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To the 100th Anniversary of A.N. Pudovik

2-Ethoxy-2,3-dihydro[*d*][1,2]oxaphosphole 2-Oxide in the Synthesis of Dialkyl(diaryl)(2-hydroxybenzyl)phosphine Oxides

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Abstract—A facile method of synthesis of functionally substituted P(IV) derivatives—dialkyl(diaryl)(2-hydroxybenzyl)phosphine oxides—by the reaction of 2-ethoxy-2,3-dihydro[*d*][1,2]oxaphosphole 2-oxide with organomagnesium compounds, which allows wide variation of substituents on the phosphorus atom was developed.

Keywords: phosphine oxides, dihydrobenzophospholes, phosphonates, Grignard reagent, salicyl alcohol

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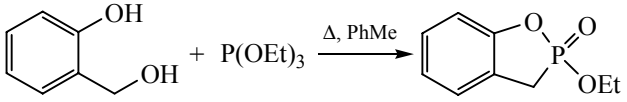
Phosphine oxides are one of the most widely used organophosphorus compounds. They are used as extractants [1, 2], ligands for metal complex catalysts [3–5] and photoluminescent complexes [6–8], as well as organocatalysts [9, 10]. Phosphine oxides containing substituents with carbonyl [11–14] or hydroxy groups [15, 16] can extend the range of potential applications of this class of compounds.

One of the most facile and convenient synthetic approaches to phosphine oxides is based on reactions of phosphorus acid halides with organomagnesium compounds to form a P–C bond [17–19]. These reactions allow one to synthesize only simple trialkyl- or triarylphosphine oxides containing no additional functionalities. Reactions of five- and six-membered phosphorus heterocycles already containing one P–C bond with organomagnesium compounds allow to synthesize a functionally substituted phosphine oxides with oxoalkyl [20], hydroxyvinyl [21], and hydroxyphenyl fragments [22, 23].

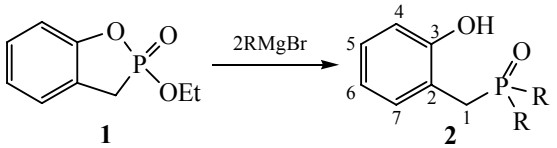
In the present work we describe a facile method of synthesis of dialkyl(diaryl)(2-hydroxybenzyl)phosphine oxides by the reaction of 2-ethoxy-2,3-dihydro-[*d*][1,2]oxaphosphole 2-oxide (**1**) with organomagnesium compounds.

The starting benzophosphole **1** was prepared by the reaction of salicyl alcohol with triethyl phosphate as described in [24].



1

Compound **1** was reacted with organomagnesium compounds in a 1 : 2 ratio in THF to obtain dialkyl-(diaryl)(2-hydroxynenzyl)phosphine oxides **2a**–**2d** in 53–63% yields.

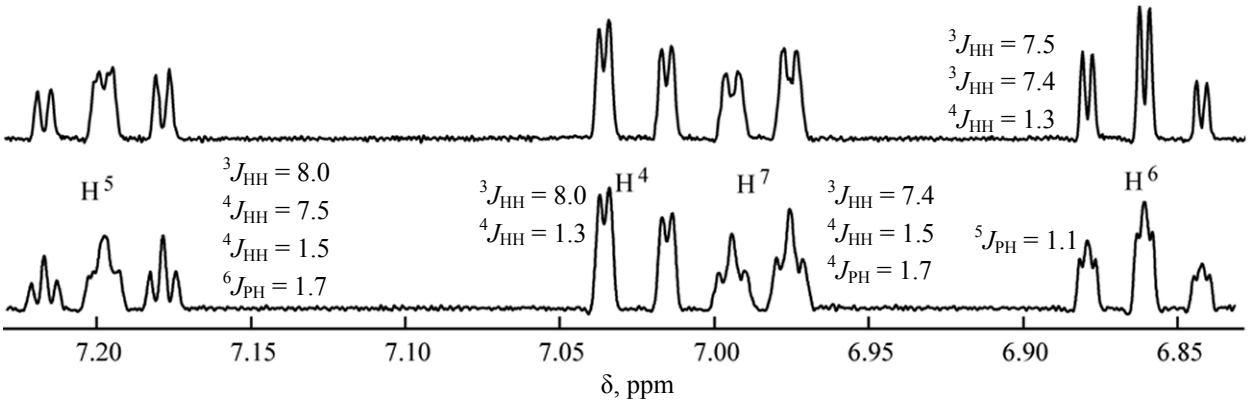


1 **2**

R = Et (**2a**), Pr (**2b**), Bu (**2b**), Ph (**2d**).

