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Synthetic Approach for Novel Bis(α-aminophosphonic acid) Derivatives of Chromone Containing 1,2,4,3-Triazaphosphole Moieties

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ABSTRACT: *Terephthalic acid hydrazide (**1**) was reacted with phenylphosphonic dichloride to yield bis(1,3,4,2-oxadiazaphosphole) derivative* **3**, *which was condensed with α-aminophosphonic acid derivative* **4** *to afford bis[(1,2,4,3-triazaphospholyl)(chromonyl)methylphosphonic acid] derivative* **5**. *The addition of diethyl phosphite to Schiff bases* **9** *and* **10** *derived from the condensation of bis(4-amino-1,2,4,3-triazaphosphole) derivative* **6** *or bis(4-phosphorylamino-1,2,4,3-triazaphosphole) derivative* **7** *with 3-formyl-6-methylchromone (**8**) yielded bis[(1,2,4,3-triazaphospholyl)(chromonyl)aminomethylphosphonate] derivative* **11** *and bis[(1,2,4,3-triazaphospholyl)(chromonyl)phosphorylaminomethylphosphonate] derivative* **12**, *respectively. The structures of all products were established by elemental analysis and spectral data.* © 2009 Wiley Periodicals, Inc. Heteroatom Chem 20:117–122, 2009; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20520

INTRODUCTION

α-Aminophosphonic acids, bioisosteres of natural amino acids, have been found to exhibit a wide range of biological activities. A large volume of research on their synthesis and biological activities has been reported during the last 20 years. Several α-aminophosphonate derivatives are bioactive. They display antiviral, antimicrobial, antifungal, and antitumor activities [1–7]. At the same time, we noticed that phosphorus-containing heterocyclic systems also possess a wide range of biological and pharmacological activities [8–13]. On the other hand, the chromone nucleus is an important feature in a variety of natural products and medicinal agents [14–19]. Having these facts in mind and in continuation of our efforts to synthesize novel α-aminophosphonate derivatives containing chromone moieties [20], the present work was aimed to synthesize novel bis(α-aminophosphonic acid) derivatives of chromone containing 1,2,4,3-triazaphosphole moieties.

RESULTS AND DISCUSSION

The key intermediate in the present study is the terephthalic acid hydrazide (**1**), which was prepared according to a previously described procedure [21]. The reaction of **1** with phenylphosphonic dichloride in THF and DMF in the presence of sodium hydride gave the nonisolable intermediate **2**, which was converted to 5,5′-(1,4-phenylene)bis(2-oxo-2-phenyl-2,3-dihydro-1,3,4,2-oxadiazaphosphole) (**3**) (Scheme 1). The structural assignment of compound **3** was established by elemental analysis and spectroscopic

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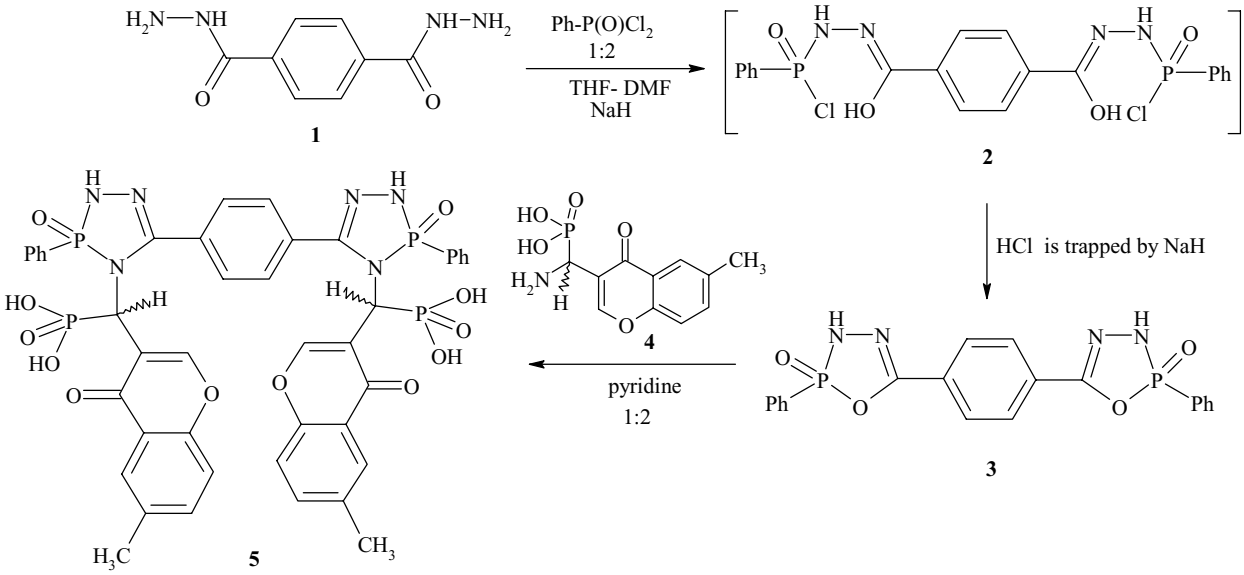


