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Free Radical Hydrophosphorylation of Fluoroalkyl Vinyl Ethers: Synthesis of Fluoroalkyl Phosphonates

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Abstract—An effective method for the synthesis of dialkyl [2-(polyfluoroalkoxy)ethyl]phosphonates by free radical hydrophosphorylation of fluoroalkyl vinyl ethers with dialkyl (*H*)-phosphonates was developed. The reaction proceeds in the presence of catalytic amounts of azabisisobutyric acid dinitrile (AIBN) (150°C, 2 h, portionwise addition of AIBN) to afford the target fluoroalkyl phosphonates in up to 85% isolated yield.

Keywords: dialkyl (*H*)-phosphonates, fluoroalkyl vinyl ethers, radical addition, atom-economic synthesis, fluoroalkyl phosphonates

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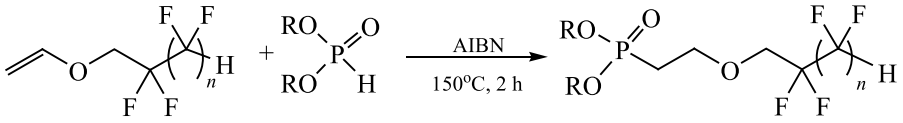
Fluorine-containing organophosphorus compounds have a wide range of useful properties and are used in various fields of industry, agriculture, and medicine. Among them, effective additives to the electrolytes of lithium-ion batteries were found to increase their fire and explosion safety [1–9]. A great deal of attention is also paid to fluoroalkyl phosphonates [10, 11], among which compounds have already been identified that are active against various viral and retroviral DNA infections, such as hepatitis B and HIV [12–15], malignant tumors [16], as well as anti-inflammatory effect [17]. Therefore, an increasing interest in the development of the chemistry of fluoroalkyl phosphonates and the development of convenient approaches to their preparation is not accidental.

Herein, we first reported the radical addition reaction of dialkyl (*H*)-phosphonates to vinyl ethers of polyfluoroalkanols resulting in new representatives of fluoroalkyl phosphonates.

Initial experiments showed that 2,2,3,3-tetrafluoropropan-1-ol vinyl ether **1a** does not react with (*H*)-phosphonate **2a** under conditions typical for the phosphorylation of alkyl vinyl ethers [18–21] (molar ratio **1a** : **2a** = 1 : 4, 1.5 wt % AIBN, 80°C, 8 h): only the starting compounds were isolated from the reaction mixture. Varying the initiator concentration (up to 3 wt %) and the process duration (20 h) did not lead to a positive result. Raising the reaction temperature to 120°C with a single injection of AIBN also proved to be ineffective, probably due to the rapid thermal destruction of the initiator.

At the same time, we succeeded in carrying out the process at 150°C with the portionwise addition of AIBN and vinyl ether **1a** to phosphonate **2a**. The developed technique involves the slow (1.5 h) dropwise addition of a solution of 1.5 wt % AIBN (a mixture of 1 equiv. of ether **1a** and 2 equiv. of phosphonate **2a** was used as a solvent) to phosphonate **2a** (2 equiv.) heated to 150°C. This approach provided a large excess of phosphorylating reagent **2a** with respect to substrate **1a**, which made it possible to suppress the telomerization of vinyl ether **1a** and to obtain the target dimethyl 2-(2,2,3,3-tetrafluoropropoxy)-ethylphosphonate **3a** in 85% yield. The reaction is of a general nature: the target polyfluoroalkyl phosphonates **3b**–**3d** were synthesized with a yield of 83, 66 and 67%, respectively (Scheme 1). These data indicate that the selectivity of the reaction decreases with increasing length of the fluoroalkyl substituent in ether **1** (Scheme 1).

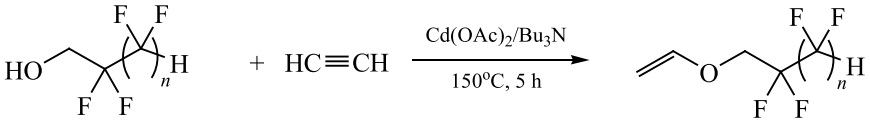
Scheme 1.