Phosphine oxides **2a**, **2c**, and **2d** were previously prepared by the reactions of ethyl dialkyl- [25, 26] or diarylphosphinoites [25, 27] with salicyl alcohol. However, the synthesis of phosphine oxide **2a** is described in the patent [25], where no its physicochemical and spectral characteristics are given.

The lower yields of phosphine oxides **2a**–**2d** in the reactions of benzophosphole **1** with organomagnesium



Downfield regions of the ^{1}H and ^{1}H–{^{31}P} NMR spectra (400 MHz, $CDCl_3$) of phosphine oxide **2a**.

compounds compared with those in the published syntheses of dialkyl(diaryl)(2-methyl-4-oxopent-2-yl)-phosphine oxides [20] or (*Z*)-4-[dialkyl(diphenyl)-phosphoryl]-2-methylbut-3-en-1-ols [21], are, probably, explained by a lower reactivity of the exocyclic P–O bond in compound **1** in these reactions.

The structure of the synthesized (hydroxybenzyl)-phosphine oxides was confirmed by IR and ^{31}P, ^{1}H, and ^{13}C NMR spectroscopy. The benzyl methylene proton signals in the ^{1}H NMR spectra of phosphine oxides **2a**–**2d** appear as a characteristic doublet at δ = 3.15 ppm, $^2J_{PH}$ = 11.9 Hz (**2d**, δ = 3.84 ppm, $^2J_{PH}$ = 13.7 Hz). The phenyl proton signals were assigned as we further exemplify by the assignments for compound **2a.** The figure shows the aromatic parts of the ^{1}H and ^{1}H–{^{31}P} NMR spectra (400 MHz, $CDCl_3$) of this compound. The hydroxybenzyl H^6 proton in the ^{1}H–{^{31}P} NMR spectrum resonates in the farthest downfield region of the aromatic part of the spectrum as a doublet of doublet of doublets at 6.85 ppm, which in the ^{1}H NMR spectrum appears as a doublet of doublet of doublet of doublets ($^3J_{HH}$ = 7.5, $^3J_{HH}$ = 7.4, $^4J_{HH}$ = 1.3, $^5J_{PH}$ = 1.1 Hz). In the ^{1}H–{^{31}P} spectrum the H^7 proton appears as doublet of doublets at 7.0 ppm; in the ^{1}H spectrum this signal transforms into a doublet of doublet of doublets ($^3J_{HH}$ = 7.4, $^4J_{HH}$ = 1.5, $^4J_{PH}$ = 1.7 Hz). The H^4 appears as a doublet of doublets at ~7 ppm ($^3J_{HH}$ = 8.0, $^4J_{HH}$ = 1.3 Hz). The H^5 appears as a doublet of doublet of doublets at 7.2 ppm in the ^{1}H–{^{31}P} spectrum and as a doublet of doublet of doublet of doublets ($^3J_{HH}$ = 8.0, $^3J_{HH}$ = 7.5, $^4J_{HH}$ = 1.5, $^6J_{PH}$ = 1.7 Hz) in the ^{1}H NMR spectrum.

The signals which are the easiest to identify in the ^{13}C and ^{13}C–{^{1}H} NMR spectra of phosphine oxides **2a**–**2d** belong to carbons spin-coupled to phosphorus. Thus, the alkyl carbon atoms directly attached to phosphorus appear as a doublet ($^1J_{PC}$ = 64–66 Hz) at 19–29 ppm in the ^{13}C–{^{1}H} NMR spectra. The PC^1 carbon signal is observed at 32–33 ppm ($^1J_{PC}$ ~ 61 Hz). The *ipso*-carbon atoms C^2 and C^3 give weak signals at 119.9 and 156.5 ppm, respectively. The C^4 and C^6 signals appear very close to each other, and they are difficult to differentiate by the chemical shift and multiplicity (~119.5 and ~120 ppm). The C^5 carbon appears as a a doublet of doublet of doublets near 129 ppm ($^1J_{CH}$ = 160–161, $^3J_{HC}$ ~ 8.5, $^5J_{PC}$ = 1.8 Hz), and the C^7 carbon signal is observed near 130 ppm as a doublet of multiplets ($^1J_{HC}$ = 156–157, $^3J_{PC}$ = 5–6 Hz).