One diastereomeric form meso (*R,S*) or racemic (*R,R and S,S*)

SCHEME 1

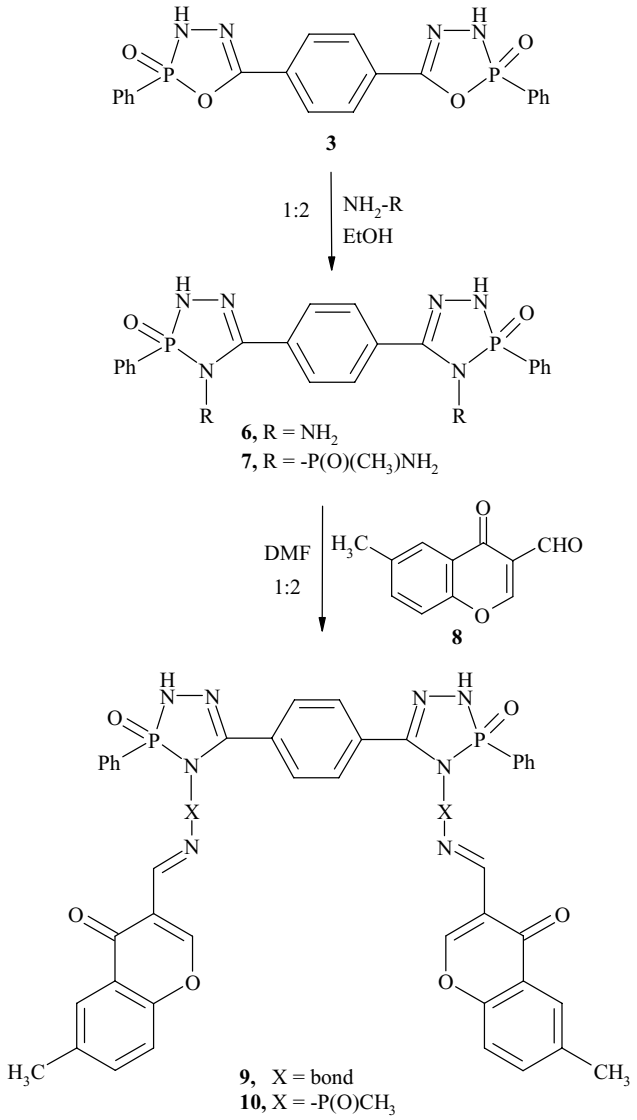
studies. Its ^{1}H NMR spectrum showed signals of NH and aromatic protons at δ 14.16 and 7.23–7.55 ppm, respectively, whereas ^{31}P{^{1}H}NMR spectrum showed a singlet at δ 29.4 ppm [22]. In the IR spectrum of **3**, the absorption bands of 1,3,4,2-oxadiazaphosphole moiety appeared at 3168 (NH), 1654 (C=N), and 1224 cm^{-1} (P=O).

Interestingly, bis compound **3** on treatment with [amino(6-methyl-4-oxo-4*H*-chromen-3-yl)methyl]-phosphonic acid (**4**) in dry pyridine afforded only one diastereomeric form of 5,5′-(1,4-phenylene)bis-[(3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl)(6-methyl-4-oxo-4*H*-chromen-3-yl)]-methylphosphonic acid) (**5**) (Scheme 1).

