ISSN 1070-3632, Russian Journal of General Chemistry, 2018, Vol. 88, No. 9, pp. 1776–1779. © Pleiades Publishing, Ltd., 2018.
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Dedicated to the 110th anniversary of M.I. Kabachnik's birth

Electrophilic Catalysis in the Synthesis of Aryl Methyl- and Phenylphosphonochloridates

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Received June 29, 2018

Abstract—The reaction of methyl- and phenylphosphonic dichlorides with phenols in the presence of anhydrous magnesium chloride as catalyst or magnesium metal as precatalyst provides a simple, efficient, and practical method of synthesis of the corresponding aryl methyl- and phenylphosphonochloridates.

Keywords: methylphosphonic dichloride, phenylphosphonic dichloride, phosphorylating agent, phenols, phosphorylation, aryl methyl(phenyl)phosphonochloridates

DOI: 10.1134/S1070363218090025

Aryl phenyl- and methylphosphonochloridates are widely used as starting compounds in the synthesis of various practically important organophosphorus compounds, in particular extractants for *f*-elements [1, 2], fire-retardant additives for polymeric materials [3, 4], and especially biologically active substances [5–11]. However, none of the currently known methods for the preparation of aryl phenyl- and methylphosphonochloridates is free from some disadvantages. For instance, the most frequently used synthesis based on the reaction of the corresponding organylphosphonic dichlorides $RP(O)Cl_2$ [**1**, R = Ph (**a**), Me (**b**)] with phenols in the presence of a tertiary amine as hydrogen chloride acceptor requires large amounts of organic solvents, and the isolated products are not always as pure as necessary [5, 10, 12]. Although aryl methylphosphonochloridates free from tertiary amine hydrochloride impurity can be obtained by analogous reaction in the absence of tertiary amine, the process requires prolonged heating at elevated temperature, which reduces the yield of the target products [13].

A way to improve the efficiency of the above reaction could be electrophilic catalysis which was successfully used previously in the synthesis of various polyfluoroalkyl phosphonochloridates at the Organophosphorus Compounds Laboratory (Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences) under the guidance of Academician M.I. Kabachnik [14]. In fact, it was found that phosphorylation of phenol with the simplest alkyl(or aryl)phosphonic dichlorides (**1a**, **1b**) can be catalyzed by a number of metal salts possessing Lewis acid properties (e.g., anhydrous lithium, magnesium, and calcium chlorides; magnesium dichloride turned out to be the most effective), as well as by magnesium metal[1] acting as precatalyst. In this case, neither hydrogen chloride acceptor nor organic solvent is required. Catalytic reactions are characterized by a high rate even at moderate temperature provided that the phosphorylating agent is taken in excess (the optimal amounts are 2 mol of **1a** and 3 mol of **1b**), and the target phosphonochloridates RP(O)(Cl)OPh (**2a**, **2b**) are obtained in fairly high yields (66–91%; Scheme 1) with high purity.

The catalytic reactions are easily scalable, and excess phosphorylating agent can be recovered from the reaction mixture with high yield and purity, so that it can be recycled without additional purification. Thus, the catalytic processes are very attractive from the practical viewpoint.

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[1] Magnesium metal dissolves during the reaction to form magnesium chloride.

Scheme 1.

$$\underset{1}{\mathrm{PhOH}} + \underset{2\text{–}3}{\mathrm{RP(O)Cl_2}} \xrightarrow{120°\mathrm{C},\ [\mathrm{MgCl_2/Mg}]} \mathrm{RP(O)(OPh)Cl},$$

$1 : 2\text{–}3$

1a, 1b **2a, 2b**

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

Scheme 2.

$$\mathrm{ArOH} + \mathrm{MeP(O)Cl_2} \xrightarrow{120°\mathrm{C},\ [\mathrm{Mg}]} \mathrm{MeP(O)(OAr)Cl},$$

$1 : 3$

3a–3i **1b** **4a–4i**

Ar = 4-FC_6H_4 (**a**), 4-ClC_6H_4 (**b**), 4-EtC_6H_4 (**c**), 4-*iso*-PrC_6H_4 (**d**), 4-*tert*-BuC_6H_4 (**e**), 2-$MeOC_6H_4$ (**f**), 4-$MeOC_6H_4$ (**g**), 2,6-$Me_2C_6H_3$ (**h**), 2-Cl-5-MeC_6H_3 (**i**).

