Alum Catalyzed Convenient Synthesis of Quino[2,3-b][1,5]benzoxazepine α-Aminophosphonate Derivatives

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We have described an efficient synthesis of quino[2,3-b][1,5]benzoxazepine α-aminophosphonate derivatives by the nucleophilic addition of triethyl phosphite to substituted quino[2,3-b][1,5]benzoxazepines promoted by easily available, inexpensive and mild catalyst $KAl(SO_4)_2 \cdot 12H_2O$ (alum). The reactions proceed smoothly at room temperature under solvent-free reaction conditions and providing high yield of product in very short reaction time.

Key Words: α-Aminophosphonate, Alum, Quino[2,3-b][1,5]benzoxazepine, Triethyl phosphite, Solvent-free

Introduction

Organophosphorus compounds are important organic compounds due to their broad spectrum of applications.[1] α-Aminophosphonates have attracted considerable attention since they are considered as structural analogues of the corresponding α-amino acids and their utilities as enzyme inhibitors, antibiotics, pharmacological agents, peptidomimetics, hapten design in antibody generation and many other applications are well documented.[2] In addition, phosphonates show good antibacterial activity with the quinoline nucleus.[3]

Quinoline ring systems represents a major class of heterocycles as they occur in various natural products especially in alkaloids.[4] It possess diverse biological and physiological activities such as antimalarial,[5a] anti-inflammatory,[5b] antitumor,[5c] DNA binding capacity,[5d] antibacterial properties.[5e] Recently, quinoline has been employed in the study of bio-organic and bio-organometallic processes.[5f] Seven membered heterocycles with two heteroatoms at 1,4-position in particular, derivatives of benzoxazepine exhibit a broad spectrum of biological properties such as neurotropic and psychotropic,[6a] anti-inflammatory,[6b] anticonvulsive,[6c] antagonistic properties against prostaglandin,[6d] high ceiling diuretics[6e] and antidepressant activities.[6f] Also the metal complexes of quino[2,3-b][1,5]benzoxazepine show effective antibacterial and antifungal activities.[7] Taking into account the biological properties associated with the α-aminophosphonates, quinolines and 1,4-oxazepines; our aim was to synthesize a series of quino[2,3-b][1,5] benzoxazepine α-aminophosphonate from 1,4-oxazepines of quinoline.

Generally, α-aminophosphonates are prepared by the reaction of phosphite with imine in the presence of a Bronsted acid[8a] or Lewis acids like $ZnCl_2$,[8b] BF_3-Et_2O,[8c] CdI_2/Benzene,[8d] and CdI_2/Microwave.[8e] One-pot syntheses of α-aminophosphonates have been carried out by the reaction of an aldehyde or ketone, an amine and dialkyl phosphonate or trialkylphosphite in organic solvents using $InCl_3$,[9a] $ZrCl_4$,[9b] GaI_3,[9c] $BiCl_3$,[9d] $SbCl_3/Al_2O_3$[9e] and under solvent-free conditions uncatalyzed[10a] or using TFA,[10b] $LiClO_4$,[10c] metal triflates,[10d] $Na_2CaP_2O_7$,[10e] $ZrOCl_2 \cdot 8H_2O$ or $ZrO(ClO_4)_2 \cdot 6H_2O$,[10f] and TsCl.[10g]

Solvent-free reaction condition has been demonstrated to be an efficient technique for various organic reactions. It often leads to a remarkable decrease in reaction time, increased yields, easier workup, enhancement of regio and stereoselectivity of reaction.[11]

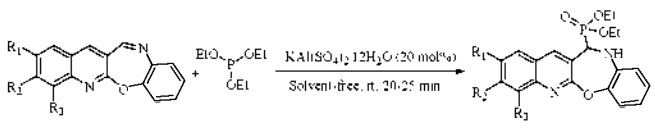
$KAl(SO_4)_2 \cdot 12H_2O$ (alum) was found to be effective in the synthesis of *cis*-isoquinolic acids,[12a] mono- and disubstituted 2,3-dihydroquinazolin-4(1H)-ones,[12b] dihydropyrimidines *via* Biginelli reaction,[12c] coumarins,[12d] trisubstituted imidazoles,[12e] 2-arylbenzothiazoles, and 2-arylbenzoxazoles.[12f]

Results and Discussion

In continuation of our research devoted to the phosphorus chemistry[3,13] and interest in the development of novel synthetic methodologies,[12f,14] herein, we would like to report a simple, efficient and rapid method for the synthesis of quino[2,3-b][1,5]benzoxazepine α-aminophosphonate derivatives at room temperature (Scheme 1).

