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Dedicated to Full Member of the Russian Academy of Sciences O.N. Chupakhin on his 80th anniversary

β- and γ-Amino Acetals Containing Phosphine Oxide Groups. Synthesis and Reactions with Resorcinol Derivatives

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Abstract—New β- and γ-amino acetals containing phosphine oxide groups were synthesized by the Kabachnik–Fields reaction of β- and γ-amino acetals with paraformaldehyde and dialkylphosphine oxides. Heating of the resulting β- and γ-amino acetals with 2-methylresorcinol and pyrogallol in boiling trifluoroacetic acid afforded (dialkylphosphoryl)(ω,ω-diarylalkyl)ammonium trifluoromethanesulfonates.

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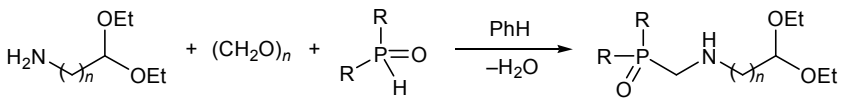
Condensation of functionalized acetals with resorcinol and its derivatives in acidic medium provides a general procedure for one-step synthesis of acyclic and cyclic polyphenols, in particular calix[4]resorcinarenes [1], compounds of the diarylmethane series [2], and heterocycles containing polyphenol fragments [3]. The results of numerous studies have shown that the synthetic outcome of this reaction depends on the conditions (acid and solvent nature), reactant structure, and reactant ratio. For instance, α-amino acetals react with resorcinol and its derivatives in ethanol in the presence of aqueous HCl to give diarylmethane derivatives [2], while unsubstituted β- and γ-amino acetals under analogous conditions give rise to calix[4]resorcinarenes [3, 4].

While continuing studies in this line, we synthesized new α-amino acetals containing phosphonate and phosphine oxide groups [5]. Phosphonate α-amino acetals reacted with resorcinol and its derivatives in ethanol to produce both ammonium salts of the diarylmethane series and 2,5-diaryl-1,4-bis(diethoxyphosphorylmethyl)piperazines. Dialkyl(2,2-dimethoxyethylaminomethyl)phosphine oxides (Alk = C_8H_{17}, $C_{10}H_{21}$) reacted with polyphenols on heating in boiling trifluoroacetic acid with formation of only diarylmethane derivatives.

In the present work we tried to elucidate how the structure of phosphorylated amino acetals (namely, the length of the hydrocarbon chain connecting the acetal group and nitrogen atom and substituents on the phosphorus atom) affects their reaction with 2-methylresorcinol and pyrogallol. For this purpose, we have synthesized for the first time β- and γ-amino acetals containing phosphine oxide groups with alkyl substituents of different lengths on the phosphorus atom. Compounds **IVa**–**IVc**, **Va**, and **Vc** were obtained by

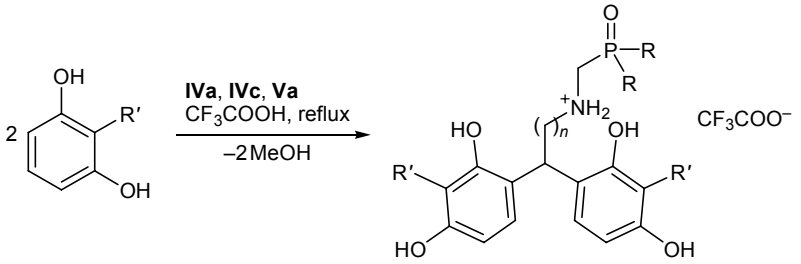
Scheme 1.



I, **II** **IIIa**–**IIIc** **IVa**–**IVc**, **Va**–**Vc**

R = C_6H_{13} (a), C_8H_{17} (b), $C_{10}H_{21}$ (c); **I**, **IV**, n = 2; **II**, **V**, n = 3.

Scheme 2.



