ISSN: 2249 –4820

Chemistry & Biology Interface, 2012, 2, 1, 31-37

Chemistry & Biology Interface

An official Journal of ISCB, Journal homepage; www.cbijournal.com

Research Paper

Solvent free and High yielding Synthesis of new ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates from ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate

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Received 24 December 2011; Accepted 7February 2012

Keywords: 2-chloroquinoline-3-carbaldehyde, Knoevenagel condensation, Michael addition, triethylphosphite, TMSCl.

Abstract: Solvent free, and high yielding synthesis of new ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates from ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate, obtained from 2-chloroquinolin-3-carbaldehydes by using triethylphosphite in the presence of TMSCl at room temperature.

Introduction

Quinolines [1] are an important class of heterocyclic compounds and possesses several biological activities such as bactericidal [2], antitumor [3], anti-inflammatory [4], antimalarial [5]. Quinolines such as 2-chloroquinoline-3-carbaldehyde occupy a prominent position as they are key intermediates for further annelation and for various functional group interconversions [6].

Organophosphorous compounds are important substrates in the study of biochemical processes [7] and are widely used as biologically active compounds. Phosphonates are versatile intermediates in organic synthesis due to their application in the Wadsworth-Emmons and related reactions [8]. In the last few years, phosphonates have been the focus of intensive studies due to their interest as stable transition state analogue enzyme inhibitors. In fact, the phosphonates and phosphonic acid moieties may be accepted by enzymes as false substrates and interfere with biological processes [9].

Owing to their synthetic and biological values, the chemistry of phosphonates has stimulated increasing interest and the development of new organophosphorous compounds and new methodologies for their preparation still remains of great interest [10].

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ISSN: 2249 –4820

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Simoni et al [11] reported the tetramethylguanidine catalyzed addition of dialkylphosphates to a variety of α,β-unsaturated compounds including carboxylic acid esters, ketones, and nitriles as well as, saturated aldehydes, ketones and imines. Wasielewski and coworkers [12] described the addition of sodium diethylphosphite to ethyl acrylate to give 3-phosphonopropionates. Chambers et al [13] reported the addition of dimethyl phosphonate to methyl N-acetyl-2-aminoacrylate, which was prepared by trimethylphosphite mediated esterification of the corresponding acid. Synthesis of the GABA-B antagonist, Phaclofen, which features a Michael addition of a phosphonates to β-nitrostyrene was reported by Hall [14]. Addition of H-P bond to olefins promoted by AIBN or base described by Zhao [15]. Tan and co-workers [16] showed TBD catalyzed P-C bond formation via the conjugate addition.

Knoevenagel condensation reactions have been extensively studied as an important carbon–carbon bond forming reaction. Generally, this reaction is catalyzed by a Lewis acid or base [17]. Herein we would like to describe 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) as an efficient catalyst for Knoevenagel condensation of 2-chloro-3-formyl substituted quinoline with ethyl cyanoacetate.

Literature survey revealed that condensation of ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate with triethylphosphite have not been reported. Herein we wish to report an efficient, environmentally benign method for the preparation of ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates

Results and Discussion

Ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate **(2a-h) (Scheme 1, Table I)** were synthesized by the Knoevenagel condensation of substituted 2-chloroquinoline-3-carbaldehyde and ethyl cyanoacetate using catalytic amount of DBU under solvent free condition in excellent yields. The products were characterized by physical and spectroscopic data. In ^{1}H NMR of compound Ethyl 3-(2-chloro-8-methylquinolin-3-yl)-2-cyanoacrylate **(2d)** showed the signal at 8.76 δ ppm for -CH=C(COOEt) (CN) and also in IR spectral, the values 2221 cm^{-1} (-C≡N) and 1722 cm^{-1} (-COOEt) for nitrile (-C≡N) and α,β-unsaturated ester -CH=C(COOEt).

Ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates **(3a-h) (Scheme 1, Table II)** were then prepared in excellent yields by reacting Ethyl-3-(2-chloroquinolin-3-yl)-2-cyanoacrylate **(2a-h)** with triethylphosphite in the presence of TMSCl without solvent at room temperature. Michael addition product has been confirmed by spectral analysis (IR, NMR and Mass). In ^{1}H NMR of compound Ethyl 2-(ethoxyphosphono)-2-(2-chloro-8-methylquinolin-3-yl)-1-cyanoethanoates **(3d)** 4.40 δ ppm for –CH-CH(COOEt) (CN) and also in IR spectral, the values 2257 cm^{-1} (-C≡N) and 1744 cm^{-1} (-COOEt) for nitrile (-C≡N) and ester –CH-CH(COOEt).