The most characteristic band in the IR spectra of compounds **2a**–**2d** is a strong and broad OH stretching absorption band above 3000 cm^{-1}.

Thus, we developed a facile method of synthesis of dialkyl(diaryl)(2-hydroxybenzyl)phosphine oxides by the reaction of 2-ethoxy-2,3-dihydro[*d*][1,2]oxaphos-phole 2-oxide with organomagnesium compounds, which allows one to widely vary substituents on the phosphorus atom.

EXPERIMENTAL

The ^{1}H, ^{13}C, ^{13}C–{^{1}H}, and ^{31}P–{^{1}H} NMR spectra were measured on a Bruker Avance-400 spectrometer [400 (^{1}H), 160.9 (^{31}P), and 100.6 MHz (^{13}C)] against solvent signals. The IR spectra were obtained on a Bruker Vector-22 instrument for suspensions in mineral oil, thin films between KBr plates, or KBr pellets. The melting points were measured using a Boetius hot stage.

2-Ethoxy-2,3-dihydro[*d*][1,2]oxaphosphole 2-oxide (1). A mixture of 20.0 g (0.1611 mol) *o*-hydroxybenzyl

alcohol and 26.8 g (0.161 mol) of triethyl phosphite in 40 mL of toluene was heated under reflux for 12 h. The solvent was removed, and the residue was heated in a vacuum (80°C, 20 mmHg) and then distilled in a vacuum of 0.03 mmHg at 113–116°C. Yield 24.3 g (76%). $^{31}P–\{^1H\}$ NMR spectrum: δ_P 46.0 ppm. The physicochemical characteristics of the product are consistent with those reported in [24, 28].

Synthesis of dialkyl(diaryl)(2-hydroxybenzyl) phosphine oxides (*general procedure*). Benzophosphole **1,** 5 g (0.025 mol), was added dropwise to a Grignard reagent prepared by standard procedure from 0.063 mol of alkyl(aryl) bromide and 1.5 g (0.063 mol) of Mg in 30 mL of THF. The mixture was refluxed with stirring under argon for 2 h, let to cool to room temperature, and hydrolyzed with a solution of 0.063 mol of H_2SO_4 in 30 mL of water. The aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL), the solvent was removed, and the residue was washed with diethyl ether.