VIa, **VIc**, **VII**, **VIII**

VI, $n = 2$, R′ = Me, R = C_6H_{13} (**a**), $C_{10}H_{23}$ (**c**); **VII**, R = C_6H_{13}, R′ = OH, $n = 2$; **VIII**, R = C_6H_{13}, R′ = Me, $n = 3$.

the Kabachnik–Fields reactions of 3,3-diethoxypropan-1-amine (**I**) and 4,4-diethoxybutan-1-amine (**II**) with dialkylphosphine oxides **IIIa**–**IIIc** and paraformaldehyde at a ratio of 1:1:1 in the presence of *p*-toluenesulfonic acid (Scheme 1). The structure of **IVa**–**IVc**, **Va**, and **Vc** was determined on the basis of their IR, ^{1}H and ^{31}P NMR, and MALDI mass spectra and elemental analyses.

Compounds **IVa**–**IVc**, **Va**, and **Vc** displayed in the ^{31}P NMR spectra only one phosphorus signal in the region δ_P 46–47 ppm. The most informative signals in their ^{1}H NMR spectra were doublets from the PCH_2N methylene protons (δ 2.67–2.83 ppm), a triplet from the NCH_2 protons in the acetal fragment (δ 2.53–2.63 ppm), a triplet from the acetal CH proton (δ 4.35–4.49 ppm), and a multiplet from the OCH_2 protons in the ethoxy groups (δ 3.23–3.61 ppm).

Acetals **IVa**–**IVc**, **Va**, and **Vc** were brought into condensation with 2-methylresorcinol and pyrogallol in acidic medium. Phosphine oxide β-amino acetals **IVa** and **IVc** failed to react with 2-methylresorcinol in ethanol in the presence of HCl. The ^{31}P NMR spectrum of the reaction mixture, like the spectra of analogous phosphine oxide α-amino acetals [5], contained signals in the region δ 53–55 ppm, indicating concurrent protonation of the P=O group and deactivation of the acid catalyst (HCl).

By varying the reaction conditions, including solvent (chloroform, benzene, toluene, dioxane) and acid nature (trifluoroacetic acid, trifluoromethanesulfonic acid) we found that the condensation occurred only on heating in boiling trifluoroacetic acid. Amino phosphine oxides **IVa** and **IVc** reacted with 2-methylresorcinol and pyrogallol to afford diarylmethane derivatives **VIa**, **VIc**, and **VII** in up to 28% yield, regardless of the alkyl substituent on the phosphorus atom and polyphenol nature. The yield of the condensation product did not increase when the ratio amino acetal–2-methylresorcinol was changed from 1:1 to 1:4 (Scheme 2). The reaction of γ-amino acetal **Va** with 2-methylresorcinol followed the same path with formation of 43% of compound **VIII**.

Ammonium salts **VIa**, **VIc**, **VII**, and **VIII** were isolated as white powders which were soluble in acetone and DMSO (**VIa**, **VIc**, **VIII**) or only in DMSO (**VII**). Their structure was proved by IR, ^{31}P and ^{1}H NMR, and MALDI mass spectra and elemental analyses.

Compounds **VIa**, **VIc**, **VII**, and **VIII** showed in the ^{1}H NMR spectra doublets from protons in the benzene rings at δ 6.28–6.42 (5-H) and 6.78–7.10 ppm (6-H), a triplet from the CH proton at δ 4.51–5.1 ppm, a doublet from the PCH_2N methylene protons at δ 3.48–3.61 ppm, and a triplet from the methylene group attached to nitrogen at δ 2.88–3.14 ppm. The downfield position of signals from the methylene protons on the nitrogen atom as compared to the corresponding signals of phosphorylated amino acetals **IVa**, **IVc**, and **Va** (Δδ = 0.4–0.8 ppm), as well as IR absorption bands at 1674–1668 (C=O) and 2500–2600 cm^{-1} (NH^+) indicated formation of ammonium trifluoroacetates.