1a, **1b** **2a**, **2b** **3a**–**3d**

n = 1, R = Me (**3a**, 85%); *n* = 1, R = Et (**3b**, 83%); *n* = 3, R = Me (**3c**, 66%); *n* = 3, R = Et (**3d**, 67%).

Scheme 2.



1a, **1b**

n = 1 (**1a**); *n* = 3 (**1b**).

It should be noted that the excess of (*H*)-phosphonate **2** returns practically quantitatively when the reaction mixture is fractionated.

Lowering the reaction temperature to 120°C leads to a noticeable decrease (~ 1.5 times) in the yield of target polyfluoroalkyl phosphonates **3**, all other factors being the same.

The experimental conditions found are: a temperature of 150°C (contributing to the breaking of the P–H bond, the dissociation energy of which is 365 kJ/mol [22]), a portionwise addition of AIBN into the reaction mixture (allowing its initiating activity to be preserved), a portionwise addition of substrate **1**; the use of excess phosphorylating reagent **2** reduces a possibility of telomerization of vinyl ether **1** and increases the efficiency of the formation of polyfluoroalkyl phosphonates **3**.

The starting fluoroalkyl vinyl ethers **1a** and **1b** were synthesized according to a previously developed procedure [23] by the direct vinylation of polyfluoroalkanols with acetylene in the presence of the $Cd(OAc)_2$–Bu_3N catalyst (Scheme 2).

The use of synthesized fluoroalkyl phosphonates **3a**–**3d** as additives in electrolytes for Li-ion batteries to increase their fire and explosion safety is promising [2, 3]. These compounds dissolve well in organic solvents, have a high dielectric constant ε (14–19), low viscosity η (11–37 cP), high boiling point (> 250°C) and low glass transition temperature (<−40°C), that is, they will not significantly affect the physical characteristics of the solvents used in electrolytes for Li-ion batteries [24]. Preliminary tests of fluoroalkyl phosphonates **3a**–**3d** by Samsung (Samsung SDI Co. Ltd.) using standard methods [2, 3] showed the feasibility of further studying this class of compounds to create new effective components of Li-ion batteries that prevent their combustibility, flammability and explosiveness.

In conclusion, a convenient, efficient and atom-economic method for the synthesis of previously unknown dialkyl [2-(polyfluoroalkoxy)ethyl]phosphonates was developed based on the AIBN-initiated reaction of radical addition of dialkyl (*H*)-phosphonates to available fluoroalkyl vinyl ethers. The obtained compounds are promising additives for enhancing the thermal stability of electrolytes, drug precursors, intermediates and components for the design of innovative materials.

EXPERIMENTAL

^1H, ^{13}C, ^{19}F, ^{31}P NMR spectra were recorded on Bruker DPX 400 and Bruker AV-400 spectrometers (400.13, 101.61, 376.50 and 161.98 MHz, respectively), internal standard—HMDS (^1H, ^{13}C), $CFCl_3$ (^{19}F), external standard – 85% H_3PO_4 (^{31}P). IR spectra were recorded on a Bruker IFS 25 spectrometer from thin layer. Mass spectra of electron ionization (70 eV) were obtained on a GCMS-QP5050A Shimadzu instrument. Elemental analysis was performed on a Flash EA 1112 Series analyzer. Fluorine and phosphorus are determined by combustion.

Fluoroalkyl vinyl ethers were obtained according to the procedure described in [23], dialkyl (*H*)-phosphonates were obtained from Aldrich. As an electrolyte a 1 M

solution of $LiPF_6$ in a mixture of ethylene carbonate and methyl ethyl carbonate (3 : 7 by volume) with the addition of 10 vol % phosphonate **3** was used.

General procedure for the synthesis of dialkyl [2-(trihydrofluoroalkoxy)ethyl]phosphonates 3a–3d. To 0.1 mol of dialkyl (*H*)-phosphonate **2** heated to 150°C was added dropwise with stirring a mixture of vinyl ether **1** (0.05 mol), dialkyl (*H*)-phosphonate **2** (0.1 mol) and 0.5–0.8 g (1.5 wt %) AIBN within ~ 1.5 h. The reaction mixture was stirred for another 0.5 h at the same temperature, then it was cooled and the desired phosphonates **3** were obtained by distillation in vacuum.

