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Green Synthesis of α-hydroxyphosphonates by using DES Catalyst

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ABSTRACT

Synthesis of series of bioactive α-hydroxy phosphonates (2a-i) stirred by the benevolent choline chloride-based Urea a deep eutectic mixture wasemployed as an efficient and green ionic liquid catalyst for solvent free condition at room temperature. The current approach to generate sustainable catalyst in place of volatile organic compounds to 2-chloroquinoline-3-carbaldehyde (1a–i) with triethylphosphite. The reaction is furnished in short time and products were obtained in good yield.

Keyword- α-hydroxy phosphonates, deep eutectic mixture, volatile organic compounds

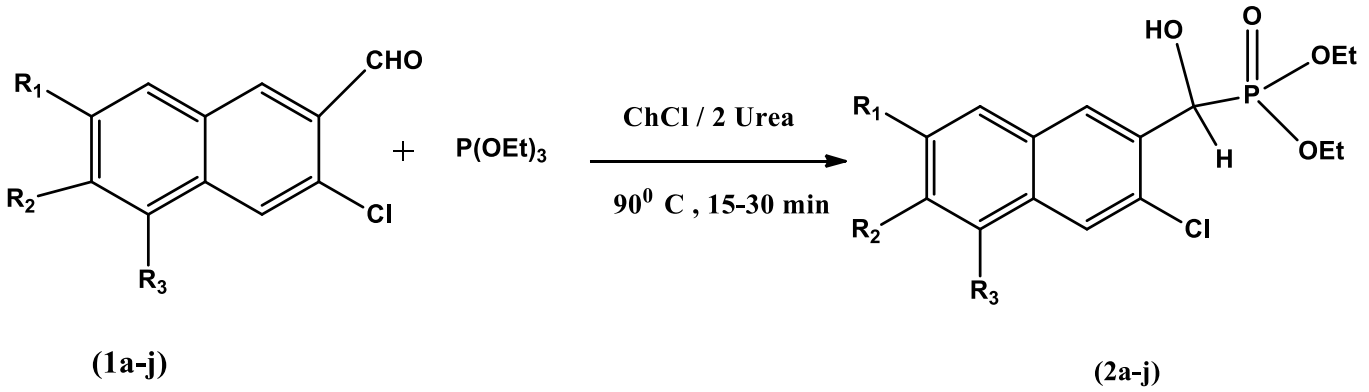
I. INTRODUCTION

The α-hydroxyphosphonates have drawn much attention due to their wide range of applications and their significance as synthetic transitional for additional biologically vital α-substituted phosphoryl compounds [1]. Noteworthy, attempts have been grownup for the formulating hydroxyphosphonates with the Abramov reaction symbolizes one of the highly fitmethods for the synthesis of α-hydroxyphosphonates [2]. The process involves a condensation between an aldehyde and triethylphosphite promoted by series of Lewis or Bronsted acid catalyst such as KH_2PO_4[3], sulphamicacid[4],camphor sulphonic acid[5],tartaric acid[6],succinic acid, guanidine hydrochloride[7], I_2catalyst functions as acid catalyst[8], $ZnBr_2$[9], β-cyclodextrin[10], Ammonium metavandate and Bismuth (III) nitrate pentahydrate and TMSCl found to be efficient catalytic system[11].However, these methods come across some drawbacks such as longer reaction time, use of volatile hazardous organic compounds VOC and expensive catalysts, harsh reaction conditions followed by low yield of product hence do not acknowledge with green chemistry. Therefore, many synthetic methods for the construction α-hydroxyphosphonates in the presence of catalyst or activator have been developed.

To overcome these drawbacks, new methodologies based on ultrasound, microwave, and use of ionic solvents have published in recent times. Owing to their significant taskin the synthesis of biologically active component, the progress of ecologically bearable synthesis of α-hydroxyphosphonates is desirable. The fresh development in chemical synthesis, the largely focusonreaction effectiveness, decrease of waste,moveaway from the harmful reagents, and theprecise utilization of resources have grown into the main objective.Deep eutectic solvents(DESs) have been stirring a lot of advantage than toxic organic acids based ionic liquid. A DES blend of choline chloride and Urea form a steady Lewis acidic liquid [12] and performances as a powerful alternate for the

usuallyactive risky ionic liquid to carbonyl oxygen, crafting the C=O bond extraelectrophilic. Choline chloride–Urea initiatedionic molten clear liquid has been successfully used for the pyrrole derivative,Fischer indole synthesis, [13] O-acetylation of cellulose and monosaccharides, [14]and protection of carbonyl compounds. [15]We report here our results of ChCl 2Urea catalyzed modified Abramov reaction.

