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Convenient synthesis of fluoroalkyl α- and β-aminophosphonates

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ABSTRACT

Addition of both alkyl phosphites and phosphonate α-carbanions to *N*-substituted aldimines derived from fluoroalkyl aldehydes presents a convenient method for synthesis of fluoroalkyl α- and β-aminophosphonates in good yield (55–86%) under mild conditions.

1. Introduction

Recently fluoroalkyl α- and β-aminophosphonic acids and their phosphonate esters have been the subject of considerable attention in organic and bioorganic chemistry because of their potential activity as metabolically stable transition state analogue inhibitors of different peptidases, antimicrobial, antibacterial, antihypertensive and anticancer agents as well as peptidomimetic units [1]. The efficient examples of aminophosphonates containing fluoroalkyl groups as inhibitors of serine esterases, alanine racemase and pyrimidine phosphorylases have been demonstrated [2]. Furthermore, considering the unique mode of hydrogen bonding observed in free aminophosphonic acids and their derivatives [3], these compounds might be very interesting models for studying self-disproportionation of enantiomers *via* achiral chromatography [4] and sublimation [5]. Despite the fact that several research groups have carried out extensive studies for synthesis of fluoroalkyl α- and β-aminophosphonic acid derivatives, their preparation is not trivial and usually includes multi-step procedures, which were summarized in recent comprehensive review [6]. General methods for preparation of fluoroalkyl α-and β-aminiophosphonates involve hydrogenation or reduction of fluoroalkyl α- and β-iminophosphonates and their isomeric enamines, base-catalyzed [1,3]-proton shift reaction of the *N*-benzyl substituted fluoroalkyl α- and β-iminophosphonates [7], exchange of hydroxyl group in fluoroalkyl *N*-acyl hemiaminals for phosphonate group *via* phosphite-phosphonate rearrangement, addition of ammonia or benzylamine to fluoroalkyl α,β-unsaturated phosphonates or ring opening of fluoroalkyl aziridine-2-phosphonates [8]. Although nucleophilic addition of alkyl phosphites and phosphonate α-carbanions to imines is a common approach for preparation of fluorine-free α- and β-aminophosphonates [9] analysis of the literature has revealed that potential of imines derived from polyfluoroalkyl aldehydes [10] in these reactions remains largely unexplored. Only multi-step procedure for assumed generation *in situ* of the corresponding *N*-phenyl imine of fluoral followed by addition of diethyl phosphite giving rise to corresponding phosphonotrifluoroalanine derivative have been developed [11]. Synthesis of phosphonotrifluoroalanine was also accomplished by treatment of *O*-trifluoroacetyl derivatives derived from hemiaminals of fluoral with dialkyl trimethylsilyl phosphites in pyridine [12]. It was suggested that intermediate *N*-acyl imines were trapped by dialkyl trimethylsilyl phosphites leading to corresponding α-trifluoromethyl α-aminophosphonates in quantitative yields. In contrast addition of dialkyl (or diaryl) phosphites or carbanions generated from phosphonates to the Schiff bases derived from hexafluoroacetone [13], trifluoromethyl ketones and trifluoropyruvates [14] as well as cyclic

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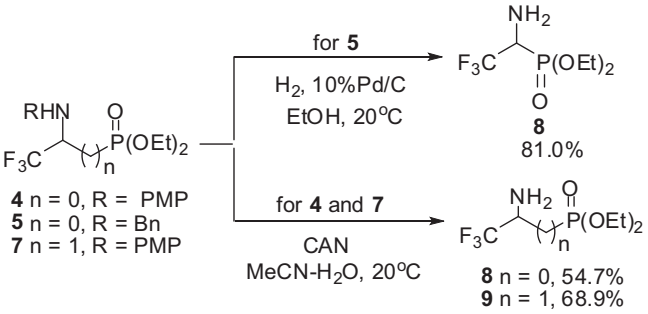
fluoroalkyl imines [15] in the presence of bases or Lewis acids was widely investigated. In this context it seemed quite reasonable to consider the addition of alkyl phosphites and α-carbanions derived from alkyl phosphonates to fluoroalkyl aldimines as a potentially convenient, straightforward and general approach for preparation of fluoroalkyl α- and β-aminophosphonates.