The presence in molecules **VIa**, **VIc**, **VII**, and **VIII** of phenolic hydroxy groups and phosphorylmethylammonium fragments opens wide prospects for their practical application, e.g., as lipophilic extractants of metal ions from acidic medium [6] and multidentate ligands [7]. Compounds of the diarylmethane series offer a wide synthetic potential as intermediate products for the preparation of new macrocyclic compounds, e.g., calix[4]resorcinarenes with alternating substituents on the lower rim [8].

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance-600 spectrometer at 600.1 and 150.9 MHz, respectively, using the residual proton and

carbon signals of deuterated solvents as reference. The ^{31}P NMR spectra were measured on a Bruker Avance II-400 instrument (161.9 MHz) relative to 85% H_3PO_4 (external). The IR spectra were recorded in the range from 4000 to 400 cm^{-1} on a Bruker Vector-22 spectrometer. The mass spectra (MALDI-TOF) were obtained on a Bruker Ultraflex III spectrometer using plastic and metal targets and 2,5-dihydroxybenzoic acid as matrix.

Dihexyl(3,3-diethoxypropylaminomethyl)phosphine oxide (IVa). A mixture of 1.50 g (6.88 mmol) of dihexylphosphine oxide (**IIIa**), 1.04 g (7.07 mmol) of 3,3-diethoxypropan-1-amine (**I**), 0.21 g of paraformaldehyde, and 21 mg of *p*-toluenesulfonic acid in 20 mL of benzene was heated for 4 h under reflux in a flask equipped with a Dean–Stark trap. When the reaction was complete, 85 mg of potassium carbonate was added, the mixture was heated for 10 min under reflux and cooled, and the precipitate was filtered off. The organic layer was washed with three portions of water and dried over $MgSO_4$, and the solvent was distilled off. Yield 2 g (75%). IR spectrum, ν, cm^{-1}: 3438 br (NH), 1151 m (P=O), 1125 w (COC). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.79 t (6H, CH_3, J = 7.0 Hz), 1.10 t (6H, OCH_2CH_3, J = 7.1 Hz), 1.18–1.31 m (12H, CH_2), 1.45–1.51 m (4H, CH_2), 1.59–1.61 m (2H, $CHCH_2$), 1.62–1.72 m (4H, PCH_2), 2.65 t (2H, NCH_2, J = 6.8 Hz), 2.79 d (2H, PCH_2N, J = 8.3 Hz), 3.36–3.59 m (4H, OCH_2), 4.49 t (1H, CH, J = 5.6 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 47.41 ppm. Mass spectrum, *m*/*z*: 378 $[M + H]^+$, 400 $[M + Na]^+$, 416 $[M + K]^+$. Found, %: C 63.47; H 11.69; N 3.79; P 8.13. $C_{20}H_{44}NO_3P$. Calculated, %: C 63.66; H 11.67; N 3.71; P 8.22.

Compounds **IVb**, **IVc**, **Va**, and **Vc** were synthesized in a similar way.

(3,3-Diethoxypropylaminomethyl)dioctylphosphine oxide (IVb) was synthesized from 1.50 g (5.47 mmol) of dioctylphosphine oxide (**IIIb**), 0.83 g (5.64 mmol) of compound **I**, and 0.17 g of paraformaldehyde in the presence of 16.6 mg of *p*-toluenesulfonic acid. Yield 1.6 g (68%). IR spectrum, ν, cm^{-1}: 3283 br (NH), 1222 m (P=O), 1125 w (COC). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.76 t (6H, CH_3, J = 7.0 Hz), 1.07 t (6H, OCH_2CH_3, J = 7.1 Hz), 1.15–1.28 m (20H, CH_2), 1.41–1.49 m (4H, PCH_2CH_2), 1.56–1.62 m (4H, PCH_2), 1.64–1.69 m (2H, $CHCH_2$), 2.62 t (2H, NCH_2, J = 6.7 Hz), 2.76 d (2H, PCH_2N, J = 8.17 Hz), 3.33–3.56 m (4H, OCH_2), 4.47 t (1H, CH, J = 5.5 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 47.46 ppm. Mass spectrum, *m*/*z*: 434 $[M + H]^+$, 456 $[M + Na]^+$, 473 $[M + K]^+$. Found, %: C 66.34; H 12.08; N 3.19; P 6.99; $C_{24}H_{52}NO_3P$. Calculated, %: C 66.51; H 12.00; N 3.20; P 7.10.