Conclusion

In conclusion, a new methodology was developed for the synthesis of novel ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoate derivatives **(3a-h)** from ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate **(2a-h)**, obtained from 2-chloroquinolin-3-carbaldehydes **(1a-h)** by using triethylphosphite in the presence of chlorotrimethylsilane at room temperature in high yields**.** All the reactions were performed under mild reaction conditions,

ISSN: 2249 –4820
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shorter reaction time and in high yields (Table II). The methodology developed will be of much use to combinatorial chemist.

Experimental Section

2-chloroquinoline-3-carbaldehydes were prepared in the laboratory by the reported method, triethylphosphite, chlorotrimethylsilane were procured from Lancaster, methylene chloride, methanol and hexane were procured from S. D. Fine-Chem. All physical constant were determined in open capillaries at atmospheric pressure. The products were characterized by their spectral data. ^{1}H NMR spectra were recorded on Varian Gemini in $CDCl_3$ at 400 MHz using TMS as an internal standard. IR spectra were recorded on a Perkin-Elmer FTIR using KBr discs. Mass spectra were recorded on Micromass Quatrro-II using electrospray Ionization technique, showing (m+1) peak as a molecular ion peak. The test for the purity of products and the progress of the reactions were accomplished by TLC on Merck silica gel plates.

Experimental procedure

Synthesis of Ethyl 3-(2-chloro-8-methylquinolin-3-yl)-2-cyanoacrylate (2d)

To the stirred solution of 2-chloro-8-methylquinolin-3-carbaldehyde (1.02 gm, 5 mmol) and ethyl cyano acetate (0.6 gm, 5.2 mmol) was added DBU (2 to 3 drops) at room temperature. The progress of reaction was monitored by the TLC (solvent system-hexane: ethyl acetate). After the completion of the reaction (10 min), reaction mixture was dissolved in 10 ml of ethanol and was added 30 ml of cold water. The obtained solid was filtered and washed with water, dried under vacuum (1.37 gm, 92%).

Synthesis of Ethyl 2-(ethoxyphosphono)-2-(2-chloro-8-methylquinolin-3-yl)-1-cyanoethanoates (3d)

To a mixture of Ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylate (1.0 gm, 3.3 mmol) and triethylphosphite (1.66 gm, 10 mmol) was added TMSCl (1.08 gm, 10 mmol) and was stirred at room temperature for 45-60 min. The progress of the reaction was monitored by the TLC using hexane: ethyl acetate (8:2) as the solvent system. After the completion of the reaction, the reaction mixture was dissolved in methanol and was concentrated. To the concentrated mass methylene chloride was added and was washed with sodium thiosulphate solution. Methylene chloride layer was dried over sodium sulphate and the solvent was removed under reduced pressure to obtain the crude product. The crude product was purified by column chromatography using hexane: ethyl acetate (7:3) as an eluent to afford the pure compound (1.24 gm, 85%).

Spectral Data

Ethyl 3-(2-chloro-8-methylquinolin-3-yl)-2-cyanoacrylate (2d)
IR (KBr, cm^{-1}): 2221 (-C≡N); 1722 (-C=O)
^{1}H NMR ($CDCl_3$, δ ppm): 1.41 (t, 3H, O-CH_2-C<u>H</u>$_3$, *J* = 8 Hz); 2.77 (s, 3H, Ar-C<u>H</u>$_3$); 4.41 (q, 2H, O-C<u>H</u>$_2$-CH_3, *J* = 8 Hz); 7.51 (t, 1H, Ar-<u>H</u>, C_6, *J* = 8 Hz); 7.69 (d, 1H, Ar-<u>H</u>, C_7, *J* = 8 Hz; 7.79 (d,1H, Ar-<u>H</u>, C_5, *J* = 8 Hz); 8.76 (s, 1H, -C<u>H</u>=C), 9.01 (s, 1H, Ar-<u>H</u>, C_4).
ES-MS: m/z 301.2 (m+1) and 303.2 (m+3).
Ethyl 3-(2-chloro-6-ethoxyquinolin-3-yl)-2-cyanoacrylate (2g)
IR (KBr, cm^{-1}): 2236 (-C≡N); 1732 (-C=O)
^{1}H NMR ($CDCl_3$, δ ppm): 1.30 (t, 3H, O-CH_2-C<u>H</u>$_3$, *J* = 8 Hz); 1.37 (t, 3H, Ar-O-CH_2-C<u>H</u>$_3$, *J* = 8 Hz); 4.17 (q, 2H, Ar-O-C<u>H</u>$_2$-CH_3, *J* = 8 Hz); 4.33 (q, 2H, O-C<u>H</u>$_2$-CH_3, *J* = 8 Hz); 7.54 (s, 1H, Ar-<u>H</u>, C_5); 7.56 (d, 1H, Ar-<u>H</u>, C_7, *J* = 8 Hz; 7.89 (d,1H, Ar-<u>H</u>, C_8, *J* = 8 Hz); 8.54 (s, 1H, -C<u>H</u>=C), 8.91 (s, 1H, Ar-<u>H</u>, C_4).
ES-MS: m/z 331.1 (m+1) and 333.1 (m+3).