Quinolines [16a]are an important class of heterocyclic compounds and have been screened for numerous biological activities such as bactericidal [16b], antitumor [16c], anti-inflammatory [16d], and antimalarial [16e] . Quinolines such as 2-chloroquinoline-3-carbaldehyde capture a noticeable position as they are key intermediates for extend annellation and for several functional group interconversions [17a]. It is furthermore reported that organophosphates are effective pesticides which have wide variety of application [17b]. Recently, some new vinyl phosphates have been reported as convincing inhibitors of phosphatase [17c] and phosphodiesterase [17d].



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

Scheme: Synthesis of [2a-j] from [1a-j] by using Deep eutectic catalyst

II. RESULTS AND DISCUSSION

The original work of Abramov reaction involved the heating under microwave of an aldehyde or a ketone withtrialkylphosphite at 70-100⁰C for several hours in sealed tube[18]. Under these stringent conditionsdialky-α-alkoxyalkylphosphonates could be isolated in various yields.To overcome these difficulties in attempting Abramov reaction silyl halide can be usedalong with the carbonyl compounds and the phosphorous reagent [19]. Removal of the residual silylester linkages at the phosphonate Centre is accomplished with water or alcohol under mildconditions. Hence the Abramov reaction with such modification is referred as "ModifiedAbramov Reaction".α-hydroxyphosphonates may serve as precursors for the synthesis of alpha aminophosphonates which are analogs of alpha amino acid [20]. Hence a search for new biological activecompound has stimulated recent studies on the synthesis of α-substituted phosphonates.In continuation of our work on phosphorus chemistry, [21], herein several examples ofmodified Abramov reaction on 2-chloroquinolin-3-carbaldehydes **(1a-j)** are presented to afford α-hydroxyphosphonates **(2a-j)** compounds.

The one pot two-component reaction between triethyl phosphite (2 mmol), and quinoline 3-carbaldehyde (2 mmol), was chosen as a model reaction. All the reaction conditions were optimized using $ChCl_2$/2Urea as a catalyst[22]. The reaction mixture was stirred at room temperature under solvent-free condition. It was observed that initially the reaction mixture was clear and after 10 min the solid product formation started. It is noteworthy that, no significant product formation was observed under similar reaction conditions in the absence of catalyst even after 5 h (Table 1)

Table 1.DES facilitated synthesis of a-hydroxyphosphonates.

Entry	*R_1*	*R_2*	*R_3*	*Time (min)*	*Yield (%)*	*MP (^{0}C)*
2a	H	H	H	25	87	128-130^{0}C
2b	CH_3	H	H	21	88.8	145-147^{0}C
2c	H	CH_3	H	20	82.6	141-143^{0}C
2d	H	H	CH_3	22	82.5	172-174 ^{0}C
2e	OCH_3	H	H	19	84.1	156-160 ^{0}C
2f	H	OCH_3	H	18	83.2	166-170 ^{0}C
2g	H	H	OCH_3	24	77.2	149-151^{0}C
2h	OCH_2CH_3	H	H	23	85.9	168-170^{0}C
2i	H	H	CH_2CH_3	27	73.2	149-151^{0}C
2j	Cl	H	H	25	79.9	115-119^{0}C

III. EXPERIMENTAL SECTION

General Procedures.

