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Synthesis of Phthalic Aldehyde and Its Diacetals

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Abstract—Acyclic phthalaldehyde diacetal without cyclic 1,3-dihydro-1,3-dimethoxybenzo[*c*]furan impurity has been obtained via the reaction of 1,2-bis(dibromomethyl)benzene with trimethyl orthoformate (1 : 6) at 90°C in the presence of 10 mol% of $ZnCl_2$. Hydrolysis of phthalaldehyde diacetal has led to the formation of phthalaldehyde without HBr evolution. The reaction of phthalaldehyde with trimethyl orthoformate in the presence of trifluoroacetic acid has proceeded abnormally, with the formation of the cyclic diacetal. The acyclic diacetal has been phosphorylated by chlorophosphines and the action of PCl_3 and a P(III) acid ester in sequence.

Keywords: phthalaldehyde, phthalaldehyde diacetals, 1,3-dihydro-1,3-dimethoxybenzo[*c*]furan, 1,2-bis-(dibromomethyl)benzene, phosphorylation

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Phthalic aldehydes exhibit strong bactericide activity and are excellent disinfectants [1]. Various formulations based on them are widely used in premises decontamination and sterilization of medical equipment, especially related to endoscopy. Therefore, development of novel methods of phthalic aldehydes synthesis from available sources is a topical issue.

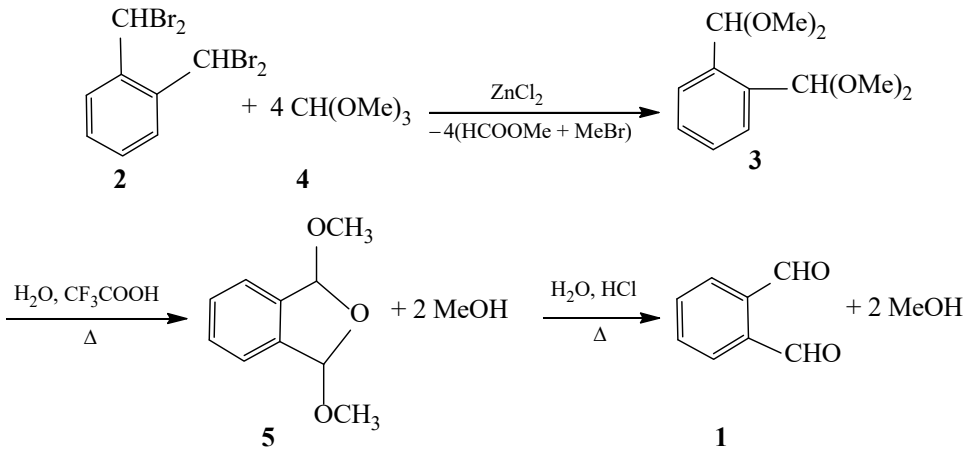
The known methods of synthesis of phthalic aldehyde **1** are based on various reactions of 1,2-disubstituted benzenes: hydrolysis, oxidation, bis(*gem*-diacetate) deprotection, and reduction. The first methods of synthesis of phthalic aldehydes have been based on the hydrolysis of bis(dihalomethyl)benzenes with fuming or concentrated sulfuric acid, and their further development consisted in the methods of neutralization of the evolving hydrogen halide [1–6]. Hydrolysis of acyclic diacetals of phthalic aldehydes has been performed in the presence of sulfuric [7], formic [8], phthalic [9], acetic [8], or hydrochloric [10] acid as well as sodium hydroxide [7, 10].

The related oxidation processes have involved the following substrates: *o*-xylene with oxygen in water [11] or in chloroform in the presence of 10-methyl-9-phenyl-10-acridinium perchlorate [12]; naphthalene with potassium metaperiodate in the presence of potassium aquapentachlororuthenate(III) in water, dichloromethane, or acetonitrile [13], with potassium bromate in the presence of Ru-catalyst in dichloromethane, water, or acetonitrile under ultrasonication [14], with sodium periodate in dichloromethane, water, or acetonitrile under ultrasonication [15], with ozone in methanol or butyl acetate [16], or with sodium iodate in the presence of the $Ru_3(CO)_{12}$ catalyst in acetonitrile, water, or dichloromethane [16]; 1,2-di(hydroxymethyl)benzene with manganese dioxide in dichloromethane [17], with iron nitrate [18] or oxygen in the presence of $Ru(PPh_3)_3Cl_2$ in various solvents [19], with dipyridinesilver permanganate in benzene [20], or with dimethyl sulfoxide and oxalyl chloride in dichloromethane [21]; 1,2-di(bromomethyl)-benzene with di(4-methoxyphenyl) selenoxide and potassium hydrocarbonate in acetonitrile [22].