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Ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates (3a)

IR (KBr, cm^{-1}): 2252 (-C≡N); 1748 (-C=O); 1235 (-P=O); 1032 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.10 – 1.22 (m, 6H, (O-CH$_2$-C<u>H</u>$_3$)$_2$); 1.28 (t, 3H, -COOCH$_2$-C<u>H</u>$_3$, *J* = 8 Hz); 4.02 – 4.18 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.36 (d, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.65 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.42 (t, 1H, Ar-<u>H</u>, C$_6$, *J* = 8 Hz); 7.54 (t, 1H, Ar-<u>H</u>, C$_7$, *J* = 4 Hz; 7.68 (d,1H, Ar-<u>H</u>, C$_5$, *J* = 8 Hz); 8.04 (d, 1H, Ar-<u>H</u>, C$_8$, *J* = 8 Hz); 8.69 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 425.1 (m+1) and 427.1 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-6-methylquinolin-3-yl)-1-cyanoethanoates (3b)

IR (KBr, cm^{-1}): 2254 (-C≡N); 1746 (-C=O); 1238 (-P=O); 1030 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.10 – 1.34 (m, 12H, (O-CH$_2$-C<u>H</u>$_3$)$_2$; -COOCH$_2$-C<u>H</u>$_3$; Ar-CH$_2$C<u>H</u>$_3$); 2.50 (q, 2H, Ar-C<u>H</u>$_2$CH$_3$); 4.05 – 4.27 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.45 (t, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.68 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.56 (d, 1H, Ar-<u>H</u>, C$_7$, *J* = 8 Hz); 7.63 (s, 1H, Ar-<u>H</u>, C$_5$); 7.85 (d,1H, Ar-<u>H</u>, C$_8$, *J* = 8 Hz); 8.60 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 439.1 (m+1) and 441.1 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-7-methylquinolin-3-yl)-1-cyanoethanoates (3c)

IR (KBr, cm^{-1}): 2255 (-C≡N); 1748 (-C=O); 1240 (-P=O); 1025 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.12 – 1.24 (m, 6H, (O-CH$_2$-C<u>H</u>$_3$)$_2$); 1.29 (t, 3H, -COOCH$_2$-C<u>H</u>$_3$, *J* = 8 Hz); 2.58 (s, 3H, Ar-C<u>H</u>$_3$); 4.02 – 4.25 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.42 (t, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.66 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.34 (d, 1H, Ar-<u>H</u>, C$_6$, *J* = 8 Hz); 7.62 (d, 1H, Ar-<u>H</u>, C$_5$, *J* = 4 Hz; 7.91 (s,1H, Ar-<u>H</u>, C$_8$, *J* = 8 Hz); 8.65 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 439.0 (m+1) and 441.1 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-8-methylquinolin-3-yl)-1-cyanoethanoates (3d)

IR (KBr, cm^{-1}): 2257 (-C≡N); 1744 (-C=O); 1241 (-P=O); 1028 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.18 – 1.26 (m, 6H, (O-CH$_2$-C<u>H</u>$_3$)$_2$); 1.30 (t, 3H, -COOCH$_2$-C<u>H</u>$_3$, *J* = 8 Hz); 2.74 (s, 3H, Ar-C<u>H</u>$_3$); 4.06 – 4.20 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.40 (t, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.69 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.45 (t, 1H, Ar-<u>H</u>, C$_6$, *J* = 8 Hz); 7.59 (d, 1H, Ar-<u>H</u>, C$_7$, *J* = 4 Hz; 7.71 (d,1H, Ar-<u>H</u>, C$_5$, *J* = 8 Hz); 8.69 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 439.2 (m+1) and 441.2 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-6-methoxyquinolin-3-yl)-1-cyanoethanoates (3e)