The spectral data are in good agreement with the proposed structure. Thus, the IR spectrum of **5** showed absorption bands of NH and OH groups in the region of 2675–3339 cm^{-1}, C=O pyrone at 1646 cm^{-1}, and P=O groups as strong bands at 1287 and 1205 cm^{-1}. ^{1}H and ^{31}P{^{1}H}NMR spectra of the product gave conclusive evidence for α-aminophosphonic moieties. The ^{1}H NMR spectrum showed a broad signal at δ 3.40 ppm for hydroxyl protons and a doublet at δ 6.31 ppm (J = 19.2 Hz) for CH—P proton. Furthermore, its ^{31}P{^{1}H}NMR spectrum displayed two signals at δ 25.9 and 13.9 ppm in accordance with 1,3,4,2-triazaphosphole and α-aminophosphonic acid moieties, respectively [23].

When compound **3** was heated with hydrazine hydrate or methylphosphonic diamide in absolute ethanol, bis-amino compounds **6** and **7**, respectively, were formed. The condensation of compound **6** or **7** with 3-formyl-6-methylchromone (**8**) in boiling DMF afforded the corresponding Schiff bases **9** and **10**, respectively (Scheme 2). Structures of compounds **6–10** were deduced on the basis of analytical and spectral data (see the "Experimental" section). The chemical shifts of NH_2 protons in compounds **6** and **7** were recorded at δ 5.78 and 4.98 ppm, respectively. Also, ^{1}H NMR spectra of **9** and **10** showed broad signals at δ 9.76 and 9.11 ppm for CH=N protons, respectively.

The addition of diethyl phosphite to azomethine bonds of compounds **9** and **10** on fusion at 80°C–100°C in the presence of a catalytic amount of triethylamine yielded only one diastereomeric form of 5,5′-(1,4-phenylene)bis(diethyl[{[3-phenyl-3-oxo-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl]amino}(4-oxo-4*H*-chromen-3yl)methyl]phosphonate) (**11**) and two diastereomeric forms of 5,5′-(1,4-phenylene)bis-{diethyl[[(3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl)methylphosphoryl]amino][6-methyl-4-oxo-4*H*-chromen-3-yl)methyl]phosphonate} (**12**), respectively, (Scheme 3). IR spectra of **11** and **12** showed absorption bands for NH and P=O groups at regions 3295–3198 and 1305–1215 cm^{-1}, respectively. The ^{1}H NMR spectrum of compound **11** was characterized by a broad signal for CH—P hydrogens at δ 5.36 ppm. Beside its ^{31}P{^{1}H}NMR spectrum revealed a signal for α-aminophosphonate moiety at δ 21 ppm. On the other hand, the ^{1}H NMR spectrum of compound **12**



SCHEME 2

showed two doublet signals at δ 5.17 ($J = 23$ Hz) and 5.24 ($J = 23$ Hz) ppm for CH—P hydrogens. Beside its $^{31}P\{^{1}H\}$NMR spectrum revealed two signals for α-aminophosphonate moieties at δ 23.1 and 23.5 ppm, which confirmed that compound **12** exists in two diastereomeric forms (~91%:9%).