Bis(*gem*-diacetate) deprotection has been performed via the heating of phthalic aldehyde bis(diacetate) with poly(4-vinylpyridinium) chlorate [P(4-VPH)ClO_4] in ethanol [23] or poly(4-vinylpyridinium) hydrosulfate in methanol under ultrasonication [24] or via keeping of bis(diacetate) and *N*-sulfonopoly-(4-vinylpyridinium) chloride in methanol [25].

The following substrates have been subject to reduction: Восстановлению подвергали фталевые кислоты и их производные: phthalic acid in the presence of *tert*-hexyl-*s*-butoxyborane in tetrahydrofuran [26] or in the presence of $LiAlH_4$ [27]; phthaloyl dichloride in the presence of pentacoordinate hydrosilane [28]; phthaloyl diamide in the presence of $LiAlH_4$ [29]; and phthaloyl dinitrile

Scheme 1.



in the presence of sodium hydrotris(dibutylamino)-aluminate in tetrahydrofuran [30].

It should be underscored that hydrolysis of readily available 1,2-bis(dibromomethyl)benzene **2** is among the oldest [1–6] yet used up to now [7–10] methods of phthalic aldehyde synthesis. However, this method suffers from several drawbacks, the major one being vigorous evolution of hydrogen bromide corroding the equipment and limiting the chemicals loading.

Herein we elaborated a novel method of preparation of phthalic aldehyde **1** from 1,2-bis(dibromomethyl)benzene **2** avoiding the formation of HBr. First, the tetrabromide **2** reacted with trimethyl orthoformate to be converted into the acyclic diacetal **3**, which was transformed into phthalic aldehyde **1** upon heating in acidified water. The ^{1}H NMR study revealed that cyclic phthalic aldehyde diacetal (1,3-dihydro-1,3-dimethoxybenzo[*c*]furan **5**) was initially formed at room temperature and then was converted into the aldehyde **1** upon heating in acidic aqueous medium (Scheme 1).

The last stage of the scheme was performed separately, the yield of phthalic aldehyde **1** being 81%.

As seen in Scheme 1, phthalic aldehyde diacetals **3** and **5** were intermediate products, and therefore their synthesis became an important issue. Acetals have been successfully prepared via the interaction of available organic *gem*-dihalides with alkali metals alcoholates [31–37]: di(dehaloalkoxylation) of aryldihalomethanes or benzylidene halides. The interaction has involved *gem*-dichlorides [33–36] and (much rarer) *gem*-dibromides [37–39]. The major drawbacks of this approach are the need to prepare an alkali metal alcoholate and the possibility of transformation of other functional groups under the action of a strong base (alkoxide anion).

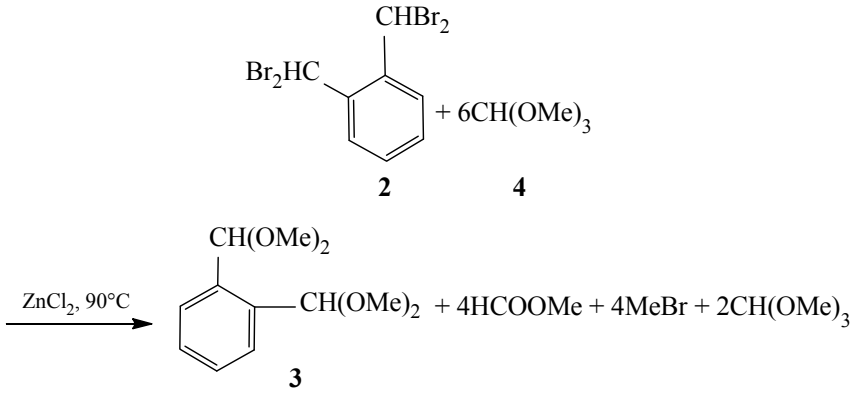
We were the first to use trialkyl orthoformates instead of alcoholates for di(dehaloalkoxylation) of dihalomethylarenes [40]. We have synthesized a series of acetals of substituted benzaldehydes including terephthalic aldehyde diacetals **6** via the interaction of 1,4-bis(dibromomethyl)benzene **7** with orthoformates **4** at 50°C during 4–5 h in the presence of 10 mol% of zinc chloride as catalyst [41–42].