Dimethyl [2-(2,2,3,3-tetrafluoropropoxy)ethyl]-phosphonate (3a). Yield 11.38 g (85%), clear colorless liquid, bp 113–115°C (2 mmHg), d_4^{20} 1.4336, n_D^{20} 1.3940, ε 19.6±0.1, η 1.69×10^{-3} Pa s. IR spectrum, ν, cm^{-1}: 3005 w (CH–F), 2960 s, 2929 m, 2893 m, 2856 m (CH_2, CH_3), 1463 m, 1453 sh, 1414 w, 1403 w, 1382 w [δ(CH_3, CH_2)], 1254 s, 1237 s [P=O, δ(CF_2)], 1205 s, 1189 s (P–O–C), 1137 sh, 1116 s, 1104 s, 1059 s, 1033 s (C–O–C), 1001 sh [δ(C–O, P–O)], 938 m, 833 m, 800 sh [δ(CH_2), C–C], 742 w, 684 w (P–O), 592 w, 557 sh, 541 w, 469 w [δ(P–O–C, O–P=O)]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.08 d. t (2H, PCH_2, $^2J_{HP}$ = 18.6, $^3J_{HH}$ = 7.0 Hz), 3.70 d (6H, OCH_3, $^3J_{HP}$ = 11.0 Hz), 3.82 m (4H, OCH_2), 5.93 t. t (1H, CF_2H, $^2J_{HF}$ = 53.0, $^3J_{HF}$ = 5.0 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 25.7 d (PCH_2, $^1J_{CP}$ = 141.5 Hz), 52.2 d (OCH_3, $^2J_{CP}$ = 6.3 Hz), 66.2 (CH_2O), 67.7 t ($\underline{C}H_2CF_2$, $^2J_{CF}$ = 32.1 Hz), 109.0 t. t (HCF_2, $^1J_{CF}$ = 249.1, $^2J_{CF}$ = 34.3 Hz), 114.8 t. t (CF_2, $^1J_{CF}$ = 249.9, $^2J_{CF}$ = 26.9 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –140.24 d (HCF_2, $^2J_{HF}$ = 53.0 Hz), –125.55 (CF_2). ^{31}P NMR spectrum ($CDCl_3$): δ_P 31.11 ppm. Mass spectrum, *m/z* (I_{rel}, %): 153 (75) $[M - CH_2CF_2CF_2H]^+$, 137 (17) [153 − Me], 110 (80) $[(MeO)_2P(O)H]$, 109 (100) $[(MeO)_2P(O)]^+$, 79 (56) $[110 - MeO]^+$. Found, %: C 31.44; H 4.99; F 28.31; P 10.92. $C_7H_{13}F_4O_4P$ (*M* 268.143). Calculated, %: C 31.35; H 4.89; F 28.46; P 11.56.

Diethyl [2-(2,2,3,3-tetrafluoropropoxy)ethyl]-phosphonate (3b). Yield 12.35 g (83%), clear colorless liquid, bp 119–121°C (2 mmHg), d_4^{20} 1.2583, n_D^{20} 1.3970, ε 16.0±0.1, η 1.17×10^{-3} Pa s. IR spectrum, ν, cm^{-1}: 2986 s, 2934 s, 2912 s (CH_2, CH_3), 1482 m, 1460 m, 1446 m, 1394 m, 1371 m [δ(CH_3, CH_2, C–F)], 1270 sh, 1251 s, 1236 s (P=O), 1206 s, 1164 s (C–F, P–O–C), 1133 sh, 1116 s, 1103 s, 1058 s, 1028 s (C–O, C–F), 994 s, 963 s, 832 s, 793 m [δ(CH_2), C–C], 742 m, 684 m (P–C), 592 w, 557 sh, 541 m, 485 w [δ(P–O–C, O–P=O)]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.91 t (6H, CH_3, $^3J_{HH}$ = 7.0 Hz), 1.70 d. t (2H, PCH_2, $^2J_{HP}$ = 16.2, $^3J_{HH}$ = 7.0 Hz), 3.40 d. t (4H, $OC\underline{H}_2CH_3$, $^3J_{HP}$ = 11.1, $^3J_{HH}$ = 7.0 Hz), 3.68 m (4H, OCH_2), 5.56 t. t (1H, CF_2H, $^2J_{HF}$ = 53.2, $^3J_{HF}$ = 4.2 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 16.1 d (CH_3, $^3J_{CP}$ = 5.7 Hz), 26.7 d (PCH_2, $^1J_{CP}$ = 141.0 Hz), 61.5 d ($\underline{C}H_2Me$, $^3J_{CP}$ = 6.2 Hz), 66.5 (CH_2O), 67.7 t (CH_2CF_2, $^2J_{CF}$ = 30.1 Hz), 109.0 t. t (HCF_2, $^1J_{CF}$ = 249.2, $^2J_{CF}$ = 33.9 Hz), 114.8 t. t (CF_2, $^1J_{CF}$ = 249.9, $^2J_{CF}$ = 27.1 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –140.2 d (HCF_2, $^2J_{HF}$ = 53.2 Hz), –125.43 (CF_2). ^{31}P NMR spectrum ($CDCl_3$): δ_P 28.54 ppm. Mass spectrum, *m/z* (I_{rel}, %): 251 (11) $[M - EtO]^+$, 181 (89) $[M - CH_2CF_2CF_2H]^+$, 138 (52) $[(EtO)_2P(O)H]$, 137 (27) $[(EtO)_2P(O)]^+$, 109 (100) $[138 - Et]^+$, 93 (26) $[138 - EtO]^+$. Found, %: C 36.02; H 5.78; F 24.86; P 9.98. $C_9H_{17}F_4O_4P$ (*M* 296.196). Calculated, %: C 36.49; H 5.79; F 25.66; P 10.46.