In search for an efficient catalyst and the best experimental reaction conditions, the reaction of quino[2,3-b][1,5]benzoxazepine **1a** and triethyl phosphite **2** at room temperature has been considered as a standard model reaction.

When the reaction was carried out in the absence of catalyst the product formed in trace amount (Table 1, Entry 1). In the next step, we have screened different catalysts for the model



1(a-j) **2** **3(a-j)**

Scheme 1

Table 1. Screening of Catalyst

Entry	Catalyst	Yield[a] (%)
1	-	Trace
2	Acidic alumina	23[b]
3	$SrCl_2$	35
4	$ZnCl_2$	48
5	Sulphamic acid	55
6	Alum	72[c]

Reaction conditions: **1a** (1 mmol), Triethyl phosphite (1.5 mmol), Catalyst (10 mol%), under solvent-free condition at rt for 20 min, [a]Isolated yields. [b]Catalyst (10 mg), [c]No further improvement in yield of product was observed after 20 min.

Table 2. Effect of Concentrations of Catalyst

Entry	Alum (mol%)	Yield[a] (%)
1	5	68
2	10	72
3	15	76
4	20	85
5	25	85

Reaction conditions: **1a** (1 mmol), Triethyl phosphite (1.5 mmol) under solvent-free condition at rt for 20 min. [a]Isolated yields.

Table 3. Screening of solvents

Entry	Solvent	Yield[a] (%)
1	Dichloromethane	32
2	Tetrahydrofuran	35
3	1,4-Dioxane	41
4	Toluene	44
5	Acetonitrile	52
6	Ethanol	54

Reaction conditions: **1a** (1 mmol), Triethyl phosphite (1.5 mmol), Alum (20 mol%) solvent (5 mL) at rt for 20 min, [a]Isolated yields.

reaction such as acidic alumina, $SrCl_2$, $ZnCl_2$, sulphamic acid and alum. By the use of acidic alumina and $SrCl_2$ as a catalyst the product formed in poor yields 23-35% (Table 1, Entry 2-3), whereas using $ZnCl_2$ and sulphamic acid the product was obtained in moderate yields 48-55% (Table 1, Entry 4-5). In comparison with these results alum provided the better yield of product (72%) at room temperature under solvent-free conditions (Table 1, Entry 6).

To determine the appropriate concentration of the catalyst alum, we investigated the model reaction at different concentrations of alum such as 5, 10, 15, 20 and 25 mol%. The product formed in 68, 72, 76, 85 and 85% yield respectively (Table 2). This indicates that 20 mol% of alum is sufficient for the best result by considering yield of product.

To evaluate the effect of solvent, various solvents such as dichloromethane, tetrahydrofuran, 1,4-dioxane, toluene, acetonitrile and ethanol were used for the model reaction. It was observed that the use of solvent retards the rate of reaction and affords the desired product in lower yields than that for neat condition (Table 3).

Table 4. Synthesis of oxazepine α-aminophosphonates **3**[a]

Entry	R_1	R_2	R_3	Time (min)	Yield[b] (%)	M. P. (°C)
3a	H	H	H	20	85	192-194
3b	CH_3	H	H	20	90	153-155
3c	H	CH_3	H	20	83	113-115
3d	H	H	CH_3	25	83	133-135
3e	OCH_3	H	H	25	85	119-121
3f	H	OCH_3	H	25	84	120-122
3g	OC_2H_5	H	H	20	87	150-152
3h	F	H	H	25	86	180-182
3i	H	F	H	25	80	134-136
3j	Cl	H	H	20	88	144-146

[a]Reaction conditions: **1** (1 mmol), **2** (1.5 mmol), Alum (20 mol%) solvent-free, at rt. [b]Isolated yields.