The next stage of our study was aimed at estimating the scope of application of electrophilic catalysis in the synthesis of aryl organylphosphonochloridates.

It should be noted that the use of phenyl phenylphosphonochloridate (**2a**) for the preparation of physiologically active compounds (the major field of application of organylphosphonochloridates) has been reported [11]; however, phenyl methylphosphonochloridate (**2b**) turned out to be the most appropriate for this purpose [5–10]. Therefore, we examined phosphorylation of a series of commercially available mono- and disubstituted phenols **3a**–**3i** with 3 mol of of methylphosphonic dichloride (**1b**) on heating in the presence of magnesium metal[2] as precatalyst (Scheme 2). Fractional distillation of the reaction mixtures under reduced pressure afforded analytically and spectrally pure aryl methylphosphonochloridates **4a**–**4i**[3] in fairly high yields (61–89%). As in the catalytic synthesis of methylphosphonochloridate **2b**, excess phosphorylating agent can readily be regenerated, and it is sufficiently pure to be recycled.

The structure of phosphoryl chlorides **4a**–**4i** was confirmed by ^{1}H, ^{1}H{^{31}P}, ^{13}C{^{1}H}, ^{19}F{^{1}H}, and ^{31}P{^{1}H} NMR spectra.[4] In particular, all compounds **4a**–**4i** characteristically showed in the ^{1}H and ^{13}C{^{1}H} NMR spectra doublet signals in the regions δ 1.48–1.64[5] and δ_C 19.6–20.4 ppm, respectively, due to methyl group linked to the phosphorus atom. The phosphorus nucleus resonated at δ_P 33–36 ppm in the ^{31}P–{^{1}H} NMR spectra, which is consistent with the presumed structure. As expected, the phosphorus signal of **4b**–**4i** was a singlet, whereas fluorine-containing compound **4a** displayed a doublet signal of the phosphorus atom due to long-range coupling with fluorine.

Thus, our results demonstrated that electrophilic catalysis provides a general, simple, efficient, and practical synthetic approach to various aryl methylphosphonochloridates, which makes these practically important organophosphorus reagents readily accessible. Furthermore, the proposed method can be successfully applied to the synthesis of both other representatives of the same series and structurally related aryl alkyl(aryl) phosphonochloridates.

EXPERIMENTAL

The ^{1}H, ^{1}H–{^{31}P}, ^{13}C, ^{19}F, and ^{31}P NMR spectra were recorded on a Bruker AV-400 spectrometer at [400.13 (^{1}H), 100.61 (^{13}C), 376.49 (^{19}F), and 161.98 MHz (^{31}P)] either without a solvent (compounds **2**) or in C_6D_6 (**4**); the ^{1}H and ^{13}C chemical shifts were measured relative to the residual proton and carbon signals of the deuterated solvent; the ^{19}F and ^{31}P chemical shifts were determined relative to $CFCl_3$ and 85% H_3PO_4, respectively (external standards). The elemental analyses were obtained at the Microanalysis Laboratory (Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences).

Phenylphosphonic dichloride (**1a**, 97%, Acros) and methylphosphonic dichloride (**1b**, 98%, Aldrich) were distilled under reduced pressure prior to use. Phenol and its mono- and disubstituted derivatives **3a**–**3i** (97–99%, Acros), anhydrous magnesium chloride (99.9%, Acros), and magnesium metal (98%, granules, 20–230 mesh, Aldrich) were used without additional purification.

Phenyl phenylphosphonochloridate (2a). Phenylphosphonic dichloride (**1a**), 40 g (0.205 mol), was

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[2] This procedure provides the best combination of high catalytic efficiency and convenience in operation, so that the given version of catalytic synthesis of aryl organylphosphonochloridates seems to be preferable to the use of anhydrous magnesium chloride.