IR (KBr, cm^{-1}): 2254 (-C≡N); 1745 (-C=O); 1236 (-P=O); 1026 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.07 – 1.26 (m, 6H, (O-CH$_2$-C<u>H</u>$_3$)$_2$); 1.29 (t, 3H, -COOCH$_2$-C<u>H</u>$_3$, *J* = 8 Hz); 3.85 (s, 3H, Ar-OC<u>H</u>$_3$); 4.04 – 4.16 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.44 (d, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.96 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.37 (s, 1H, Ar-<u>H</u>, C$_5$); 7.62 (d, 1H, Ar-<u>H</u>, C$_7$, *J* = 8 Hz; 7.85 (d,1H, Ar-<u>H</u>, C$_8$, *J* = 8 Hz); 8.61 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 455.1 (m+1) and 457.1 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-7-methoxyquinolin-3-yl)-1-cyanoethanoates (3f)

IR (KBr, cm^{-1}): 2250 (-C≡N); 1748 (-C=O); 1232 (-P=O); 1024 (-P-O-C).

^{1}H NMR (CDCl$_3$, δ ppm): 1.02 – 1.16 (m, 6H, (O-CH$_2$-C<u>H</u>$_3$)$_2$); 1.26 (t, 3H, -COOCH$_2$-C<u>H</u>$_3$, *J* = 8 Hz); 3.87 (s, 3H, Ar-OC<u>H</u>$_3$); 3.99 – 4.23 (m, 6H, (O-C<u>H</u>$_2$-CH$_3$)$_2$, -COOC<u>H</u>$_2$-CH$_3$); 4.35 (d, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.55 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.14 (d, 1H, Ar-<u>H</u>, C$_6$, *J* = 4 Hz); 7.25 (s, 1H, Ar-<u>H</u>, C$_8$); 7.65 (d,1H, Ar-<u>H</u>, C$_5$, *J* = 8 Hz); 8.58 (d, 1H, Ar-<u>H</u>, C$_4$, *J* = 4 Hz).

ES-MS: m/z 455.2 (m+1) and 457.2 (m+3).

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Ethyl 2-(ethoxyphosphono)-2-(2-chloro-6-ethoxyquinolin-3-yl)-1-cyanoethanoates (3g)

IR (KBr): 2248 cm^{-1} (-C≡N); 1749 cm^{-1} (-C=O); 1237 cm^{-1} (-P=O); 1046 cm^{-1} (-P-O-C).

^{1}H NMR ($CDCl_3$, δ ppm): 1.10 – 1.24 (m, 6H, (O-CH_2-C<u>H</u>$_3$)$_2$); 1.31 (t, 3H, -COOCH_2-C<u>H</u>$_3$, *J* = 8 Hz); 1.47 (t, 3H, Ar-OCH_2-C<u>H</u>$_3$, *J* = 8 Hz); 4.04 – 4.22 (m, 8H, (O-C<u>H</u>$_2$-CH_3)$_2$, -COOC<u>H</u>$_2$-CH_3, Ar-OCH_2-C<u>H</u>$_3$); 4.40 (q, 1H, -CH-C<u>H</u>-CN, *J* = 4 Hz); 4.66 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.12 (d, 1H, Ar-<u>H</u>, C_5, *J* = 4 Hz); 7.37 (dd, 1H, Ar-<u>H</u>, C_7, *J* = 4 Hz and 8 Hz); 7.86 (d,1H, Ar-<u>H</u>, C_8, *J* = 8 Hz); 8.63 (d, 1H, Ar-<u>H</u>, C_4, *J* = 4 Hz).

