%, m.p. 282–284 °C. IR (KBr): ν_{max} 3330 (NH), 1237 (P=O), 1167, 984 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO): δ –4.51. ^{1}H NMR (DMSO-d_6): δ 6.36 (t, 7.4, 2H, NH), 4.56 (d, 6.8, 4H, CH_2), 6.52–7.31 (m, 10H, Ar-H), 4.27 (q, 2H, OCH_2), 1.27 (s, 18H, *t*-butyl), 1.16 (t, 3H, CH_3). ^{13}C NMR (DMSO-d_6): δ 164.3 (C=O), 156.6 (d, 9.0, C-1'), 154.7 (C-12 and C-16), 148.6 (d, 7.5, C-4 and C-24), 147.2 (C-7 and C-21), 134.8 (C-5'), 132.3 (C-3'), 128.9 (C-2'), 126.8 (C-6 and C-22), 123.7 (C-8 and C-20), 121.5 (d, 7.6, C-9 and C-19), 119.6 (d, 8.4, C-5 and C-23), 118.0 (C-4'), 117.2 (C-6'), 63.1 (OCH_2), 49.4 (C-10 and C-18), 34.8 (C-25 and C-27), 31.8 (C-26 and C-28), 14.7 (CH_3). found: C, 62.2; H, 6.1. $C_{33}H_{39}O_7N_4P$ requires: C, 62.5, H, 6.15.

2-(2-Naphthalyoxy) macroheterocyclic phosphate **(4d):** Yield 52%, m.p. 225–227°C. IR (KBr): ν_{max} 3337 (NH), 1242 (P=O), 1174, 986 (POC_{ar}) cm^{-1}. ^{1}H NMR (DMSO-d_6): δ 6.48 (t, 7.6, 2H, NH), 6.67–7.83,(m,13H,Ar-H),4.44(d,6.6,4H,CH_2),1.27(s,18H,*t*-butyl).^{31}P NMR (DMSO-d_6): δ –4.01. found: C, 66.4; H, 6.0. $C_{34}H_{37}O_5N_4P$ requires: C, 66.65, H, 6.0.

2-(4-Nitrophenoxy) macroheterocyclic phosphate **(4e):** Yield 63%, m.p. 294–296°C. IR (KBr): ν_{max} 3339 (NH), 1239 (P=O), 1171, 982 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –5.47. ^{1}H NMR (DMSO-d_6): δ 6.34 (t, 7.2, 2H, NH), 6.41–7.72 (m, 10H, Ar-H), 4.38 (d, 6.7, 4H, CH_2), 1.20 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 155.0 (C-12 and C-16), 148.8 (d, 8.6, C-1'), 147.8 (d, 7.3, C-4 and C-24), 146.2 (C-7 and C-21), 144.6 (C-4'), 129.0 (C-3' and 5'), 128.8 (C-6'), 128.3 (d, 4.4 C-2' and C-6'), 125.8 (C-6 and C-22), 122.8 (C-8 and C-20), 122.4 (d, 7.5, C-9 and C-19), 117.9 (d, 8.2, C-5 and C-23),

34.2 (C-25 and C-27), 31.2 (C-26 and C-28). found: C, 59.2; H, 5.5. $C_{30}H_{34}O_7N_5P$ requires: C, 59.3; H, 5.6.

2-(Phenoxy)macroheterocyclic phosphate **(4f):** Yield 58%, m.p. 220–221°C. IR (KBr): ν_{max} 3328 (NH), 1226 (P=O), 1169, 978 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –2.97. ^{1}H NMR (DMSO-d_6): δ 6.54 (t, 7.4, 2H, NH), 6.87–7.29 (m, 11H, Ar-H), 4.52 (d, 6.6, 4H, CH_2), 1.25 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 154.5 (C-12 and C-16), 148.8 (d, 8.6, C-1'), 148.0 (d, 7.6, C-4 and C-24), 146.2 (C-7 and C-21), 129.3 (C-3' and 5'), 126.4 (C-6 and C-22), 125.0 (C-4'), 124.7 (C-8 and C-20), 123.0 (d, 7.2, C-9 and C-19), 121.4 (C-2' and 6'), 117.7 (d, 8.1, C-5 and C-23), 47.6 (C-10 and C-18), 34.6 (C-25 and C-27), 30.9 (C-26 and C-28). found: C, 63.7, H, 6.2. $C_{30}H_{35}O_5N_4P$ requires: C, 64.05; H, 6.2.