The corresponding acyclic phthalic aldehyde diacetals **3** have not been described in the literature, however their synthesis has been attempted via the interaction of phthalic aldehyde **1** with ethanolic solution of triethyl orthoformate (1 : 1) in the presence of aluminum chloride as catalyst [43], via acetalization of aldehyde **1** with methanol using catalytic amount of $TiCl_4$ or in the presence of NH_3 or Et_3N [38], and via the reaction of aldehyde **1** with dimethyl sulfite in methanol [44]. A mixture of acyclic diacetal **3** and 1,3-dihydro-1,3-dialkoxybenzo[*c*]furan **5** has been formed in the mentioned cases.

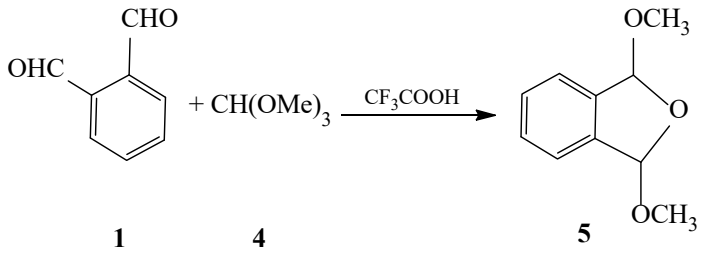
We found that, in contrast to 1,4-bis(dibromomethyl)-benzene **7**, 1,2-bis(dibromomethyl)benzene **2** practically did not interact with trimethyl orthoformate under the mentioned conditions, the latter being decomposed into methyl formate. However, acyclic phthalic aldehyde diacetals **3** were formed in high yield at 90°C or above and at the threefold excess of the orthoester (ratio 1 : 6) (Scheme 2).

Hence, we elaborate a novel method of the synthesis of acyclic phthalic aldehyde diacetal **3** free of 1,3-dihydro-1,3-dialkoxybenzo[*c*]furan **5** admixture.

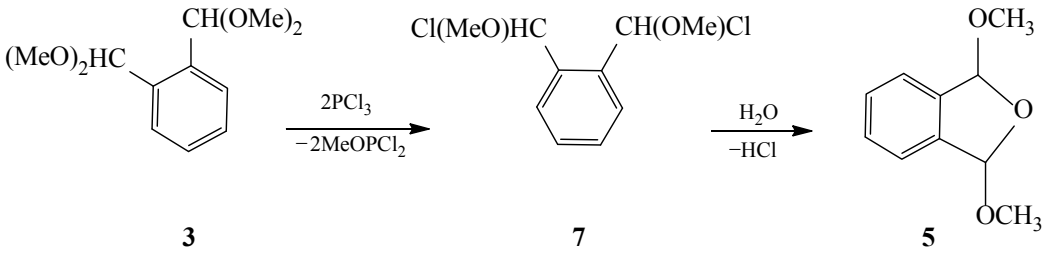
Scheme 2.



Scheme 3.



Scheme 4.



It is known that terephthalic aldehyde diacetals **6** can be formed via the interaction of the aldehyde with orthoesters **4** in the presence of acids [43]. We were the first to find that the interaction of phthalic aldehyde **1** with orthoesters **4** in the presence of trifluoroacetic acid as catalyst occurred abnormally to yield cyclic diacetal **5** in good yield (Scheme 3).

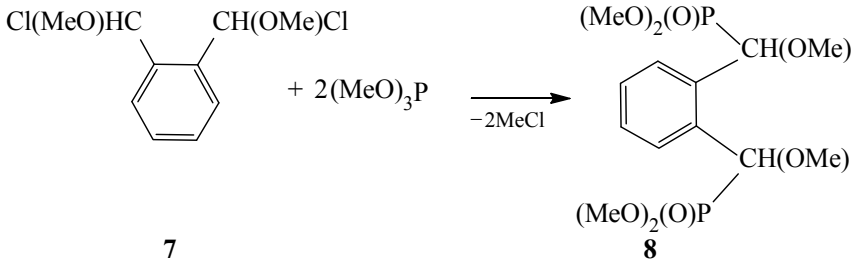
Compound **5** was also prepared from acyclic diacetal **3**: the interaction of the latter with PCl_3 gave the corresponding di-α-chloroether **7**, hydrolysis of which led to cyclic diacetal **5** (Scheme 4).