Diethyl(2-hydroxybenzyl)phosphine oxide (2a). Yield 54%, mp 114–116°C. IR spectrum (KBr), ν, cm^{-1}: 438, 450, 467, 498, 537, 627, 693, 732, 747, 763, 801, 832, 862, 942, 974, 996, 1038, 1085, 1108, 1158, 1176, 1248, 1280, 1308, 1377, 1457, 1506, 1594, 2595, 2707, 2921, 3461. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.16 d.t (6H, $PCH_2\underline{CH_3}$, $^3J_{PH}$ = 16.7, $^3J_{HH}$ = 7.7), 1.73–1.86 m (4H, PCH_2, AB part of the ABX system), 3.15 d (2H, H^1, $^2J_{PH}$ = 12.3), 6.85 d.d.d.d (1H, H^6, $^3J_{HH}$ = 7.5, $^3J_{HH}$ = 7.4, $^4J_{HH}$ = 1.3, $^5J_{PH}$ = 1.3), 7.00 d.d.d (1H, H^7, $^3J_{HH}$ = 7.5, $^4J_{HH}$ = 1.8, $^4J_{PH}$ = 1.7), 7.03 d.d (1H, H^4, $^3J_{HH}$ = 8.0, $^4J_{HH}$ = 1.3), 7.20 d.d.d.d (1H, H^5, $^3J_{HH}$ = 8.0, $^3J_{HH}$ = 7.4, $^4J_{HH}$ = 1.8, $^6J_{PH}$ = 1.7), 9.74 s (1H, OH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 5.77 qd.t (d) ($PCH_2\underline{CH_3}$, $^1J_{HC}$ = 129.1, $^2J_{HC}$ = 4.77, $^2J_{PC}$ = 4.8), 19.76 br.t.d.q (d) ($P\underline{CH_2}CH_3$, $^1J_{HC}$ = 126.9, $^1J_{PC}$ = 65.7, $^2J_{HC}$ = 3.6), 32.07 br.t.d.d (d) (C^1, $^1J_{HC}$ = 127.1, $^1J_{PC}$ = 60.9, $^3J_{HC}$ = 3.1), 119.47 d.d (d) [$C^{4(6)}$, $^1J_{HC}$ = 160.4, $^3J_{HC}$ = 8.0, $^4J_{PC}$ 2.4], 119.81 m (d) (C^2, $^2J_{PC}$ = 8.1), 120.70 d.d (d) [$C^{6(4)}$, $^1J_{HC}$ = 161.7, $^3J_{HC}$ = 8.1, $^4J_{PC}$ 1.9], 129.06 d.d.d (d) (C^5, $^1J_{HC}$ = 160.3, $^3J_{HC}$ = 8.6, $^5J_{PC}$ = 1.8), 131.15 d.m (d) (C^7, $^1J_{HC}$ = 156.6, $^3J_{PC}$ = 5.9), 156.49 m (d) (C^3, $^3J_{PC}$ = 3.7). ^{31}P NMR spectrum ($CDCl_3$): δ_P 55.3 ppm. Found, %: C 62.29; H 8.22; P 14.77. $C_{11}H_{17}O_2P$. Calculated, %: C 62.26; H 8.02; P 14.62.

(2-Hydroxybenzyl)dipropylphosphine oxide (2b). Yield 63%, mp 72°C. IR spectrum (KBr), ν, cm^{-1}: 449, 467, 494, 535, 554, 601, 619, 730, 755, 793, 842, 869, 906, 939, 970, 1040, 1082, 1128, 1251, 1275, 1308, 1347, 1381, 1401, 1457, 1487, 1506, 1598, 2624, 2736, 2874, 2933, 2963, 3067. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.05 t.d (6H, $PCH_2CH_2\underline{CH_3}$, $^3J_{HH}$ = 7.2, $^4J_{PH}$ = 1.0), 1.55–1.70 m (4H, $PCH_2\underline{CH_2}$, AB part of the ABX system), 1.7–1.8 m (4H, PCH_2, AB part of the ABX system), 3.15 d (2H, H^1, $^2J_{PH}$ = 12.0), 6.86 br.d.d (1H, H^6, $^3J_{HH}$ = 7.6, $^3J_{HH}$ = 7.1), 6.97 d.d.d (1H, H^7, $^3J_{HH}$ = 7.6, $^4J_{HH}$ = 1.8, $^4J_{PH}$ = 1.5), 7.02 d.d (1H, H^4, $^3J_{HH}$ = 8.1, $^4J_{HH}$ = 1.3), 7.20 d.d.d.d (1H, H^5, $^3J_{HH}$ = 7.9, $^3J_{HH}$ = 7.4, $^4J_{HH}$ ~ 1.8, $^6J_{PH}$ = ~ 1.6). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm (*J*, Hz): 15.54 t.m (d) ($PCH_2\underline{CH_2}$, $^1J_{HC}$ = 129.7, $^2J_{PC}$ = 3.9), 15.86 br.q.d.t (d) ($PCH_2CH_2\underline{CH_3}$, $^1J_{HC}$ = 126.2, $^3J_{PC}$ = 14.7, $^2J_{HC}$ = 3.7), 29.55 br t.d (d) (PCH_2, $^1J_{HC}$ = 127.66, $^1J_{PC}$ = 64.3), 33.21 t.d.d (d) (C^1, $^1J_{HC}$ = 127.9, $^1J_{PC}$ = 61.0, $^3J_{HC}$ = 4.6), 119.64 br.d.d (d) [$C^{4(6)}$, $^1J_{HC}$ = 161.4, $^4J_{PC}$ = 2.3], 119.93 m (d) (C^2, $^2J_{PC}$ = 8.3), 120.77 br.d.d (d) [$C^{6(4)}$, $^1J_{HC}$ = 161.3, $^3J_{HC}$ = 8.2, $^4J_{PC}$ = 1.7], 129.14 br.d.d.m (d) (C^5, $^1J_{HC}$ = 161.2, $^3J_{HC}$ = 8.5, $^5J_{PC}$ = 2.5), 131.15 d.m (d) (C^7, $^1J_{HC}$ = 157.2, $^3J_{PC}$ = 6.0), 156.5 m (d) (C^3, $^3J_{PC}$ = 3.8). ^{31}P NMR spectrum ($CDCl_3$): δ_P 54.8 ppm. Found, %: C 65.17; H 8.39; P 12.56. $C_{13}H_{21}O_2P$. Calculated, %: C 64.98; H 8.81; P 12.89.