2. Results and discussion

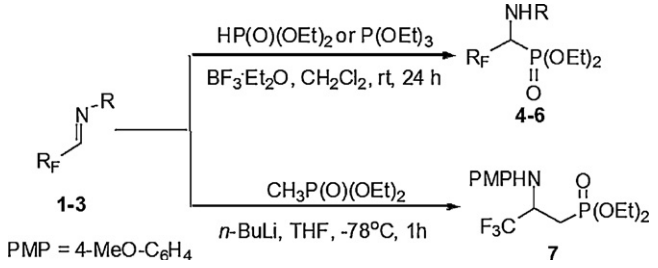
Initially the addition of diethyl phosphite to *N*-substituted fluoroalkyl aldimines **1**–**3** readily obtained by condensation of hydrate or hemiacetal of corresponding aldehydes with amines was studied. We found that reaction did not proceed in CH_2Cl_2 or ether at room temperature without any catalyst. However, the addition of diethyl phosphite on fluoroalkyl aldimines **1**–**3** in CH_2Cl_2 proceeded smoothly in the presence of boron trifluoride etherate (1 equiv.) at room temperature to give the desired α-fluoroalkyl α-aminophosphonates **4**–**6** (Table 1). After simple work-up procedure and purification by column chromatography α-fluoroalkyl α-aminophosphonates **4**–**6** were isolated in good yield. However, the purity of product **4** thus obtained was 96% (according the ^{19}F NMR data) and additional purification by recrystallization from hexane was required. The scaling up of the reactions did not affect the yield of products. The study of reaction of aldimine **2** with triethyl phosphite in CH_2Cl_2 showed that addition also proceeded in the presence of boron trifluoride etherate at room temperature to result in the formation of α-trifluoromethyl α-aminophosphonate **5**. The addition reaction between aldimine **1** and lithium salt of diethyl methylphosphonate proceeded smoothly in THF at −78 °C within 1 h to afford, after mild acidic work up, the corresponding β-trifluoromethyl β-aminophosphonate **7**, which was purified by crystallization. In general, the best results were obtained when the solution of the carbanion was added *via* cannula to a pre-cooled solution of the aldimine **1**. Variation of the solvents did not improve the efficiency of addition and resulted in incomplete conversion of starting aldimine **1** as indicated by ^{19}F NMR and TLC analysis of crude products.

For the removal of the substituent on the nitrogen, various methods were used depending of the chemical structure of substrates. Catalytic hydrogenolysis of **5** in EtOH in the presence of 20% palladium hydroxide on carbon under hydrogen atmosphere (1 atm) occurred smoothly within 1 h and afforded the desired α-aminophosphonate **8** with primary amino group as a single reaction product in excellent yield (Scheme 1). In spite of the presence of strong electron-withdrawing trifluoromethyl substituent, deprotection of **4** and **7** was effectively realized by treatment with excess of cerium ammonium nitrate (CAN) in a mixture of acetonitrile and water at room temperature giving rise to the *N*-dearylated product **8** and **9** in good chemical yield according to the literature procedure [16]. The structure and purity of the known products were confirmed by spectroscopic analysis and the new products were fully characterized.



Scheme 1.

Table 1
Addition of phosphorus reagents to *N*-substituted fluoroalkyl aldimines **1–3**



Entry	*N*-substituted Aldimine	R_F	R	Phosphorus reagent	Product	Yield[a] (%)
1	**1**	CF_3	PMP	$HP(O)(OEt)_2$	**4**	55.4
2	**2**	CF_3	Bn	$HP(O)(OEt)_2$	**5**	86.3
3	**3**	$H(CF_2)_6$	Bn	$HP(O)(OEt)_2$	**6**	63.3
4	**2**	CF_3	Bn	$P(OEt)_3$	**5**	58.5
5	**1**	CF_3	PMP	$CH_3P(O)(OEt)_2$	**7**	83.5

[a] Isolated yield.

3. Conclusions

In summary, we have reported that nucleophilic addition of alkyl phosphites or phosphonate α-carbanions to aldimines derived from polyfluoroalkyl aldehydes under mild reaction conditions followed by subsequent deprotection constitutes a convenient synthetic route leading to fluoroalkyl α- and β-aminophosphonates. Scope and limitation of this method are currently under investigation and will be reported in due course.

4. Experimental

NMR spectra were recorded on a Bruker DRX 500 or Varian Union Plus 400 spectrometers using TMS (^1H) and $CFCl_3$ (^{19}F) as an internal standards and H_3PO_4 (^{31}P) as an external standard. Chemical shifts (δ) are reported in ppm. IR spectra were recorded on Bruker Fourier-transform spectrometer VERTEX 70. Melting points are uncorrected. Analytical TLCs were performed with Merck silica gel 60 F_{254} plates. Visualization was accomplished by ultraviolet light (254 nm). Flash chromatography was carried out using Merck silica gel 60.

The starting *N*-substituted fluoroalkyl aldimines **1**–**3** were prepared according to known procedures in 60–80% yields by heating a toluene solution of hemiacetal or hydrate of corresponding fluoroalkyl aldehydes with amines in the presence of catalytic amount of *p*-toluenesulfonic acid in a Dean-Stark apparatus [17].