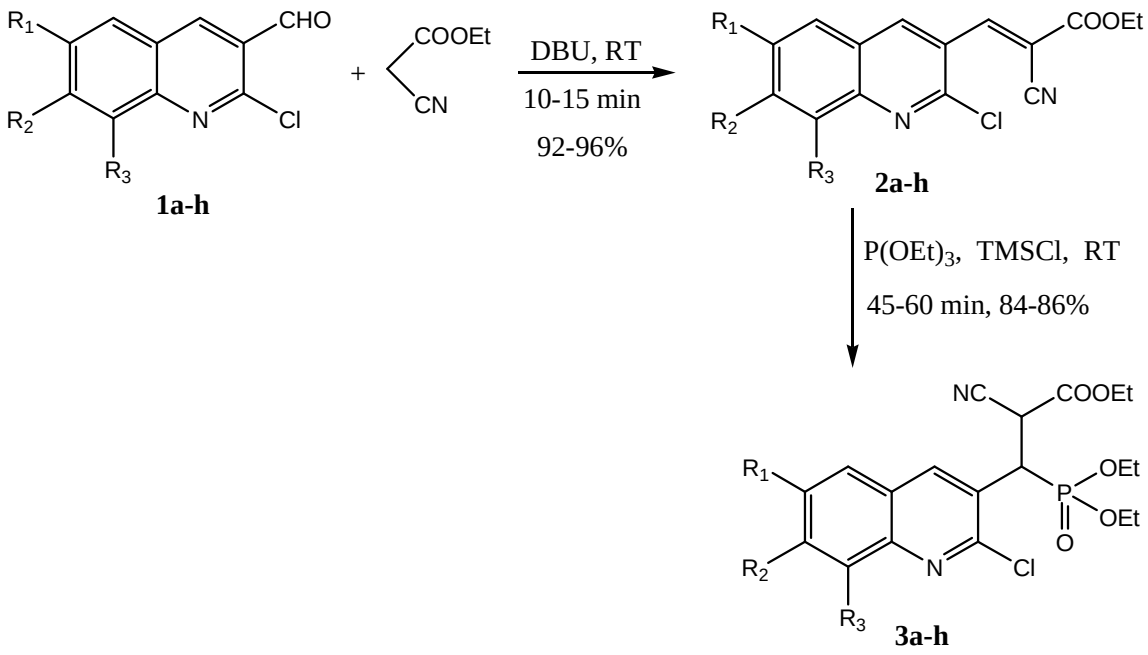
ES-MS: m/z 469.2 (m+1) and 471.2 (m+3).

Ethyl 2-(ethoxyphosphono)-2-(2-chloro-8-ethylquinolin-3-yl)-1-cyanoethanoates (3h)

IR (KBr, cm^{-1}): 2255 (-C≡N); 1746 (-C=O); 1230 (-P=O); 1028 (-P-O-C).

^{1}H NMR ($CDCl_3$, δ ppm): 1.06 – 1.16 (m, 6H, (O-CH_2-C<u>H</u>$_3$)$_2$); 1.22 – 1.36 (m, 6H, -COOCH_2-C<u>H</u>$_3$ and Ar-CH_2C<u>H</u>$_3$); 3.63 (q, 2H, Ar-C<u>H</u>$_2$$CH_3$); 4.09 – 4.26 (m, 6H, (O-C<u>H</u>$_2$-CH_3)$_2$, -COOC<u>H</u>$_2$-CH_3); 4.41 (d, 1H, -CH-C<u>H</u>-CN, *J* = 8 Hz); 4.67 (dd, 1H, -CH-C<u>H</u>-P=O, *J* = 4 Hz); 7.21 (t, 1H, Ar-<u>H</u>, C_6, *J* = 8 Hz); 7.38 d, 1H, Ar-<u>H</u>, C_7, *J* = 8 Hz); 7.53 (d,1H, Ar-<u>H</u>, C_5, *J* = 8 Hz); 8.65 (d, 1H, Ar-<u>H</u>, C_4, *J* = 4 Hz).

ES-MS: m/z 455.2 (m+1) and 457.2 (m+3).



Scheme 1

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Table I

Characterization data of Ethyl 3-(2-chloroquinolin-3-yl)-2-cyanoacrylates

Entry	R_1	R_2	R_3	Time (min)	Yield (%)	Melting Point (°C)
2a	H	H	H	10	94	160-162
2b	CH_3	H	H	15	95	150-152
2c	H	CH_3	H	10	94	146-148
2d	H	H	CH_3	10	92	162-164
2e	OCH_3	H	H	15	96	144-146
2f	H	OCH_3	H	15	94	155-157
2g	OC_2H_5	H	H	10	95	168-170
2h	H	H	C_2H_5	15	93	165-167

Table II

Characterization data of Ethyl 2-(ethoxyphosphono)-2-(2-chloroquinolin-3-yl)-1-cyanoethanoates

Entry	R_1	R_2	R_3	Time (min)	Yield (%)	MP/BP (°C)
3a	H	H	H	50	85	122-124 (Liq)
3b	CH_3	H	H	45	84	120-122 (Liq)
3c	H	CH_3	H	45	85	140-142 (Liq)
3d	H	H	CH_3	50	85	110-112
3e	OCH_3	H	H	60	86	238-240 (Liq)
3f	H	OCH_3	H	50	84	80-82 (Liq)
3g	OC_2H_5	H	H	45	84	118-120
3h	H	H	C_2H_5	50	86	140-142 (Liq)

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