Dimethyl [2-(2,2,3,3,4,4,5,5-octafluoropentyloxy)-ethyl]phosphonate (3c). Yield 12.15 g (66%), clear colorless liquid, bp 130–132°C (2 mmHg), d_4^{20} 1.4879, n_D^{20} 1.3798, ε 16.2±0.1, η 3.72×10^{-3} Pa s. IR spectrum, ν, cm^{-1}: 3011 w (CH–F), 2961 s, 2931 m, 2896 m, 2857 m (CH_2, CH_3), 1464 m, 1452 sh, 1402 m, 1381 m [δ(CH_3, CH_2)], 1253 s, 1237 sh [P=O, δ(CF_2)], 1171 s, 1128 s, 1062 s, 1035 s (C–O–C), 994 sh [δ(C–O, P–O)], 929 sh, 901 s, 841 sh, 809 s [δ(CH_2), C–C], 766 m, 708 m (P–O), 608 w, 544 m, 470 w [δ(P–O–C, O–P=O)]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.12 d. t (2H, PCH_2, $^2J_{HP}$ = 18.6, $^3J_{HH}$ = 7.1 Hz), 3.72 d (6H, OCH_3, $^3J_{HP}$ = 11.0 Hz), 3.83 d. t (2H, OCH_2, $^3J_{HP}$ = 14.2, $^3J_{HH}$ = 7.1 Hz,), 3.92 t (2H, $O\underline{CH}_2CF_2$, $^3J_{HF}$ = 13.9 Hz), 6.05 t. t (1H, CF_2H, $^2J_{HF}$ = 52.0, $^3J_{HF}$ = 5.5 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 25.4 d (PCH_2, $^1J_{CP}$ = 141.2 Hz), 51.8 d (OCH_3, $^2J_{CP}$ = 6.2 Hz), 66.4 (CH_2O), 67.4 t ($\underline{C}H_2CF_2$, $^2J_{CF}$ = 25.4 Hz), 107.2 t. t (HCF_2, $^1J_{CF}$ = 254.0, $^2J_{CF}$ = 30.2 Hz), 109.7–113.4 m ($\underline{C}F_2\underline{C}F_2$), 115.2 t. t ($\underline{C}F_2CH_2$, $^1J_{CF}$ = 256.5, $^2J_{CF}$ = 30.6 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –137.37 d (HCF_2, $^2J_{HF}$ = 52.0 Hz), –130.36 (CF_2), –125.69 (CF_2), –119.86 (CF_2). ^{31}P NMR spectrum ($CDCl_3$): δ_P 30.98 ppm. Mass spectrum, *m/z* (I_{rel}, %): 153 (100) $[M - CH_2(CF_2)_4H]^+$, 137 (34) [153 − Me], 110 (89) $[(MeO)_2P(O)H]$, 109 (96) $[(MeO)_2P(O)]^+$, 79 (56) $[110 - MeO]^+$. Found, %: C 28.83; H 3.73; F 41.87; P 8.36. $C_9H_{13}F_8O_4P$ (*M* 368.158). Calculated, %: C 29.36; H 3.56; F 41.28; P 8.41.