2-Chloroquinoline-3-carbaldehydes were prepared in the laboratory by thereported method. [23]triethylphosphitewere procured from Sigma Andrich. methanol andhexane,Choline chloride, Urea, were procured from S. D. Fine-chem.All melting points were determined in open capillaries on Kumar's melting point apparatus. The products were characterized by their spectral data. ^{1}H NMR spectra were recorded on a Varian INOVA-500 (500 MHz) spectrometer.Chemical shifts are reported in ppm from the solvent resonance as the internal standard (DMF-d7; 8.03 ppm) or tetramethylsilane (0.0 ppm) resonance as the internal standard ($CDCl_3$). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, br = broad) and coupling constants (Hz).Infrared spectra were recorded on a FT/IR-300E spectrometer.Mass spectra were recorded onMicromass Quatrro-II using electrospray Ionization technique, showing (m+1) peak as a basepeak. Analytical thin layer chromatography (TLC) was performed on Merck precoated TLC plates (silica gel 60 GF254, 0.25 mm). Other simple chemicals were purchased and used as such.

General procedure. Diethyl (2 –chloro-quinolin-3-yl) (hydroxy) methylphosphonate. (2a)

Amixture of2-chloroquinoline-3-carbaldehyde (0.95 gm, 3 mmol) and triethylphosphite (1.66 gm,6 mmol) all the reaction conditions are optimized usingChCl.2Urea mixture as catalyst. The reaction was added with constant stirring. Progress of reaction was monitored on TLC. Aftercompletion of reaction (25min.), the mixture was concentrated on rotary-evaporator underreduced pressure, to obtain an oily residue. The oily residue was dissolved in methanol. This methanolic solution was concentrated, dissolved indichloromethane and reprecipitated with hexane. Thus, obtained solid was filtered, washed withhexane and dried in oven at 40^{0}C. (Choline chloride–Urea-based DES preparation-The choline chloride–urea deep eutectic solvent was prepared according to the literature. In a 250-mL flask with constant magnetic stirring, Urea (300 mmol) and choline chloride (150 mmol) were mixed, and heated at 70 ^{0}C until a clear liquid appeared. The obtained deep eutectic solvent was used without any further purification).All of the a-hydroxyphosphonates is known compounds to which the spectroscopic data were compared.

(2a).diethyl ((2-chloroquinolin-3-yl)(hydroxy)methyl) phosphonate- (1.02 gm, yield 87%, m.p. 128-130^{0}C).IR (KBr), cm^{-1}: 3245 (- OH); 1218 (- P=O); 104(-P-O-C). ^{1}H NMR ($CDCl_3$), δ ppm: 1.2 (t, 3H,O-CH_2-CH_3); 1.4 (t, 3H, O-CH_2-CH_3); 2.1 (s, 1H, -CH-OH); 4.1 (m, 4H, O-CH_2-CH_3 and OCH_2-CH_3); 5.5 (d, 1H, -CH-P=O); 7.4 (t, 1H, Ar-H, C_6); 7.9 (t, 1H, Ar-H, C_7); 7.7 (d,1H, Ar-H,C_5); 8.1 (d, 1H, Ar-H, C_8); 8.5(s, 1H, Ar-H, C_4). ES-MS: m/z 330 (m+1) base peak and 332(m+3).Elemental analysis: $C_{14}H_{17}ClNO_4P$ Calculated: C: 51.01 %, H: 5.19%, N: 4.25 %; Found: C: 51.03%, H: 5.389 %, N: 4.355%.

(2b).diethyl ((2-chloro-6-methylquinolin-3-yl)(hydroxy)methyl) phosphonate-Yield 80.8 %, m.p. 145-147 ^{0}C. IR (KBr), cm^{-1}: 3278 (-OH); 1218 (-P=O); 1037 (-P-O-C). ^{1}H NMR ($CDCl_3$), δ ppm: 1.2 (t, 3H, O-CH_2-CH_3); 1.3 (t, 3H , O-CH_2-CH_3); 2.4 (s, 1H, -CH-OH); 2.5 (s, 3H , ArCH_3); 4.1 (q, 2H , O-CH_2-CH_3); 4.2 (q, 2H , O-CH_2-CH_3); 5.6 (d, 1H , CH-P=O); 7.5 (s, 1H , Ar-H, C_5); 7.6 (d, 1H , Ar-H, C_7); 7.9 (d, 1H , Ar-H, C_8); 8.5 (s, 1H , Ar-H, C_4). ES-MS: m/z 344 (m+1) base peak and 345.9 (m+3). Elemental analysis: $C_{15}H_{19}ClNO_4P$ Calca.: C: 52.41 %, H: 5.57 %, N: 4.07 %; Found: C: 52.50 %, H: 5.67 %, N: 4.17 %.

