Hany M. Hassanin, Magdy A. Ibrahim,* Yassin A. Gabr,
and Youssif A. Alnamer

Department of Chemistry, Faculty of Education, Ain Shams University, Roxy 11711, Cairo, Egypt
*E-mail: magdy_ahmed1977@yahoo.com
Received February 4, 2011
DOI 10.1002/jhet.906
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The present review covers the methods developed for the synthesis of different pyrano[3,2-*c*]quinolinones as well as the chemical reactivity of these compounds towards various types of chemical reactions.

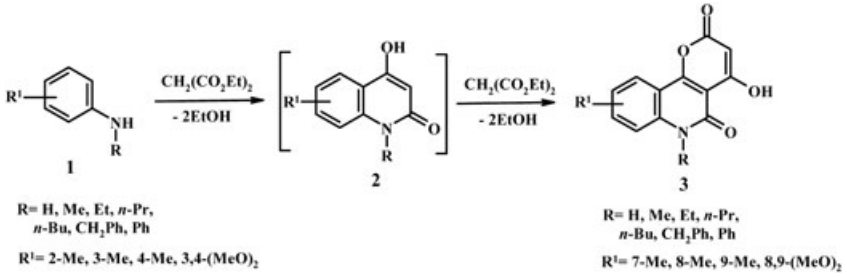
J. Heterocyclic Chem., **49**, 1269 (2012).

Contents

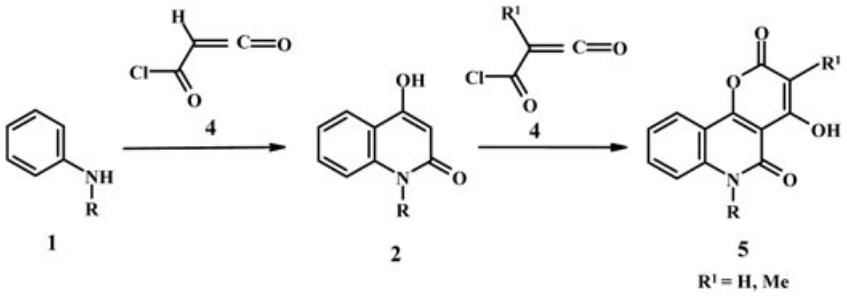
1. INTRODUCTION

Pyranoquinolinones constitute the parent structure of pyranoquinoline alkaloids which occur in the plant family Rutaceae. The synthesis of pyranoquinolinones and their derivatives is of great interest in organic chemistry because these compounds exhibit antibacterial activity [1,2], anticoagulant [3], antitumor [4], antihypertensive agents [5], possess moderate acetyl cholinesterase inhibitory activity [6,7], potent, and selective inhibitors of the mitotic kinesin-5 and potently inhibiting the ATPase activity of kinesin-5 [8]. Pyrano[3,2-*c*]quinolinones are good synthones for substituted quinolinones which have been of increasing interest since many of these compounds have found useful applications as chemotherapeutic agents against malaria parasites and microbes [9–13] antimicrobial [14,15], antibacterial [16,17], antiproliferative and antitubulin [18], antiamebics and antischistosomal agents [19]. They are also useful intermediates in the manufacture of azo dyestuffs, these azo-dyes are used for dyeing both synthetic and naturally occurring fibers [20]. Furthermore, pyrano[3,2-*c*] quinolinones constitute a large group of naturally occurring alkaloids, of these flindersine, 2,2-dimethylpyrano[3,2-*c*]quinolin-5(2*H*)-one, various substituted flindersine and pyrano[3,2-*c*]quinolin-5(6*H*)-one derivatives that are widely distributed in nature [21–25]. On the basis of the above importance, the present review aims to study the chemistry of pyrano[3,2-*c*]quinolinones as an important family in field of organic chemistry.