[3] Chlorides **4a** and **4b** synthesized by classical methods were high-boiling liquids; both compounds synthesized by catalytic phosphorylation were crystalline solids.

[4] Signals in the ^{1}H and ^{13}C NMR spectra were assigned using COSY, HMQC, and HMBC shift correlation techniques.

[5] These multiplets were converted to singlets in the ^{1}H–{^{31}P} NMR spectra.

added under argon to 9.7 g (0.102 mol) of phenol, and 0.243 g (0.00256 mol) of finely powdered anhydrous magnesium chloride was then added. The mixture was heated for 1.5 h at 120°C until hydrogen chloride no longer evolved, kept for 0.5 h under reduced pressure (~15 Torr) at room temperature, and subjected to fractional distillation under reduced pressure. Yield 17.2 g (66%), bp 154–155°C (0.5 Torr) {bp 152–155°C (0.3 Torr) [15]}. ^{31}P NMR spectrum: δ_P 24.1 ppm.

Phenyl methylphosphonochloridate (2b) was synthesized in a similar way from 47 g (0.5 mol) of phenol and 200 g (1.5 mol) of molten methylphosphonic dichloride (**1b**) in the presence of 300 mg (0.0125 mol) of magnesium metal; reaction time 3.0 h. Yield 86.3 g (91%), bp 107°C (1 Torr) {bp 80°C (0.5 Torr) [16]}. ^{31}P NMR spectrum: δ_P 36.3 ppm.

Aryl methylphosphonochloridates 4a–4i were synthesized according to analogous procedure from phosphonic dichloride **1b** and mono- and disubstituted phenols **3a–3i** in the presence of magnesium metal as precatalyst.

4-Fluorophenyl methylphosphonochloridate (4a). Yield 89%, bp 91–92°C (1 Torr), mp 35–37°C {bp 89–91°C (1 Torr) [5]}. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.52 d (3H, CH_3, $^2J_{HP}$ = 17.3), 6.68–6.77 m (2H, 3-H, 5-H), 7.10–7.17 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.6 d (CH_3, $^1J_{CP}$ = 128.4), 116.4 d.d (C^3, C^5, $^2J_{CF}$ = 23.5, $^4J_{CP}$ = 1.5), 122.3 d.d (C^2, C^6, $^3J_{CF}$ = 8.1, $^3J_{CP}$ = 5.1), 145.6 d.d (C^1, $^4J_{CF}$ = 2.9, $^2J_{CP}$ = 11.0), 160.3 d.d (C^4, $^1J_{CF}$ = 244.3, $^5J_{CP}$ = 1.5). ^{19}F NMR spectrum: δ_F –116.4 ppm, d ($^6J_{FP}$ = 2.5 Hz). ^{31}P NMR spectrum: δ_P 35.5 ppm, d ($^6J_{PF}$ = 2.5 Hz).

4-Chlorophenyl methylphosphonochloridate (4b). Yield 66%, bp 129–130°C (1 Torr), mp 36–38°C {bp 119–121°C (0.14 Torr) [17]}. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.48 d (3H, CH_3, $^2J_{HP}$ = 17.3), 6.98–7.03 m (2H, 3-H, 5-H), 7.04–7.10 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.6 d (CH_3, $^1J_{CP}$ = 128.4), 122.1 d (C^2, C^6, $^3J_{CP}$ = 5.1), 129.9 d (C^3, C^5, $^4J_{CP}$ = 1.5), 131.2 d (C^4, $^5J_{CP}$ = 2.2), 148.2 d (C^1, $^2J_{CP}$ = 11.0). ^{31}P NMR spectrum: δ_P 35.5 ppm.