(2c).diethyl ((2-chloro-7-methylquinolin-3-yl) (hydroxy)methyl) phosphonate - Yield82.6 %, m.p. 141-143 ^{0}C. IR (KBr), cm^{-1}: 3278 (-OH); 1218 (-P=O); 1037 (-P-O-C). ^{1}H NMR ($CDCl_3$), δ ppm: 1.2 (t, 3H, O-CH_2-CH_3); 1.3 (t, 3H , O-CH_2-CH_3); 2.4 (s, 1H, -CH-OH); 2.5 (s, 3H , ArCH_3); 4.1 (q, 2H , O-CH_2-CH_3); 4.2 (q, 2H , O-CH_2-CH_3); 5.6 (d, 1H , CH-P=O); 7.5 (s, 1H , Ar-H, C_5); 7.6 (d, 1H , Ar-H, C_7); 7.9 (d, 1H , Ar-H, C_8); 8.5 (s, 1H , Ar-H, C_4). ES-MS: m/z 344 (m+1) base peak and 345.9 (m+3). Elemental analysis: $C_{15}H_{19}ClNO_4P$ Calca.: C: 52.41 %, H: 5.57 %, N: 4.07 %; Found: C: 52.50 %, H: 5.67 %, N: 4.17 %.

(2d).diethyl ((2-chloro-8-methylquinolin-3-yl)(hydroxy)methyl)phosphonate-Yield 82.5 %,m.p. 172-174^{0}C. IR (KBr) cm^{-1}: 3277 (-OH); 1222 (-P=O); 1037(-P-O-C).**),** δ ppm^{1}H NMR($CDCl_3$: 1.1 (t, 3H, O-CH_2-CH_3); 1.3 (t, 3H, O-CH_2-CH_3); 2.7 (s, 1H, -CH-OH); 3.9 (s, 3H, Ar-O-CH_3); 4.2 (q, 2H, O-CH_2-CH_3); 4.3 (q, 2H, O-CH_2-CH_3); 5.8 (d, 1H, CH-P=O); 7.0 (s, 1H, Ar-H, C_5); 7.5 (d, 1H, Ar-H, C_7); 8.1 (d, 1H, Ar-H, C_8); 8.8 (s, 1H, Ar-H, C_4). ES-MS: m/z 360(m+1) base peak and 362 (m+3).Elemental analysis: $C_{15}H_{19}ClNO_5P$ Calculated: C: 50.08 %, H: 5.32 %, N: 3.89 %; Found: C: 50.10%, H: 5.42 %, N: 3.99 %.

(2e). diethyl ((2-chloro-6-methoxyquinolin-3-yl) (hydroxy)methyl) phosphonate-Yield 84.1%,m.p. 156-160^{0}C. IR (KBr) cm-1: 3265 (-OH); 1219 (-P=O); 1034 (-P-O-C). 1H NMR ($CDCl_3$), δppm: 1.1(t, 3H, O-CH_2-CH_3); 1.3 (t, 3H, O-CH_2-CH_3); 2.7 (s, 1H, -CH-OH); 4.0 (s, 3H, Ar-OCH_3);4.3 (q, 2H, O-CH_2-CH_3); 4.4 (q, 2H, O-CH_2-CH_3); 5.7 (d, 1H, CH-P=O); 7.2 (d, 1H, Ar-H,C_6); 7.4 (s, 1H, Ar-H, C_8), 7.8 (d, 1H, Ar-H, C_5); 8.7 (s, 1H, Ar-H, C_4). ES-MS: m/z 343.9 (m+1) base peak and 346 (m+3).Elemental analysis: $C_{15}H_{19}ClNO_5P$ Calculated.: C: 50.08 %, H: 5.32 %, N: 3.89 %; Found: C: 50.27%, H: 5.52 %, N: 4.21 %.