4.1. *N*-Substituted diethyl α-fluoroalkyl α-aminophosphonates 4–6; general procedure

To a solution of corresponding *N*-substituted imine of fluoroalkyl aldehyde (4.92 mmol) in dichloromethane (10 mL) were added alkyl phosphite (9.84 mmol) and $BF_3{\cdot}Et_2O$ (0.62 mL, 4.92 mmol) at 0 °C. After being stirred at the room temperature for 24 h, the reaction mixture was poured into a saturated aqueous solution of $NaHCO_3$ (5 mL). The aqueous layer was extracted with dichloromethane (2 × 5 mL), and the combined organic extracts were washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure.

4.1.1. Diethyl 2,2,2-trifluoro-1-(4-methoxyphenylamino)-ethylphosphonate (4)

Purification by flash chromatography, eluent hexane–ether 1:1, followed by recrystallization from hexane gave **4** in 55.4% as white solid, mp 72–73 °C. Spectral properties are in agreement with those reported in the literature [16]. IR (CH_2Cl_2): ν 3412, 3058, 2986, 2837, 1515, 1243, 1173, 1114, 1024, 979 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.27 (t, *J* 7.0 Hz, 3H), 1.33 (t, *J* 7.0 Hz, 3H), 3.75 (s, 3H), 4.00–4.03 (m, 1H), 4.11–4.23 (m, 5H), 6.69 (d, *J* 8.2 Hz, 2H), 6.80 (d, *J* 8.2 Hz, 2H); ^{19}F NMR (376 MHz, $CDCl_3$): δ −69.78 (m); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 19.57 (q, *J* 8,6 Hz).

4.1.2. Diethyl 1-benzylamino-2,2,2-trifluoroethylphosphonate (5)

Purification by flash chromatography, eluent hexane–ether 1:1, gave **5** as colorless oil in 86.3% yield using diethyl phosphite or 58.5% using triethyl phosphite. IR (CH_2Cl_2): ν 3365, 3001, 1455, 1257, 1168, 1114, 1051, 1026, 977 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.34 (t, *J* 7.1 Hz, 3H), 1.35 (t, *J* 7.1 Hz, 3H), 2.07 (br. s, 1H), 3.43 (dd, *J* 21.0 and 8.4 Hz, 1H), 3.99 (d, *J* 13.1 Hz, 1H), 4.09 (d, *J* 13.1 Hz, 1H), 4.12–4.22 (m, 4H), 7.28–7.35 (m, 5H); ^{19}F NMR (470 MHz, $CDCl_3$): δ −68.32 (dd, *J* 9.4 and 8.4 Hz); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 21.12 (q, *J* 9.4 Hz). Calcd. for $C_{13}H_{19}F_3NO_3P$: C, 48.00; H, 5.89; N, 4.31; P 9.52. Found: C, 48.26; H, 5.93; N, 4.54; P, 9.47.

4.1.3. Diethyl 1-benzylamino-2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptylphosphonate (6)

Purification by flash chromatography, eluent hexane–ethylacetate 2:1, gave 63.3% of **6** as colorless oil. IR (CH_2Cl_2): ν 3357, 2985, 2910, 1454, 1203, 1142, 1050, 1024, 975 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.39 (m, 6H), 1.84 (br. s, 1H), 3.67–3.75 (m, 1H), 3.93 (d, *J* 11.0 Hz, 1H), 4.12 (d, *J* 11.0 Hz, 1H), 4.24 (m, 4H), 6.07 (t, *J* 52.4 Hz, 1H), 7.29–7.36 (m, 5H); ^{19}F NMR (470 MHz, $CDCl_3$): δ −110.50 (d, *J* 285.0 Hz, 1F), −116.70 (d, *J* 285.0 Hz, 1F), −119.61 (d, *J* 296. 9 Hz, 1F), −121.20 (d, *J* 296.9 Hz, 1F), −121.56 (d, *J* 302.1 Hz, 1F), −123.08 (d, *J* 302.4 Hz, 1F), −123.24 (d, *J* 302.1 Hz, 1F), −124.68 (d, *J* 302.4 Hz, 1F), −130.14 (s, 2F), −136.95 (dd, *J* 309.7 and 52.4 Hz, 2F), −138.19 (dd, *J* 309.7 and 52.4 Hz, 1F); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 18.62 (s). Calcd. for $C_{18}H_{20}F_{12}NO_3P$: C, 38.79; H, 3.62; N, 2.51; P 5.56. Found: C, 38.81; H, 3.65; N, 2.63; P, 5.51.