2-(2,3-Dimethylphenoxy)macroheterocyclic phosphate **(4g):** Yield 64%, m.p. 224–226°C. IR (KBr): ν_{max} 3331 (NH), 1238 (P=O), 1176, 987 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –3.25. ^{1}H NMR (DMSO-d_6): δ 6.65 (t, 7.5, 2H, NH), 6.98–7.37 (m, 9H, Ar-H), 4.51 (d, 7.0, 4H, CH_2), 2.34 (s, 3H, 3'-CH_3), 2.26 (s, 3H, 2'-CH_3), 1.20 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 154.3 (C-12 and C-16), 150.2 (d, 8.7, C-1') 148.4 (d, 7.2, C-4 and C-24), 146.5 (C-7 and C-21), 138.5 (C-3'), 127.6 (C-5'), 126.5 (C-2'), 126.8 (C-6 and C-22), 125.5 (C-4'), 123.5 (C-8 and C-20), 122.4 (d, 7.0, C-9 and C-19), 119.2 (C-6'), 118.6 (d, 8.6, C-5 and C-23), 48.5 (C-10 and C-18), 34.3 (C-25 and C-27), 31.6 (C-26 and C-28), 12.8 (2'-CH_3), 20.3 (3'-CH_3). found: C, 64.8; H, 6.6. $C_{32}H_{39}O_5N_4P$ requires: C, 65.1; H, 6.6.

2-(2,4-Dimethylphenoxy)macroheterocyclic phosphate **(4h):** Yield 68%, m.p. 243–244°C. IR(KBr): ν_{max} 3331 (NH), 1240 (P=O), 1174, 984 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –3.37. ^{1}H NMR (DMSO-d_6): H 6.52 (t, 7.2, 2H, NH), 6.85–7.43 (m, 9H, Ar-H), 4.56 (d, 6.7, 4H, CH_2), 2.32(s, 3H, 4'-CH_3), 2.28 (s, 3H, 2'-CH_3), 1.21 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 153.2 (C-12 and C-16), 150.4 (d, 8.7, C-1'), 147.6 (d, 7.4, C-4 and C-24), 145.8 (C-7 and C-21), 132.4 (C-4'), 128.5 (C-2'), 128.3, (C-3'), 126.4 (C-5'), 125.7 (C-6 and C-22), 122.6 (d, 7.4, C-9 and C-19), 122. 5 (C-8 and C-20), 119.4 (d, 6.7, C-6'), 118.0 (d, 8.3, C-5 and C-23), 48.4 (C-10 and C-18), 34.4 (C-25 and C-27), 31.4 (C-26 and C-28), 17.8 (2'-CH_3), 20.81 (4'-CH_3). found: C, 64.7; H, 6.6. $C_{32}H_{39}O_5N_4P$ requires: C, 65.1; H, 6.6.

2-(4-Methoxyphenoxy)macroheterocyclic phosphate **(4i):** Yield 70%, m.p. 214–215°C. IR(KBr): ν_{max} 3325 (NH), 1234 (P=O), 1168, 985 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –4.37. ^{1}H NMR (DMSO-d_6): δ 6.58 (t, 7.6, 2H, NH), 6.85–7.71 (m, 10H, Ar-H), 4.50 (d, 6.8, 4H, CH_2), 3.71 (s, 3H, OCH_3), 1.27 (s, 18H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 155.1 (C-12 and C-16), 152.1 (C-4'), 149.4 (d, 9.0, C-1'), 147.9 (d, 7.6, C-4 and C-24), 146.4 (C-7 and C-21), 125.4 (C-6 and C-22), 124.9 (C-3' and 5'), 123.5 (C-8 and C-20), 122.3 (d, 7.3, C-9 and C-19), 121.3 (C-2' and 6'), 118.7 (d, 8.1, C-5 and C-23), 54.2 (OCH_3), 47.9 (C-10 and C-18), 34.5 (C-25 and C-27), 31.3 (C-26 and C-28). Found: C, 62.6; H, 6.1. $C_{31}H_{37}O_6N_4P$ requires: C, 62.8; H, 6.25.

2-(2-Chlorophenoxy)macroheterocyclic phosphate **(4j):** Yield 54%, m.p. 235–237°C. IR(KBr): ν_{max} 3332 (NH), 1246 (P=O), 1172, 981 (POC_{ar}) cm^{-1}. ^{31}P NMR (DMSO-d_6): δ –3.41. ^{1}H NMR (DMSO-d_6): δ 6.62 (t,7.5, 2H, NH), 6.89–7.72 (m, 10H, Ar-H), 4.48 (d, 6.8, 4H, CH_2), 1.26 (s, 18 H, *t*-butyl). ^{13}C NMR (DMSO-d_6): δ 152.8 (C-12 and C-16), 148.9 (d, 9.0, C-1'), 148.7 (d, 7.5, C-4 and C-24), 146.3 (C-7 and C-21), 132.8 (C-3'), 129.5 (C-5'), 126.3 (C-6 and C-2), 125.1 (C-2'), 124.2 (C-4'), 122.9 (C-8 and C-20), 121.8 (d, 7.5 C-9 and C-19), 120.3 (C-6'), 118.2 (d, 7.9, C-5 and 23), 48.1 (C-10 and 18), 34.0 (C-25 and 27), 31.2 (C-26 and 28). found: C, 60.1; H, 5.6. $C_{30}H_{34}O_5N_4PCl$ requires: C, 60.4; H, 5.7.

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