Diethyl [2-(2,2,3,3,4,4,5,5-octafluoropentyloxy)-ethyl]phosphonate (3d). Yield 13.27 g (67%), clear colorless liquid, bp 133–135°C (2 mmHg), d_4^{20} 1.3690,

n_D^{20} 1.3840, ε 14.2±0.1, η 2.37×10^{-3} Pa s. IR spectrum, ν, cm^{-1}: 3011 w (CH–F), 2961 s, 2931 m, 2896 m, 2857 m (CH_2, CH_3), 1464 m, 1452 sh, 1402 m, 1381 m [δ(CH_3, CH_2)], 1253 s, 1237 sh [P=O, δ(CF_2)], 1171 s, 1128 s, 1062 s, 1035 s (C–O–C), 994 sh [δ(C–O, P–O)], 929 sh, 901 s, 841 sh, 809 s [δ(CH_2), C–C], 766 m, 708 m (P–O), 608 w, 544 m, 470 w [δ(P–O–C, O–P=O)]. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.32 t (6H, CH_3, $^3J_{HH}$ = 7.0 Hz), 2.12 d. t (2H, PCH_2, $^2J_{HP}$ = 18.7, $^3J_{HH}$ = 7.3 Hz), 3.84 d. t (2H, OCH_2, $^3J_{HP}$ = 12.8, $^3J_{HH}$ = 7.3 Hz), 3.93 t (2H, O$\underline{CH_2}CF_2$, $^3J_{HF}$ = 14.0 Hz), 4.09 m (4H, O$\underline{CH_2}CH_3$), 6.05 t. t (1H, CF_2H, $^2J_{HF}$ = 52.0, $^3J_{HF}$ = 5.4 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 16.1 d (Me, $^3J_{CP}$ = 6.0 Hz), 26.8 d (PCH_2, $^1J_{CP}$ = 140.8 Hz), 61.6 d (OCH_2, $^2J_{CP}$ = 6.3 Hz), 66.9 (CH_2O), 67.4 t ($\underline{C}H_2CF_2$, $^2J_{CF}$ = 25.8 Hz), 107.5 t. t (HCF_2, $^1J_{CF}$ = 254.0, $^2J_{CF}$ = 30.2 Hz), 108.1–115.1 m ($\underline{C}F_2\underline{C}F_2$), 115.2 t. t ($CF_2$, $^1J_{CF}$ = 256.9, $^2J_{CF}$ = 30.8 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –137.41 d (HCF_2, $^2J_{HF}$ = 52.0 Hz), –130.40 (CF_2), –125.78 (CF_2), –119.91 (CF_2). ^{31}P NMR spectrum ($CDCl_3$): δ_P 28.13 ppm. Mass spectrum, *m/z* (I_{rel}, %): 351 (13) [M – EtO]$^+$, 181 (100) [M – $CH_2(CF_2)_4H$]$^+$, 138 (57) [$(EtO)_2P(O)H$], 137 (38) [$(EtO)_2P(O)$]$^+$, 109 (87) [138 – Et]$^+$, 93 (13), 91 (37), 83 (18), 82 (30), 81 (46), 65 (28), 51 (19), 45 (18) [EtO]. Found, %: C 33.02; H 4.41; F 37.94; P 7.38. $C_{11}H_{17}F_8O_4P$ (M 396.211). Calculated, %: C 33.35; H 4.32; F 38.36; P 7.82.

Determination of the self-extinguishing time. The electrolyte solution (0.25 mL) was placed into a standard 2032 battery cell and set on fire. Burning time without a flame source was fixed by a stopwatch. The exact quantities of electrolyte samples were determined by weighing, the average values of the self-extinguishing time (s/g) were obtained from three measurements and recalculated for 1 g of electrolyte.

Additive	**3a**	**3b**	**3c**	**3d**	No additive
Self-extinguishing time, s/g	106	108	107	108	125

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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