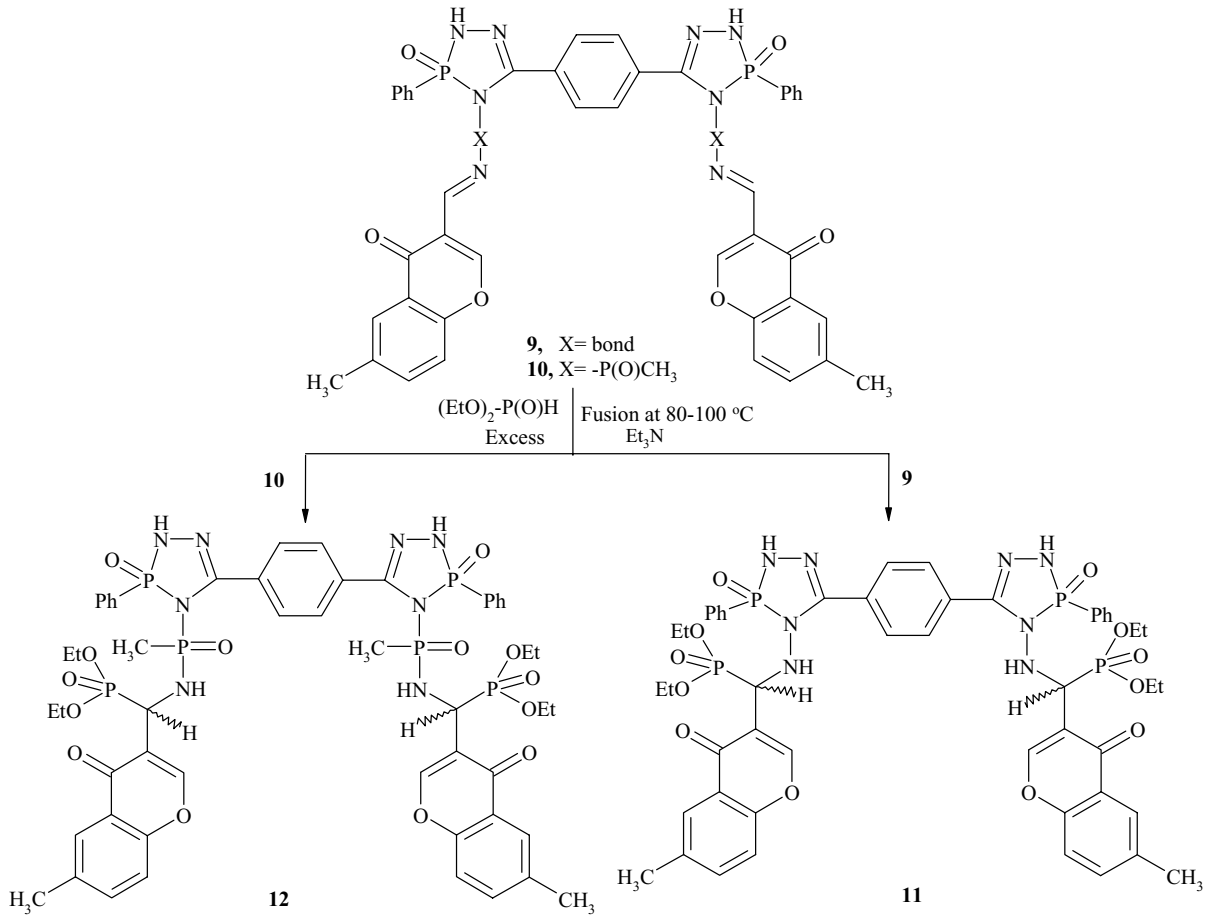
The formation of bis(α-aminophosphonate) derivatives **5, 11**, and **12** should yield a mixture of *meso* and *racemic* diastereomers [24]. However, ^{1}H and $^{31}P\{^{1}H\}$NMR spectra do not show the complexity expected for a mixture of diastereomers and seem to indicate the presence of only one isomer (one CHP signal, one triplet for ethoxy methyl group) except compound **12**. Furthermore, this interpretation is also nicely confirmed by $^{31}P\{^{1}H\}$NMR spectra, which show only one sharp signal for α-aminophosphonate moieties for compounds **5** and **11** and two signals for compound **12**. Thus, we conclude that, once again, the synthesis of bis(aminophosphonate) in most cases is occurring with high stereoselectivity, yielding as major product only one of the diastereomers, as previously obtained in similar reactions [20,25].