Didecyl(3,3-diethoxypropylaminomethyl)phosphine oxide (IVc) was synthesized from 1.50 g (4.55 mmol) of didecylphosphine oxide (**IIIc**), 0.83 g (5.64 mmol) of compound **I**, and 0.16 g of paraformaldehyde in the presence of 16.60 mg of *p*-toluenesulfonic acid. Yield 1.89 g (82%). IR spectrum, ν, cm^{-1}: 3436 br (NH), 1220 m (P=O), 1123 w (COC). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.66 t (6H, CH_3, J = 7.0 Hz), 0.98 t (6H, OCH_2CH_3, J = 7.1 Hz), 1.03–1.19 m (28H, CH_2), 1.32–1.39 m (4H, PCH_2CH_2), 1.47–1.51 m (4H, PCH_2), 1.53–1.60 m (2H, $CHCH_2$), 2.53 t (2H, NCH_2, J = 6.7 Hz), 2.67 d (2H, PCH_2N, J = 8.2 Hz), 3.23–3.46 m (4H, OCH_2), 4.38 t (1H, CH, J = 5.5 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 47.36 ppm. Mass spectrum, *m*/*z*: 490 $[M + H]^+$. Found, %: C 68.64; H 12.28; N 2.90; P 6.36. $C_{28}H_{60}NO_3P$. Calculated, %: C 68.71; H 12.27; N 2.86; P 6.34.

Dihexyl(4,4-diethoxybutylaminomethyl)phosphine oxide (Va) was synthesized from 2 g (9.17 mmol) of dihexylphosphine oxide (**IIIa**), 1.48 g (9.19 mmol) of 4,4-diethoxybutan-1-amine (**II**), and 0.28 g of paraformaldehyde in the presence of 30 mg of *p*-toluenesulfonic acid. Yield 2.9 g (81%). IR spectrum, ν, cm^{-1}: 3436 br (NH), 1151 m (P=O), 1126 w (COC). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.76 t (6H, CH_3, J = 7.0 Hz), 1.07 t (6H, OCH_2CH_3, J = 7.1 Hz), 1.15–1.28 m (16H, CH_2), 1.41–1.53 m (4H, $CHCH_2$), 1.56–1.62 m (4H, PCH_2CH_2), 2.55 t (2H, NCH_2, J = 6.9 Hz), 2.75 d (2H, PCH_2N, J = 8.1 Hz), 3.32–3.54 m (4H, OCH_2), 4.35 t (1H, CH, J = 5.6 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 47.33 ppm. Mass spectrum, *m*/*z*: 414 $[M + Na]^+$, 430 $[M + K]^+$. Found, %: C 64.11; H 11.88; N 3.44; P 8.02. $C_{21}H_{46}NO_3P$. Calculated, %: C 64.45; H 11.76; N 3.58; P 7.93.

Didecyl(4,4-diethoxybutylaminomethyl)phosphine oxide (Vc) was synthesized from 1.5 g (4.55 mmol) of didecylphosphine oxide (**IIIc**), 0.73 g (4.53 mmol) of amino acetal **II**, and 0.14 g of paraformaldehyde in the presence of 15 mg of *p*-toluenesulfonic acid. Yield 2.0 g (88%). IR spectrum, ν, cm^{-1}: 3277 br (NH), 1143 m (P=O), 1125 w (COC). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.82 t (6H, CH_3, J = 6.9 Hz), 1.14 t (6H, OCH_2CH_3, J = 7.1 Hz), 1.20–1.35 m (28H, CH_2), 1.45–1.53 m (4H, PCH_2CH_2), 1.55–1.62 m (4H, $CHCH_2$), 1.63–1.72 m (4H, PCH_2), 2.63 t (2H, NCH_2, J = 6.9 Hz), 2.83 d (2H, PCH_2N, J =