With these optimized reaction conditions, we have synthesized a series of quino[2,3-b][1.5]benzoxazepine α-aminophosphonate derivatives (**3a-j**) by reacting substituted quino [2.3-b][1,5]benzoxazepines (**1a-j**) with triethyl phosphite **2** in presence of alum at room temperature under solvent-free conditions. The products formed within 20-25 min in 80-90% yields and have been confirmed by IR, ^{1}H NMR, mass spectroscopic data and elemental analysis.

The reaction was compatible with the various substituents such as CH_3, OCH_3, OC_2H_5, F and Cl and no significant substituent effects were observed. No competitive nucleophilic methyl/ethyl ether cleavage was observed for the substrate having OCH_3 or OC_2H_5 groups.

The reaction is proposed to proceed by the binding of alum with the nitrogen of imine which ultimately enhances the electrophilicity of the carbon and leads to tremendous increase in the reaction rate as compared to uncatalyzed reaction.

Conclusions

We conclude that, alum allows the convenient and ecological synthesis of quino[2,3-b][1.5]benzoxazepine α-aminophosphonates. The remarkable advantages offered by this method are solvent-free reaction conditions, short reaction times, high yields of products. Further studies to investigate the applications of alum as a catalyst for the synthesis of other α-aminophosphonates and biological screening of titled compounds are in progress.

Experimental Section

All the melting points were determined in open capillaries in paraffin bath and are uncorrected. IR spectra were recorded on a Perkin-Elmer FTIR using KBr discs. ^{1}H NMR spectra were recorded on Mercury Plus Varian in DMSO or $CDCl_3$ at 400 MHz using TMS as an internal standard. Mass spectra were recorded on Micromass Quattro II using electrospray Ionization technique. The elemental analysis was carried out on Flash EA 1112, 50/60 Hz, 1400 VA CHNS analyzer. The

progress of the reactions was monitored by TLC.

General experimental procedure. A mixture of quino[2,3-b][1,5]benzoxazepine (1 mmol), triethyl phosphite (1.5 mmol) and powdered alum (20 mol%) was stirred magnetically at room temperature. After the completion of reaction as monitored by TLC; ice cold water (20 mL) was added to the reaction mixture. The crude mixture was extracted with ethyl acetate and purified by column chromatography on silica gel to afford α-aminophosphonate.