EXPERIMENTAL

Melting points of the products were determined on a Stuart SMP3 apparatus. IR spectra were recorded on an Elmer 293 spectrophotometer (γ in cm^{-1}), using KBr disks. ^{1}H NMR spectra were recorded on a Gemini-200 spectrometer (200 MHz), using DMSO-d_6 as a solvent and TMS ($\delta = 0.0$ ppm) as an internal standard. $^{31}P\{^{1}H\}$NMR spectra were recorded on a Varian Inova 500 MHz spectrometer at room temperature with DMSO-d_6 as a solvent and TMS as an internal standard and 85% H_3PO_4 as an external reference, respectively. Elemental microanalyses were performed at the microanalysis center in Bulgarian Academy of Science, Sofia, Bulgaria. [Amino(6-methyl-4-oxo-4*H*-chromen-3-yl)methyl]phosphonic acid (**4**) [26], methylphosphonic diamide [27], and 3-formyl-6-methylchromone (**8**) [28] were prepared by the published methods.

*5,5′-(1,4-Phenylene)bis(2-oxo-2-phenyl-2,3-dihydro-1,3,4,2-oxadiazaphosphole) (**3**)*

Sodium hydride (480 mg, 20 mmol) was added in portions to a solution of terephthalic acid hydrazide (**1**) (970 mg, 5 mmol) in DMF (20 mL) at room temperature. Phenylphosphonic dichloride (1950 mg, 10 mmol) in THF (5 mL) was added drop wise to this mixture while the temperature of the reaction mixture was maintained at 25°C. The whole mixture was heated under reflux for 6 h. The mixture was filtered off while hot to remove sodium chloride. The filtrate was evaporated under reduced pressure to give thick gum, which is treated by diethyl ether. The resulting solid was collected by filtration and crystallized from THF to give pale yellow crystals in 72% yield, m.p. 88°C–90°C. IR (in cm^{-1}): 3168 (NH), 3060 (C—H_{arom}), 1654 (C=N), 1552 (C=C), 1224 (P=O), 1055 (P—O—C). ^{1}H NMR (DMSO): δ 7.23–7.55 (m 14H, aromatic protons), 14.16 (s, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 29.4 ppm. Anal. calcd. for $C_{20}H_{16}N_4O_4P_2$ (438.31): C, 54.80; H, 3.68; N, 12.78. Found: C, 54.43; H, 3.49; N, 12.43.



SCHEME 3

*5,5′-(1,4-Phenylene)bis[(3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl)(6-methyl-4-oxo-4*H*-chromen-3-yl)]methyl-phosphonic acid) (***5***)*

A mixture of compound **3** (2190 mg, 5 mmol) and [amino(6-methyl-4-oxo-4*H*-chromen-3-yl)methyl]-phosphonic acid (**4**) (2690 mg, 10 mmol) in dry pyridine (40 mL) was heated under reflux for 12 h. The reaction mixture was poured into ice–HCl mixture and stirred for 10 min. The obtained solid was collected by filtration, dried, and crystallized from ethanol to give white crystals in 62% yield, m.p. 238°C–240°C. IR (in cm^{-1}): 3339 (NH), 3020 (C—H_{arom}), 2979 (C—H_{aliph}), 2737, 2675 (OH), 1646 (C=O), 1603 (C=N), 1581 (C=C), 1287, 1205 (2 P=O). ^{1}H NMR (DMSO): δ 2.19 (s, 6H, 2 CH_3), 3.40 (br, 4H, P—OH), 6.31 (d, $J = 19.2$ Hz, 2H, 2 CH—P), 7.45–8.07 (m, 18H, aromatic protons), 8.57 (s, 1H, H-5), 8.77 (s, 1H, H-5′), 9.16 (s, 1H, H-2), 9.20 (s, 1H, H-2′), 13.69 (s, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 13.9 (O=P—OH), 25.9 (Ph—P=O) ppm. Anal. calcd. for $C_{42}H_{36}N_6O_{12}P_4$ (940.66): C, 53.63; H, 3.86; N, 8.93. Found: C, 53.32; H, 3.62; N, 8.69.

*5,5′-(1,4-Phenylene)bis(4-amino-3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triaza-phosphole) (***6***) and 5,5′-(1,4-Phenylene)bis[*P*-methyl-P-(3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl)phosphinic amide] (***7***)*

Hydrazine hydrate or methylphosphonic diamide (10 mmol) was added to a solution of compound **3** (2190 mg, 5 mmol) in absolute ethanol (25 mL). The reaction mixture was heated under reflux for 5 h. The obtained solids were filtered off, dried, and crystallized from DMF/ethanol to give **6** and **7**, respectively.