8.0 Hz), 3.39–3.61 m (4H, OCH_2), 4.42 t (1H, CH, J = 5.5 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 47.40 ppm. Mass spectrum, m/z: 504 $[M + H]^+$. Found, %: C 69.02; H 12.38; N 2.76; P 6.24. $C_{29}H_{62}NO_3P$. Calculated, %: C 69.18; H 12.33; N 2.78; P 6.16.

***N*-(Dihexylphosphorylmethyl)-3,3-bis(2,4-dihydroxy-3-methylphenyl)propan-1-aminium trifluoroacetate (VIa).** A mixture of 0.5 g (1.33 mmol) of compound **IVa** and 0.41 g (3.31 mmol) of 2-methylresorcinol in 2 mL of trifluoroacetic acid was heated for 4 h under reflux. When the reaction was complete, trifluoroacetic acid was removed under reduced pressure (water-jet pump), and the oily residue was either ground or washed with diethyl ether. The precipitate was filtered off and dried under reduced pressure (oil pump). Yield 0.24 g (28%), mp 186°C. IR spectrum, ν, cm^{-1}: 3403 br (OH), 2652 br (NH^+), 1674 s (C=O), 1608 m ($C=C_{arom}$), 1183 m (P=O). ^1H NMR spectrum (acetone-d_6), δ, ppm: 0.91 t (6H, CH_3, J = 6.8 Hz), 1.31–1.65 m (16H, CH_2), 2.08–2.13 m (4H, PCH_2), 2.16 s (6H, 3′-CH_3), 2.57 m (2H, CHC**H**$_2$), 3.14 t (2H, NCH_2, J = 7.5 Hz), 3.61 d (2H, PCH_2N, J 6.4 = Hz), 4.61 t (1H, CH, J = 8.0 Hz), 6.47 d (2H, 5′-H, J = 8.4 Hz), 6.82 d (2H, 6′-H, J = 8.4 Hz). ^{31}P NMR spectrum (acetone-d_6): δ_P 46.92 ppm. Mass spectrum, m/z: 534 $[M - CF_3COOH + H]^+$, 556 $[M - CF_3COOH + Na]^+$, 572 $[M - CF_3COOH + K]^+$. Found, %: C 59.23; H 7.54; N 2.15; P 4.66. $C_{30}H_{48}NO_5P \cdot CF_3COOH$. Calculated, %: C 59.35; H 7.57; N 2.16; P 4.79.

Compounds **VIc**, **VII**, and **VIII** were synthesized in a similar way.

***N*-(Didecylphosphorylmethyl)-3,3-bis(2,4-dihydroxy-3-methylphenyl)propan-1-aminium trifluoroacetate (VIc)** was synthesized from 0.50 g (1.02 mmol) of compound **IVc** and 0.31 g (2.55 mmol) of 2-methylresorcinol. Yield 0.21 g (27%), mp 190°C. IR spectrum, ν, cm^{-1}: 3386 br (OH), 2614 br (NH^+), 1610 m ($C=C_{arom}$), 1670 s (C=O), 1182 m (P=O). ^1H NMR spectrum (acetone-d_6), δ, ppm: 0.84 t (6H, CH_3, J = 6.8 Hz), 1.99–2.04 m (4H, PCH_2), 2.07 s (6H, 3′-CH_3), 1.24–1.59 (32H, CH_2), 2.42 m (2H, CHC**H**$_2$), 3.19 t (2H, NCH_2, J = 7.6 Hz), 3.64 d (2H, PCH_2N, J = 6.4 Hz), 4.55 t (1H, CH, J = 7.7 Hz), 6.35 d (2H, 5′-H, J = 8.3 Hz), 6.51 d (2H, 6′-H, J = 8.4 Hz). ^{31}P NMR spectrum (acetone-d_6): δ_P 47.17 ppm. Mass spectrum, m/z: 646 $[M - CF_3COOH + H]^+$. Found, %: C 63.12; H 8.59; N 1.99; P 4.11. $C_{38}H_{64}NO_5P \cdot CF_3COOH$. Calculated, %: C 63.24; H 8.56; N 1.84; P 4.08.