(2f).diethyl ((2-chloro-7-methoxyquinolin-3-yl) (hydroxy)methyl) phosphonate-Yield 83.2 %,m.p. 166-170 ^{0}C. IR (KBr) cm-1: 3258 (-OH); 1235 (-P=O); 1049 (-P-O-C). 1H NMR (CDCl3), δppm: 1.2 (t, 3H, O-CH2-CH3); 1.4 (t, 3H, O-CH2-CH3); 1.3(t, 3H, Ar-O-CH2-CH3); 3.3 (bs, 1H,-CH-OH); 4.2 (q, 2H, O-CH2-CH3); 4.3 (q, 2H, O-CH2-CH3); 4.3 (q, 2H,, O-CH2-CH3); 5.5 (d,1H, CH-P=O); 7.2 (s, 1H, Ar-H, C5); 7.4 (d, 1H, Ar-H, C7); 7.7 (d, 1H, Ar-H, C8); 8.5 (s, 1H, Ar-H, C4). **ES-MS:** m/z 374 (m+1) base peak and 376 (m+3). Elemental analysis: $C_{16}H_{21}ClNO_5P$ Calculated.: C: 51.41 %, H: 5.66 %, N: 3.75 %; Found: C: 51.581 %, H: 5.772 %, N: 3.91 %.

(2g). diethyl ((2-chloro-8-methoxyquinolin-3-yl) (hydroxy)methyl) phosphonate-Yield 72.2 %,m.p. 149-151^{0}C. IR (KBr) cm-1: 3252 (-OH); 1223 (-P=O); 1041 (-P-O-C). 1H NMR (CDCl3), δppm: 1.2 (t, 3H, O-CH2-CH3); 1.35 (m, 6H, O-CH2-CH3 and Ar-CH2-CH3); 2.3 (s, 1H, -CHOH);3.25 (q, 2H, Ar-CH2-CH3); 4.2 (m, 4H, O-CH2-CH3 and O-CH2-CH3); 5.6 (d, 1H, CHP=O); 7.5 (t, 1H, Ar-H, C6); 7.6 (d, 1H, Ar-H, C7); 7.7 (d, 1H, Ar-H, C5); 8.5 (s,1H, Ar-H, C4).ES-MS: m/z 359 (m+1) base peak and 361 (m+3). Elemental analysis: $C_{15}H_{19}ClNO_5P$ Calculated.: C:53.71 %, H: 5.92 %, N: 3.92 %; Found: C: 53.82 %, H: 5.99 %, N: 4.05 %.

(2h).diethyl ((2-chloro-5-ethoxyquinolin-3-yl) (hydroxy)methyl) phosphonate Yield 85.9 %, m.p. 168-170 ^{0} C. IR (KBr) cm-1: 3255 (-OH); 1234 (-P=O); 1049 (-P-O-C). 1H NMR ($CDCl_3$), δ ppm: 1.2 (t, 3H, O-CH_2-CH_3); 1.3 (t, 3H, O-CH_2-CH_3); 1.4 (t, 3H, Ar-O-CH_2-CH_3); 3.4 (bs, 1H, -CH-OH); 4.1 (q, 2H, O-CH_2-CH_3); 4.2 (q, 2H, O-CH2-CH3); 4.3 (q, 2H,, O-CH2-CH3); 5.6 (d, 1H, CH-P=O); 7.0 (s, 1H, Ar-H,C5); 7.4 (d, 1H, Ar-H, C7); 7.9 (d, 1H, Ar-H, C8); 8.4 (s, 1H, ArH, C4). ES-MS: m/z 374 (m+1) base peak and 376 (m+3). Elemental analysis: $C_{16}H_{21}ClNO_5P$ Calcd.: C: 51.41 %, H: 5.66 %, N: 3.75 %; Found: C: 51.527 %, H: 5.793 %, N: 3.85 %.

IV. CONCLUSION

We have shown a facile and efficient procedure for the one pot synthesis of α-hydroxyphosphonates using environmentally ionic liquid. This method to avoids the use of acidic and highly toxic reagents as catalysts. In addition, operational simplicity, mild reaction conditions, considerably short reaction time, high yields, reusability of the catalyst, and cost effectiveness make this protocol an important addition to the existing methods of α-hydroxyphosphonates synthesis. Further work is in progress to broaden the scope of choline chloride-based moisture stable ionic liquids to the other organic transformations in a deep eutectic solvent. In addition to short reaction times, wide scope ofsubstrates, the use of biodegradable andinexpensive DESas solvent and catalyst are the distinct features of thisprocedure.

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