6: Yellow crystals in 66% yield, m.p. 165°C–167°C. IR (in cm^{-1}): 3147 (NH_2, NH), 3048 (C—H_{arom}), 1601 (C=N), 1559 (C=C), 1235 (P=O). ^{1}H NMR (DMSO): δ 5.78 (s, 4H, 2 NH_2), 7.53–7.97 (m, 14H, aromatic protons), 13.90 (brs, 2H, NH) ppm. ^{31}P NMR (DMSO): δ 26.0 ppm. Anal. calcd. for $C_{20}H_{20}N_8O_2P_2$ (466.37): C, 51.51; H, 4.32; N, 24.03. Found: C, 51.23; H, 4.09; N, 23.88.

7: Yellow crystals in 64% yield, m.p. 202°C–205°C. IR (in cm^{-1}): 3167, 3125 (NH_2, NH), 3058 (C—H_{arom}), 2948 (C—H_{aliph}), 1597 (C=N), 1577 (C=C), 1251 (P=O), 1223 (P=O). ^{1}H NMR (DMSO): δ 2.15 (s, 6H, 2 CH_3), 4.98 (s, 4H, 2 NH_2), 7.50–7.74 (m, 14H, aromatic protons), 13.80 (brs, 2H, NH) ppm. ^{31}P NMR (DMSO): δ 19.2 (CH_3—P=O), 26.2 (Ph—P=O) ppm. Anal. calcd. for $C_{22}H_{26}N_8O_4P_4$ (590.38): C, 44.76; H, 4.44; N, 18.98. Found: C, 44.39; H, 4.19; N, 18.70.

*5,5′-(1,4-Phenylene)bis(6-methyl-3-[{[3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl]imino}methyl]-4*H*-chromen-4-one) (**9**) and 5,5′-(1,4-Phenylene)bis{[*P*-methyl-*N*-(6-methyl-4-oxo-4*H*-chromen-3-yl)methylene]-*P*-[3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triazaphosphol-4-yl]phosphinic amide} (**10**)*

To a solution of compound **6** or **7** (5 mmol) in DMF (30 mL), 3-formyl-6-methylchromone (**8**) (1880 mg, 10 mmol) was added and the solution was heated under reflux for 4 h, cooled, and filtered off. The obtained solids were crystallized from DMSO to give **9** and **10**, respectively.

9: Pale yellow crystals in 59% yield, m.p. >300°C. IR (in cm^{-1}): 3314 (NH), 3062 (C—H_{arom}), 2978 (C—H_{aliph}), 1640 (C=O), 1601 (C=N), 1581 (C=C), 1281 (P=O). ^{1}H NMR (DMSO): δ 2.18 (s, 6H, 2 CH_3), 7.57–8.15 (m, 18H, aromatic protons), 8.75 (s, 2H, H-5 and H-5′), 9.14 (s, 2H, H-2 and H-2′), 9.76 (br, 2H, 2 CH=N), 13.66 (s, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 26.0 ppm. Anal. calcd. for $C_{42}H_{32}N_8O_6P_2$ (806.70): C, 62.53; H, 4.00; N, 13.89. Found: C, 62.29; H, 3.83; N, 13.61.

10: Pale brown crystals in 71% yield, m.p. >300°C. IR (in cm^{-1}): 3409 (NH), 3055 (C—H_{arom}), 2923 (C—H_{aliph}), 1660 (C=O), 1620 (C=N), 1571 (C=C), 1222, 1168 (2 P=O). ^{1}H NMR (DMSO): δ 2.19 (s, 6H, 2 CH_3—Ar), 2.39 (s, 6H, 2 CH_3—P=O), 7.55–7.75 (m, 18H, aromatic protons), 7.92 (s, 2H, H-5 and H-5′), 8.75 (s, 2H, H-2 and H-2′), 9.11 (br, 2H, 2 CH=N), 13.65 (br, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 22.1 (CH_3—P=O), 26.3 (Ph—P=O) ppm. Anal. calcd. for $C_{44}H_{38}N_8O_8P_4$ (930.71): C, 56.78; H, 4.12; N, 12.04. Found: C, 56.50; H, 3.89; N, 11.79.