***N*-(Dihexylphosphorylmethyl)-3,3-bis(2,3,4-trihydroxyphenyl)propan-1-aminium trifluoroacetate (VII)** was synthesized from 0.50 g (1.33 mmol) of compound **IVa** and 0.42 g (3.33 mmol) of pyrogallol in 2 mL of trifluoroacetic acid. The residue was washed with acetone. Yield 0.27 g (31%), mp 197°C. IR spectrum, ν, cm^{-1}: 3421 br (OH), 2550 br (NH^+), 1672 s (C=O), 1630 m ($C=C_{arom}$), 1202 m (P=O). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 0.88 t (6H, CH_3, J = 6.8 Hz), 1.32–1.47 m (16H, CH_2), 1.75–1.84 m (4H, PCH_2), 2.16–2.18 m (2H, CHC**H**$_2$), 2.88 t (2H, NCH_2, J = 7.9 Hz), 3.56 d (2H, PCH_2N, J = 6.3 Hz), 4.42 t (1H, CH, J = 7.6 Hz), 6.23 d (2H, 5′-H, J = 8.4 Hz), 6.37 d (2H, 6′-H, J = 8.4 Hz). ^{31}P NMR spectrum (DMSO-d_6): δ_P 47.10 ppm. Mass spectrum, m/z: 538 $[M - CF_3COOH + H]^+$. Found, %: C 55.21; H 6.79; N 2.20; P 4.81. $C_{28}H_{44}NO_7P \cdot CF_3COOH$. Calculated, %: C 55.30; H 6.91; N 2.15; P 4.76.

***N*-(Dihexylphosphorylmethyl)-4,4-bis(2,4-dihydroxy-3-methylphenyl)butan-1-aminium trifluoroacetate (VIII)** was synthesized from 0.30 g (0.77 mmol) of compound **Va** and 0.25 g (2.02 mmol) of 2-methylresorcinol. Yield 0.22 g (43%), mp 118°C. IR spectrum, ν, cm^{-1}: 3439 br (OH), 2497 br (NH^+), 1667 s (C=O), 1620 m ($C=C_{arom}$), 1143 m (P=O). ^1H NMR spectrum (acetone-d_6), δ, ppm: 0.90 t (6H, CH_3, J = 6.7 Hz), 1.33–1.54 m (4H, PCH_2), 1.96–2.00 m (16H, CH_2), 1.99 s (6H, 3′-CH_3), 2.09–2.14 m (2H, NCH_2CH_2), 3.25 t (2H, NCH_2, J = 7.7 Hz), 3.56 d (2H, PCH_2N, J = 6.3 Hz), 4.45 t (1H, CH, J = 7.7 Hz), 6.44 d (2H, 5′-H, J = 8.4 Hz), 6.90 d (2H, 6′-H, J = 8.4 Hz). ^{31}P NMR spectrum (acetone-d_6): δ_P 47.00 ppm. Mass spectrum, m/z: 548 $[M - CF_3COOH + H]^+$, 570 $[M - CF_3COOH + Na]^+$, 586 $[M - CF_3COOH + K]^+$. Found, %: C 59.71; H 7.47; N 2.18; P 4.76. $C_{31}H_{50}NO_3P \cdot CF_3COOH$. Calculated, %: C 59.90; H 7.71; N 2.12; P 4.69.

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