Analytical data of principle compounds. (**3a**) IR (KBr, cm^{-1}): 3280 (N-H), 1615 (C=C), 1248 (P=O), 1040 (P-O-C). ^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 8.20 (d, 1H, J = 1.92 Hz, Ar-CH), 7.98 (d, 1H, J = 8.44 Hz, Ar-CH), 7.77 (d, 1H, J = 8.08 Hz, Ar-CH), 7.70 (t, 1H, J = 7.28 Hz, Ar-CH), 7.48 (td, 1H, J = 7.08, 0.92 Hz, Ar-CH), 7.39 (dd, 1H, J = 6.6, 1.4 Hz, Ar-CH), 6.98 (td, 1H, J = 6.16, 1.4 Hz, Ar-CH), 6.87 (m, 2H, Ar-CH), 5.0 (dd, 1H, J = 13.56, 5.6 Hz, P-CH), 4.5 (d, 1H, J = 5.6 Hz, NH), 4.0 (m, 3H, O-CH_2), 3.8 (m, 1H, O-CH), 1.2 (t, 6H, J = 5.72 Hz, CH_3). ES-MS: m/z 385. Elemental analysis: $C_{20}H_{21}N_2O_4P$ Calc.: C: 62.50%, H: 5.51%, N: 7.29%; Found: C: 62.45%, H: 5.46%, N: 7.23%. (**3d**) IR (KBr, cm^{-1}): 3270 (N-H), 1609 (C=C), 1251 (P=O), 1020 (P-O-C). ^{1}H NMR (DMSO-d_6, 400 MHz, δ ppm): 8.42 (s, 1H, Ar-CH), 7.74 (d, 1H, J = 8 Hz, Ar-CH), 7.58 (d, 1H, J = 7.2 Hz, Ar-CH), 7.42 (t, 1H, J = 8.0, 7.2 Hz, Ar-CH), 7.12 (d, 1H, J = 8.4 Hz, Ar-CH), 6.85 (m, 2H, Ar-CH), 6.66 (m, 2H, Ar-CH), 5.0 (dd, 1H, J = 13.6, 6.4 Hz, P-CH), 3.90 (m, 3H, O-CH_2), 3.80 (m, 1H, O-CH), 2.62 (s, 3H, Ar-CH_3), 1.07 (t, 6H, J = 7.2 Hz, CH_3). ES-MS: m/z 399. Elemental analysis: $C_{21}H_{23}N_2O_4P$ Calc.: C: 63.31%, H: 5.82%, N: 7.03%; Found: C: 63.27%, H: 5.76%, N: 6.97%. (**3e**) IR (KBr, cm^{-1}): 3273 (N-H), 1610 (C=C), 1249 (P=O), 1020 (P-O-C). ^{1}H NMR (DMSO-d_6, 400 MHz, δ ppm): 8.33 (s, 1H, Ar-CH), 7.76 (d, 1H, J = 8.8 Hz, Ar-CH), 7.38 (d, 1H, J = 8.4 Hz, Ar-CH), 7.30 (s, 1H, Ar-CH), 7.07 (d, 1H, J = 6.4 Hz, Ar-CH), 6.80 (m, 2H, Ar-CH), 6.63 (m, 2H, Ar-CH), 4.9 (dd, 1H, J = 12.4, 5.2 Hz, P-CH), 3.94 (m, 3H, O-CH_2), 3.90 (m, 1H, O-CH), 3.80 (s, 3H, Ar-OCH_3), 1.10 (t, 6H, J = 7.2 Hz, CH_3). ES-MS: m/z 415. Elemental analysis: $C_{21}H_{23}N_2O_5P$ Calc.: C: 60.87%, H: 5.59%, N: 6.76%; Found: C: 60.81%, H: 5.55%, N: 6.72%. (**3g**) IR (KBr, cm^{-1}): 3275 (N-H), 1606 (C=C), 1273 (P=O), 1041 (P-O-C). ^{1}H NMR (DMSO-d_6, 400 MHz, δ ppm): 8.31 (d, 1H, J = 1.2 Hz, Ar-CH), 7.75 (d, 1H, J = 9.2 Hz, Ar-CH), 7.35 (dd, 1H, J = 6.8, 2.4 Hz, Ar-CH), 7.29 (d, 1H, J = 2.4 Hz, Ar-CH), 7.07 (d, 1H, J = 8.0 Hz, Ar-CH), 6.86 (t, 1H, J = 8.0, 7.2 Hz, Ar- CH), 6.79 (d, 1H, J = 6.8 Hz, Ar-CH), 6.62 (m, 2H, Ar-CH), 4.94 (dd, 1H, J = 12.4, 6.4 Hz, P-CH), 4.13 (qua, 2H, J = 6.8 Hz, O-CH_2), 3.94 (m, 3H, O-CH_2), 3.84 (m, 1H, O-CH), 1.37 (t, 3H, J = 6.8 Hz, CH_3), 1.09 (t, 6H, J = 7.2 Hz, CH_3). ES-MS: m/z 429. Elemental analysis: $C_{22}H_{25}N_2O_5P$ Calc.: C: 61.68%, H: 5.88%, N: 6.54%; Found: C: 61.63%, H: 5.82%, N: 6.50%. (**3j**) IR (KBr, cm^{-1}): 3278 (N-H), 1607 (C=C), 1273 (P=O), 1045 (P-O-C). ^{1}H NMR (DMSO-d_6, 400 MHz, δ ppm): 8.43 (s, 1H, Ar-CH), 8.06 (d, 1H, J = 1.2 Hz, Ar-CH), 7.87 (d, 1H, J = 8.8 Hz, Ar-CH), 7.73 (dd, 1H, J = 7.2, 1.6 Hz, Ar-CH), 7.09 (d, 1H, J = 8.0 Hz, Ar-CH), 6.86 (m, 2H, Ar-CH), 6.66 (m, 2H, Ar-CH), 5.0 (dd, 1H, J = 13.6, 6.4 Hz, 5P-CH), 3.94 (m, 3H, O-CH_2), 3.82 (m, 1H, O-CH), 1.10 (t, 6H, J = 7.2 Hz, CH_3). ES-MS: m/z 419. Elemental analysis: $C_{20}H_{20}N_2O_4ClP$ Calc.: C: 57.36%, H: 4.81%, N: 6.69%; Found: C: 57.31%, H: 4.77%, N: 6.63%.

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