4.2. Diethyl 3,3,3-trifluoro-2-(4-methoxyphenylamino)-propylphosphonate (7)

To a solution of diethyl methylphosphonate (1.85 g, 12.14 mmol) in THF (25 mL), stirring at −78 °C, was added 1.6 M *n*-BuLi (7.6 mL, 12.14 mmol) dropwise. After stirring for 50 min at −78 °C the solution was transferred *via* cannula into a solution of the imine **1** (1.64 g, 8.09 mmol) in THF (5 mL) at −78 °C. Stirring was continued at −78 °C for additional 1 h before the reaction was quenched by the dropwise addition of aqueous NH_4Cl (15 mL). The layers were separated and the aqueous layer was extracted with Et_2O (3 × 15 mL). The organic layers were combined and washed with brine (10 mL), dried over Na_2SO_4 and concentrated under reduced pressure. Recrystallization from hexane gave 2.4 g (83.5%) of **7** as a colorless solid, mp 127–128 °C. IR (CH_2Cl_2): ν 2985, 1516, 1239, 1170, 1127, 1029, 972 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.19 (t, *J* 7.2 Hz, 3H), 1.26 (t, *J* 7.2 Hz, 3H), 2.10 (ddd, *J* 17.5, 15.6 and 11.3 Hz, 1H), 2.26 (ddd, *J* 20.2, 15.6 and 2.8 Hz, 1H), 3.75 (s, 4H), 3.96–4.09 (m, 4H), 4.15 (m, 1H), 6.72 (d, *J* 8.2 Hz, 2H), 6.78 (d, *J* 8.2 Hz, 2H); ^{19}F NMR (470 MHz, $CDCl_3$): δ −77.52 (d, *J* 6.4 Hz); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 30.57 (s). Calcd. for $C_{14}H_{21}F_3NO_4P$: C, 47.33; H, 5.96; N, 3.94; P 8.72. Found: C, 47.59; H, 6.28; N, 4.14; P, 9.07.

4.3. Diethyl 1-amino-2,2,2-trifluoroethylphosphonate (8)

To solution of diethyl 1-benzylamino-2,2,2-trifluoroethylphosphonate **5** (700 mg, 2.15 mmol) in absolute ethanol (19 mL) was added 20% palladium hydroxide on carbon (450 mg). The reaction mixture was stirred under hydrogen (1 atm) at room temperature for 1 h and then was filtered through Celite, washed with ethanol (3 × 10 mL) and concentrated under reduced pressure. Purification by flash chromatography, eluent ether-ethyl acetate 2:1, gave 410 mg (81.0%) of **8** as colorless oil. Spectral properties are in agreement with those reported in the literature [16,18]. IR (CH_2Cl_2): 3416, 3004, 1263, 1183, 1118, 1053, 1026, 978 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.37 (t, *J* 7.0 Hz, 6H), 1.73 (s, 2H), 3.56 (dq, *J* 19.4 and 8.4 Hz, 1H), 4.21–4.26 (m, 4H); ^{19}F NMR (470 MHz, $CDCl_3$): δ −72.03 (dd, *J* 8.4 and 7.7 Hz); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 21. 13 (q, *J* 7.7 Hz).

4.4. Diethyl 2-amino-3,3,3-trifluoropropylphosphonate (9)

To a stirred solution of diethyl 3,3,3-trifluoro-2-(4-methoxyphenylamino)propylphosphonate **7** (1.51 g, 4.25 mmol) in CH_3CN (110 mL) and H_2O (50 mL) at 0 °C, CAN (14.0 g, 25.50 mmol) was added in one portion. Then the resulting solution stirred at room temperature for 18 h, and quenched with saturated aqueous $NaHCO_3$ solution (75 mL). The aqueous layer was extracted with CH_2Cl_2 (2 × 50 mL) and the combined organic layer was washed with 5% aqueous Na_2SO_3 solution (50 mL), dried over anhydrous Na_2SO_4, and concentrated under vacuum. The resultant crude residue was purified by recrystallization from hexane to obtain 0.73 g (68.9%) of **9** as a white solid, mp 51–52 °C. Spectral properties are in agreement with those reported in the literature [8f]. IR (CH_2Cl_2): ν 3683, 3411, 2985, 1252, 1169, 1124, 1055, 1027, 967 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 1.35 (t, *J* 7.1 Hz, 3H), 1.36 (t, *J* 7.1 Hz, 3H), 1.71 (s, 2H), 1.89 (ddd, *J* 17,1, 15.4 and 11.4 Hz, 1H), 2.15 (ddd, *J* 21.1, 15.4 and 2.0 Hz, 1H), 3.60–3.73 (m, 1H), 4.09–4.22 (m, 4H); ^{19}F NMR (470 MHz, $CDCl_3$): δ −80.38 (d, *J* 6.6 Hz); ^{31}P {^1H} NMR (202 MHz, $CDCl_3$): δ 27.6 (s).

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