4-Ethylphenyl methylphosphonochloridate (4c). Yield 77%, bp 99–101°C (1 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.08 t (3H, $C\mathbf{H}_3CH_2$, $^3J_{HH}$ = 7.6), 1.59 d (3H, CH_3P, $^2J_{HP}$ = 17.4), 2.41 q (2H, CH_2, $^3J_{HH}$ = 7.5), 6.94–7.01 m (2H, 3-H, 5-H), 7.30–7.36 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 15.4 ($\mathbf{C}H_3CH_2$), 19.8 d (CH_3P, $^1J_{CP}$ = 129.1), 28.1 (CH_2), 120.6 d (C^2, C^6, $^3J_{CP}$ = 5.1), 129.2 d (C^3, C^5, $^4J_{CP}$ = 1.5), 141.7 d (C^4, $^5J_{CP}$ = 1.5), 148.0 d (C^1, $^2J_{CP}$ = 11.0). ^{31}P NMR spectrum: δ_P 35.4 ppm. Found, %: C 49.43; H 5.49; Cl 16.16; P 14.22. $C_9H_{12}ClO_2P$. Calculated, %: C 49.45; H 5.53; Cl 16.22; P 14.17.

4-Isopropylphenyl methylphosphonochloridate (4d). Yield 66%, bp 101–102°C (0.5 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.13 d (6H, $C\mathbf{H}_3CH$, $^3J_{HH}$ = 6.8), 1.64 d (3H, CH_3P, $^2J_{HP}$ = 17.2), 2.70 sept (1H, CH, $^3J_{HH}$ = 6.9), 7.02–7.08 m (2H, 3-H, 5-H), 7.32–7.38 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.8 d (CH_3P, $^1J_{CP}$ = 129.1), 23.8 ($\mathbf{C}H_3CH$), 33.5 (CH), 120.6 d (C^2, C^6, $^3J_{CP}$ = 5.1), 127.8 d (C^3, C^5, $^4J_{CP}$ = 1.5), 146.3 d (C^4, $^5J_{CP}$ = 1.5), 148.0 d (C^1, $^2J_{CP}$ = 11.0). ^{31}P NMR spectrum: δ_P 35.3 ppm. Found, %: C 51.68; H 6.16; Cl 15.22; P 13.32. $C_{10}H_{14}ClO_2P$. Calculated, %: C 51.63; H 6.06; Cl 15.24; P 13.31.

4-*tert*-Butylphenyl methylphosphonochloridate (4e). Yield 70%, bp 130–131°C (1 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.22 s (9H, CH_3C), 1.56 d (3H, CH_3P, $^2J_{HP}$ = 17.3), 7.20–7.25 m (2H, 3-H, 5-H), 7.36–7.41 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.8 d (CH_3P, $^1J_{CP}$ = 129.1), 31.1 ($\mathbf{C}H_3C$), 34.1 ($CH_3\mathbf{C}$), 120.3 d (C^2, C^6, $^3J_{CP}$ = 5.1), 126.8 (C^3, C^5), 147.8 d (C^1, $^2J_{CP}$ = 11.0), 148.5 d (C^4, $^5J_{CP}$ = 1.5). ^{31}P NMR spectrum: δ_P 35.0 ppm. Found, %: C 53.62; H 6.50; Cl 14.24; P 12.57. $C_{11}H_{16}ClO_2P$. Calculated, %: C 53.56; H 6.54; Cl 14.37; P 12.56.

2-Methoxyphenyl methylphosphonochloridate (4f). Yield 70%, bp 121–122°C (2 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.65 d (3H, CH_3P, $^2J_{HP}$ = 17.3), 3.38 s (3H, CH_3O), 6.60 d.d.d (1H, 3-H, $^3J_{HH}$ = 8.2, $^4J_{HH}$ ≈ $^5J_{HP}$ = 1.1), 6.75 t.d (1H, 5-H, $^3J_{HH}$ = 7.8, $^4J_{HH}$ = 1.5), 6.96 t.d.d (1H, 4-H, $^3J_{HH}$ = 7.8, $^4J_{HH}$ ≈ $^6J_{HP}$ = 1.5), 7.56 d.d.d (1H, 6-H, $^3J_{HH}$ = 8.0, $^4J_{HH}$ ≈ $^4J_{HP}$ = 1.8). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 20.1 d (CH_3P, $^1J_{CP}$ = 127.6), 55.3 (CH_3O), 113.0 d (C^3, $^4J_{CP}$ = 1.0), 120.8 d (C^5, $^4J_{CP}$ = 2.0), 122.3 d (C^6, $^3J_{CP}$ = 3.9), 126.5 d (C^4, $^5J_{CP}$ = 2.0), 139.2 d (C^1, $^2J_{CP}$ = 11.2), 151.0 d (C^2, $^3J_{CP}$ = 4.9). ^{31}P NMR spectrum: δ_P 36.3 ppm. Found, %: C 43.76; H 4.47; P 14.31. $C_8H_{10}ClO_3P$. Calculated, %: C 43.56; H 4.57; P 14.04.