*5,5′-(1,4-Phenylene)bis(diethyl[{[3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-triaza-phosphol-4-yl]amino}(6-methyl-4-oxo-4*H*-chromen-3-yl)methyl]phosphonate) (**11**) and 5,5′-(1,4-Phenylene)bis{diethyl[[(3-oxo-3-phenyl-2,3-dihydro-4*H*-1,2,4,3-tri-azaphosphol-4-yl)methylphosphoryl]amino][6-methyl-4-oxo-4*H*-chromen-3-yl) methyl]phosphonate} (**12**)*

A mixture of compound **9** or **10** (5 mmol) and diethyl phosphite (2085 mg, 15 mmol) in the presence of a few drops of triethylamine was heated at 80°C–100°C for 10 h. The excess of diethyl phosphite was removed under reduced pressure and the oily residue was treated with methanol. The obtained solids were filtered off, dried, and crystallized from DMF/methanol to give **11** and **12**, respectively.

11: White crystals in 46% yield, m.p. 194°C–196°C. IR (in cm^{-1}): 3295, 3216 (NH), 3096 (C—H_{arom}), 2906 (C—H_{aliph}), 1661 (C=O), 1599 (C=N), 1305, 1233 (2 P=O), 1075 (P—O—C). ^{1}H NMR (DMSO): δ 1.05 (t, $J = 6.8$ Hz, 6H, 2 OCH_2CH_3), 1.20 (t, $J = 7.2$ Hz, 6H, 2 OCH_2CH_3), 2.29 (s, 6H, 2 CH_3), 2.67 (br, 2H, 2 NH—P), 3.86 (br, 8H, 4 OCH_2CH_3), 5.36 (br, 2H, 2 CH—P), 7.18–8.10 (m, 22H, aromatic protons), 14.15 (br, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 21.0 (EtO—P=O), 26.7 (Ph—P=O) ppm. Anal. calcd. for $C_{50}H_{54}N_8O_{12}P_4$ (1082.90): C, 55.46; H, 5.03; N, 10.35. Found: C, 55.21; H, 4.84; N, 9.98.

12: Pale yellow crystals in 57% yield, m.p. 220°C–223°C. IR (in cm^{-1}): 3198 (NH), 3092, 3036 (C—H_{arom}), 2985, 2948 (C—H_{aliph}), 1665 (C=O), 1583 (C=N), 1559 (C=C), 1301, 1290, 1215 (3 P=O), 1021 (P—O—C). ^{1}H NMR (DMSO): δ 1.06 (t, $J = 6.6$ Hz, 6H, 2 OCH_2CH_3), 1.19 (t, $J = 7.0$ Hz, 6H, 2 OCH_2CH_3), 2.15 (s, 3H, CH_3-Ar), 2.16 (s, 3H, CH_3—Ar), 2.32 (s, 3H, CH_3—P), 2.34 (s, 3H CH_3—P), 2.44 (d, $J = 3.8$ Hz, 2H, 2 NH—P), 3.97 (q, $J = 8.4$ Hz, 4H, 2 OCH_2CH_3), 4.10 (q, $J = 6.2$ Hz, 4H, 2 OCH_2CH_3), 5.17 (d, $J = 23$ Hz, 1H, CH—P), 5.24 (d, $J = 23$ Hz, 1H, CH—P), 7.52–7.77 (m, 18H, aromatic protons), 7.89 (br, 2H, H-5 and H-5′), 8.49 (br, 2H, H-2 and H-2′), 13.77 (brs, 2H, 2 NH) ppm. ^{31}P NMR (DMSO): δ 20.0 and 20.5 (90.9%: 9.1%) (CH_3—P=O), 23.1 and 23.5 (91%: 9%) (EtO—P=O), 26.5 (Ph—P=O) ppm. Anal. calcd. for $C_{52}H_{60}N_8O_{14}P_6$ (1206.92): C, 51.75; H, 5.01; N, 9.28. Found: C, 51.52; H, 4.83; N, 8.93.

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