The structure of intermediate compound **7** was confirmed by the ^1H NMR data and by the reaction with trimethyl phosphite affording diphosphonate **8** (Scheme 5).

Diacetal **3** also reacted with secondary chlorophosphines **9** to yield tertiary diphosphine dioxides **10**. Evidently, the reaction occurred through intermediate formation of di-α-chloroether **7** and *O*-methylphosphinites **11**, the interaction of which gave diphosphine dioxides **10** (Scheme 6).

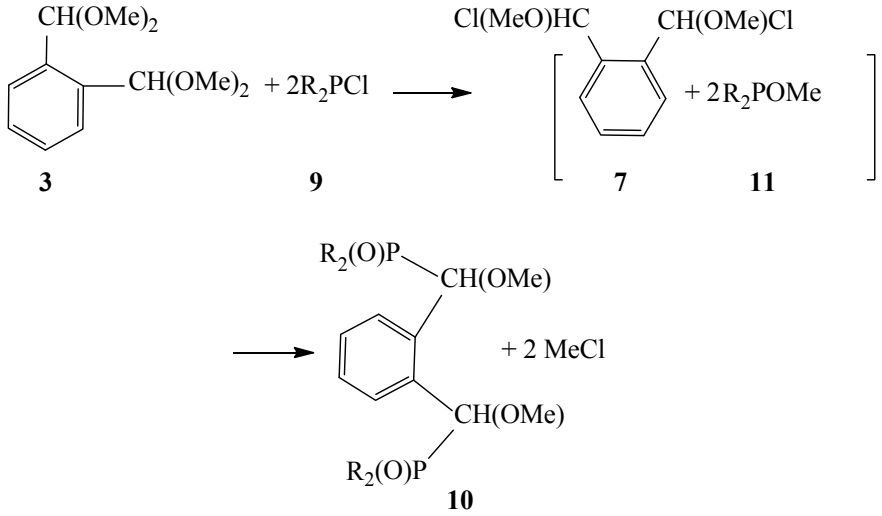
In summary, we elaborated a novel method of phthalic aldehyde synthesis avoiding evolution of HBr, via the conversion of 1,2-bis(dibromomethyl)-benzene into the acyclic diacetal under the action of trimethyl orthoformate, followed by hydrolysis. A novel method was proposed to prepare acyclic phthalic aldehyde diacetal without cyclic one (1,3-dihydro-1,3-dimethoxybenzo[*c*]furan) admixture via the interaction of 1,2-bis(dibromomethyl)benzene with trimethyl orthoformate (1 : 6) at 90°C in the presence of 10 mol % of $ZnCl_2$. Moreover, we developed two methods of synthesis of cyclic phthalic aldehyde diacetal: via the

Scheme 5.



7 **8**

Scheme 6.



3 **9** **7** **11** **10**

R = Et (**a**), Ph (**b**).

reaction of the aldehyde with trimethyl orthoformate in the presence of trifluoroacetic acid and via the interaction of the diacetal **3** with PCl_3, followed by hydrolysis of the di-α-chloroether intermediate; phosphorylation of the diacetal with secondary chlorophosphines and via sequential action of PCl_3 and P(III) acid ester was realized.

EXPERIMENTAL

^1H and ^{13}C NMR spectra were recorded using Tesla BS–567A (100 MHz) and AVANCE 400WB (400.13 and 100.61 MHz) instruments. ^{31}P NMR spectra were recorded using an AVANCE 400WB instrument (161.98 MHz) reporting the chemical shifts of phosphorus nuclei relative to 85% H_3PO_4.

Commercial *o*-xylene, bromine, trimethyl orthoformate, and phosphorus trichloride were used. 1,2-Bis(dibromomethyl)benzene **2** was synthesized as described elsewhere [1]. The solvents were purified and dried via conventional procedures [45].