4-Methoxyphenyl methylphosphonochloridate (4g). Yield 79%, bp 131–132°C (1 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.62 d (3H, CH_3P, $^2J_{HP}$ = 17.2), 3.35 s (3H, CH_3O), 6.68–6.76 m (2H, 3-H, 5-H), 7.25–7.32 m (2H, 2-H, 6-H). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.6 d (CH_3P, $^1J_{CP}$ = 128.4), 54.9 (CH_3O), 114.9 d (C^3, C^5, $^4J_{CP}$ = 1.5), 121.8 d (C^2, C^6, $^3J_{CP}$ =

5.1), 143.4 d (C^1, $^2J_{CP} = 11.0$), 157.6 d (C^4, $^5J_{CP} = 1.5$). ^{31}P NMR spectrum: δ_P 33.8 ppm. Found, %: C 43.59; H 4.70; Cl 16.23; P 14.04. $C_8H_{10}ClO_3P$. Calculated, %: C 43.56; H 4.57; Cl 16.07; P 14.04.

2,6-Dimethylphenyl methylphosphonochloridate (4h). Yield 61%, bp 126–127°C (1 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.59 d (3H, CH_3P, $^2J_{HP}$ = 17.1), 2.37 d (6H, 2-CH_3, 6-CH_3, $^5J_{HP} = 1.0$), 6.92 br.s (3H, C_6H_3). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 17.7 d (2-CH_3, 6-CH_3, $^4J_{CP} = 0.7$), 20.4 d (CH_3P, $^1J_{CP}$ = 129.1), 125.7 d (C^4, $^5J_{CP} = 2.2$), 129.3 d (C^3, C^5, $^4J_{CP}$ = 2.2), 130.2 d (C^2, C^6, $^3J_{CP} = 3.7$), 148.5 d (C^1, $^2J_{CP}$ = 12.5). ^{31}P NMR spectrum: δ_P 34.4 ppm. Found, %: C 49.48; H 5.57; Cl 15.98; P 14.19. $C_9H_{12}ClO_2P$. Calculated, %: C 49.45; H 5.53; Cl 16.22; P 14.17.

2-Chloro-5-methylphenyl methylphosphonochloridate (4i). Yield 72%, bp 115–116°C (0.1 Torr). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.48 d (3H, CH_3P, $^2J_{HP} = 17.2$), 2.06 s (3H, 5-CH_3), 7.00 d.d.d (1H, 4-H, $^3J_{HH} = 8.7$, $^4J_{HH} = 2.8$, $^6J_{HP} = 1.8$), 7.08 d (3-H, $^3J_{HH}$ = 8.6), 7.16 d.d (1H, 6-H, $^4J_{HH} = 2.6$, $^4J_{HP} = 1.9$). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 19.7 (5-CH_3), 19.8 d (CH_3P, $^1J_{CP} = 128.1$), 119.6 d (C^4, $^5J_{CP} = 5.2$), 123.2 d (C^6, $^3J_{CP} = 5.2$), 130.1 d (C^3, $^4J_{CP} = 1.4$), 131.4 d (C^5, $^4J_{CP} = 2.1$), 138.0 d (C^2, $^3J_{CP} = 1.4$), 148.2 d (C^1, $^2J_{CP}$ = 10.8). ^{31}P NMR spectrum: δ_P 35.8 ppm. Found, %: C 40.61; H 3.98; Cl 29.95; P 13.53. $C_8H_9Cl_2O_2P$. Calculated, %: C 40.20; H 3.80; Cl 29.66; P 12.96.

ACKNOWLEDGMENTS

The authors thank Molecular Structure Research Center (Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences) for performing spectral studies.

CONFLICT OF INTERESTS

No conflict of interests was declared by the authors.

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