Dibutyl(2-hydroxybenzyl)phosphine oxide (2c). Yield 53 %. IR spectrum, ν, cm^{-1}: 453, 496, 535, 553, 598, 618, 721, 754, 807, 832, 866, 902, 938, 969, 1043, 1094, 1129, 1175, 1231, 1251, 1275, 1308, 1381, 1402, 1458, 1485, 1505, 1598, 2624, 2736, 2871, 2932, 2958, 3068. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 0.92 t [6H, $P(CH_2)_3\underline{CH_3}$, $^3J_{HH}$ = 7.3], 1.37–1.46 m [4H, $P(CH_2)_2\underline{CH_2}$], 1.50–1.61 m (4H, $PCH_2\underline{CH_2}$), 1.72–1.79 m (4H, PCH_2), 3.15 d (2H, H^1, $^3J_{PH}$ = 11.9), 6.86 br.d.d (1H, H^6, $^3J_{HH}$ = 7.4, $^3J_{HH}$ = 7.3), 6.97 br.d (1H, H^7, $^3J_{HH}$ = 7.5), 7.02 d.d (1H, H^4, $^3J_{HH}$ = 8.0, $^4J_{HH}$ = 1.2), 7.19 d.d.d.d (1H, H^5, $^3J_{HH}$ = 7.9, $^3J_{HH}$ = 7.5, $^4J_{HH}$ = 1.7, $^6J_{PH}$ = 1.6). ^{31}P NMR spectrum ($CDCl_3$): δ_P 56.7 ppm. Found, %: C 66.74; H 9.52; P 11.64. $C_{15}H_{25}O_2P$. Calculated, %: C 67.14; H 9.39; P 11.54.

(2-Hydroxybenzyl)diphenylphosphine oxide (2d). Yield 53%, mp 153–154°C. IR spectrum (KBr), *ν*, cm^{-1}: 504, 559, 604, 688, 721, 742, 785, 825, 868, 938, 968, 995, 1029, 1067, 1119, 1150, 1243, 1377, 1463, 1576, 2929, 3448. ^1H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 3.84 d (2H, H^1, $^2J_{PH}$ = 13.7), 6.63 br.d.d (1H, H^6, $^3J_{HH}$ = 7.4, $^3J_{HH}$ = 7.4), 6.73 br.d (1H, H^4, $^3J_{HH}$ = 8.0), 6.98 d.d.d.d (1H, H^5, $^3J_{HH}$ = 7.7, $^3J_{HH}$ = 7.6, $^4J_{HH}$ = 1.7, $^6J_{PH}$ = 1.6), 7.09 br.d (1H, H^7, $^3J_{HH}$ = 7.7), 7.46–7.52 m (4H, H^m), 7.53–7.57 m (2H, H^p), 7.79 m (4H, H^o, $^3J_{PH}$ = 11.4), 9.66 s (OH, 1H). ^{31}P NMR spectrum (DMSO-

d_6): δ_P 39.0 ppm. Found, %: C 74.11; H 5.88; P 9.77. $C_{19}H_{17}O_2P$. Calculated, %: C 74.03; H 5.52; P 10.06.

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