Phthalic aldehyde (1). *a*. A mixture of 12.00 g (0.028 mol) of 1,2-bis(dibromomethyl)benzene **2**, 17.83 g (0.168 mol) of trimethyl orthoformate **4**, and 0.57 g (0.0042 mol) of zinc chloride was heated at 90°C during 5 h. The reaction mass was cooled, treated with 50 mL of isooctane, and filtered. 5 g (0.278 mol) of water and 3 drops of HCl were added to the solution, and the obtained mixture was heated (80°C) during 2 h distilling off the formed methanol. The organic layer was separated and dried over $MgSO_4$. The solvent was removed, and the crystalline residue was recrystallized from petroleum ether. Yield 2.97 g (79%), mp 58°C (mp 56.5–58°C [46]). ^1H NMR spectrum (acetone-d_6), δ, ppm: 7.85 d. d and 8.01 d. d (4H, C_6H_4, J_{HH} = 3.2, 5.2 Hz), 10.51 s (2H, CHO). ^{13}C NMR spectrum (acetone-d_6), δ_C, ppm: 130.72 (2C, *o*-CH_{Ar}), 133.68 (2C, *m*-CH_{Ar}), 136.65 (2C, *C*CHO), 192.00 (2C, CHO).

b. 3 mL of water and 3–4 drops of HCl were added to a solution of 1.8 g (0.01 mol) of 1,3-dihydro-1,3-dimethoxybenzo[*c*]furan **5** in 10 mL of CCl_4, and the

obtained mixture was boiled distilling off the formed methanol. The residue was dried under vacuum and recrystallized from petroleum ether. Yield 1.08 g (81%).

Phthalic aldehyde bis(dimethyl)acetal (3). A mixture of 12.00 g (0.028 mol) of 1,2-bis(dibromomethyl)benzene **2**, 17.83 g (0.168 mol) of trimethyl orthoformate **4**, and 0.57 g (0.0042 mol) of zinc chloride was heated at 90°C during 5 h. The reaction mass was cooled, treated with 50 mL of isooctane, and filtered. Isooctane was removed under vacuum, and the residue was distilled. Yield 5.25 g (85%), colorless liquid, bp 65–66°C (0.3 mmHg). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.29 s (12H, OMe), 5.67 s (2H, CHO_2), 7.30 d. d and 7.57 d. d (4H, C_6H_4, J_{HH} = 3.4, 6.4 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 47.76 (4C, OMe), 95.38 (2C, CH), 121.55 (2C, *m*-CH_{Ar}), 122.91 (2C, *o*-CH_{Ar}), 130.49 (2C, $2C_{Ar}$).

1,2-Di[(chloro)methoxymethyl]benzene (7). A solution of 2.54 g (0.011 mol) of phthalic aldehyde bis(dimethyl)acetal **3** in 5 mL of CCl_4 was added dropwise to 6.04 g (0.044 mol) of PCl_3 at 5°C. The mixture was stirred during 1 h at that temperature. The compound was thermally unstable and could be identified in crude form after elimination of the solvent and volatile product under vacuum (0.05 mmHg) on cooling. Yield 2.30 g (89%) of crude compound **7**. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.86 s (6H, OMe), 66.92 s (2H, CHCl), 7.53 m and 7.76 m (4H, C_6H_4).

1,3-Dihydro-1,3-dimethoxybenzo[*c*]furan (5). *a*. 2 mL (0.11 mol) of water was added to a solution of 2.3 g (0.0098 mol) of 1,2-di[(chloro)methoxymethyl] benzene **7** in 5 mL of CCl_4 at room temperature, and the mixture was stirred during 0.5 h. The aqueous layer was separated, the organic layer was dried over K_2CO_3. The solvent was removed under vacuum, and the residue was distilled. Yield 1.27 g (72%) of compound **5** as a 65 : 35 mixture of *cis*/*trans*-isomers, bp 62–63°C (0.2 mmHg). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.32 s (6H, OMe, *trans*), 3.35 s (6H, OMe, *cis*), 5.99 s (2H, C*H*OMe, *cis*), 6.24 s (2H, C*H*OMe, *trans*), 7.37 m and 7.43 m (4H, CH_{Ar}). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 53.28 (2C, OMe, *trans*), 55.45 (2C, OMe, *cis*), 105.25 (2C, CHO_2, *cis*), 106.39 (2C, CHO_2, *trans*), 122.92, 129.47, and 138.85 (6H, C_6H_4, *trans*), 122.97, 129.36, and 138.68 (6H, C_6H_4, *cis*).

b. 2 drops of trifluoroacetic acid were added to a mixture of 1.0 g (0.0075 mol) of phthalic aldehyde **4** and 3.18 g (0.03 mol) of trimethyl orthoformate **6**. The reaction mixture temperature was increased to 42°C. After 24 h, excess of the orthoester and other volatile compounds were removed under vacuum, and the residue was distilled. Yield 1.17 g (85%).

1,2-Benzenebis[(methoxymethyl)dimethoxyphosphonate] (8). A solution of 4.1 g (0.034 mol) of trimethyl phosphite in 5 mL of benzene was added under dry argon stream to a solution of 3.7 g (0.157 mol) of bis[(chloro)methoxymethyl]benzene **7** [prepared from 3.87 g of 1,2-bis(dimethoxymethyl)benzene] in 10 mL of anhydrous benzene. The reaction mass was stirred at 20°C during 1 h, heated at 60°C during 2 h, and kept under vacuum (0.02 mmHg, 60°C) during 1 h. Yield 5.2 g (82%), viscous liquid. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.31 s (6H, OMe), 3.53 d and 3.71 d (12H, POMe, J_{HH} = 10.4 Hz), 5.12 d (2H, CHP, $^2J_{PH}$ = 12.8 Hz), 7.28 d. d (2H, *m*-CH_{Ar}, J_{HH} = 5.2, 3.6 Hz), 7.50 d (2H, *o*-CH_{Ar}, J_{HH} = 3.6 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 53.11 and 53.93 (2C, OMe), 58.38 d (4C, $POCH_3$, $^2J_{PC}$ = 6.3 Hz), 75.20 d (2C, CH, $^1J_{PC}$ = 166.5 Hz), 127.13, 128.29, and 133.57 (6C, C_6H_4). Found, %: C 44.21; H 6.62; P 15.98. $C_{14}H_{24}O_8P_2$. Calculate, %: C 43.99; H 6.33; P 16.20.

1,2-Benzenebis[(methoximethyl)diphenylphosphine oxide] (10b). 1.28 g (0.0055 mol) of compound **3** was added dropwise at stirring to a solution of 2.5 g (0.011 mol) of diphenylchlorophosphine in 10 mL of isooctane. The reaction mixture temperature was increased to 27°C. The mixture was heated at 45–50°C during 2.5 h and then left for a day at room temperature. The crystals were filtered off and dried. Yield 2.45 g (79%), mp 183–184°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 3.46 s (6H, OCH_3), 6.48 d (2H, CHP, $^2J_{PH}$ = 10.8 Hz), 6.82 d. d and 6.90 d. d (4H, C_6H_4, J_{HH} = 5.6, 3.6 Hz), 7.97 d. d and 7.66 d. d (8H, *o*-CH_{Ar}, $^3J_{PH}$ = 11.0, J_{HH} = 7.2 Hz), 7.37 t (4H, *p*-CH_{Ar}, J_{HH} = 6.0 Hz), 7.48–7.59 m (8H, *m*-CH_{Ar}). ^{31}P NMR spectrum ($CDCl_3$): δ_P 31.67 ppm. Found, %: C 72.27; H 5.51; P 10.81. $C_{34}H_{32}O_4P_2$. Calculated, %: C 72.08; H 5.69; P 10.93.

1,2-Benzenebis[(methoxymethyl)diethylphosphine oxide (10a). 2.26 g (0.01 mol) of compound **3** was added dropwise at stirring and cooling (10°C) to 2.48 g (0.02 mol) of diethylchlorophosphine. The obtained crystalline mass was recrystallized from isooctane. Yield 2.77 g (74%), mp 102–104°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.91 t, 0.96 t, 1.29 t, and 1.33 t (12H, CH_2CH_3, J_{HH} = 7.6, $^3J_{PH}$ = 6.8 Hz), 1.47–1.62 m and 1.84–1.93 m (8H, PCH_2), 5.86 d (2H, CHP, $^2J_{PH}$ = 12.0 Hz), 7.29 d. d (2H, *m*-CH_{Ar}, J_{HH} = 5.2, 3.6 Hz), 7.42

d. d (2H, *o*-CH_{Ar}, $^3J_{PH}$ = 5.2, J_{HH} = 3.6 Hz). ^{31}P NMR spectrum ($CDCl_3$): δ_P 53.99 ppm. Found, %: C 57.63; H 8.52; P 16.48. $C_{18}H_{32}O_4P_2$. Calculated, %: C 57.75; H 8.62; P 16.55.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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