Scheme 1



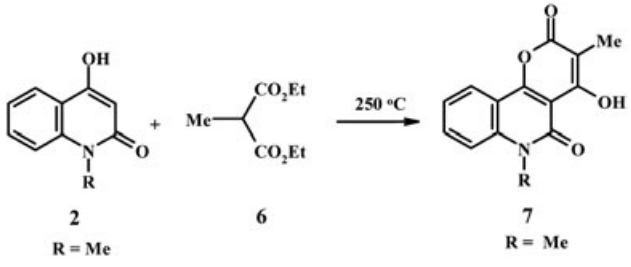
Scheme 2



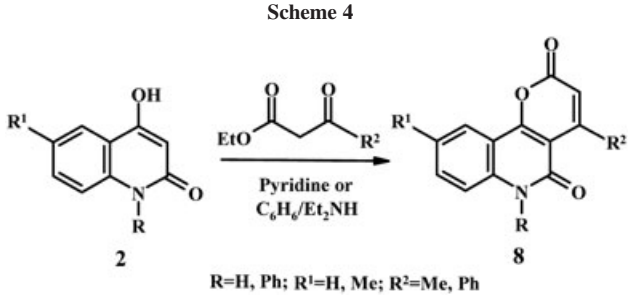
2. SYNTHESIS OF PYRANO[3,2-*c*]QUINOLINONES

2.1. From substituted aniline. Aniline and its derivatives are good precursors for the synthesis of pyrano[3,2-*c*] quinoline-2,5(6*H*)-dione derivatives. Heating aniline and its derivatives **1** (R=H) with diethyl malonate in a 1:2 ratio under fusion conditions at 250°C led to 4-hydroxyquinolin-2 (1*H*)-ones **2** as a nonisolable intermediates, which underwent another condensation with diethyl malonate to produce 4-hydroxypyrano[3,2-*c*] quinoline-2,5(6*H*)-diones **3** [26] (Scheme 1). Under the same conditions, 4-hydroxy-6-substituted-pyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **3** (R=alkyl or Ph) were prepared by condensation of the proper *N*-alkylanilines **1** (R=alkyl) or diphenylamine **1** (R= Ph) with diethyl malonate under reflux without solvent as shown in Scheme 1 [27–30]. This reaction was firstly described by Bowman *et al.* [31] and Bosson *et al.* [32] using *N*-methylaniline as the starting material and the method has been improved considerably by Roschger [33,34]. Compounds **3** (R=Me) were also prepared from the condensation of *N*-methylaniline (**1**) with two equivalent of diethyl malonate in boiling diphenyl ether [35,36] or under microwave irradiation [36].

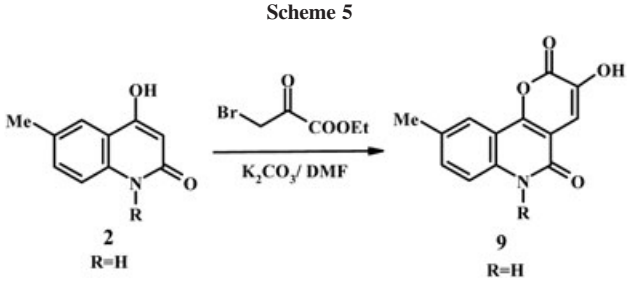
Scheme 3



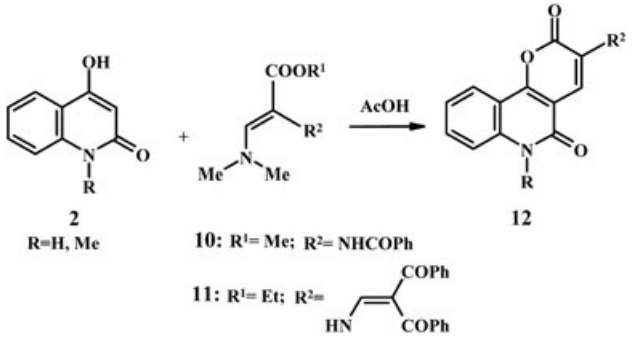
Scheme 4



Scheme 5



Scheme 6



Also, 3-substituted-pyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **5** were prepared in good to excellent yields from the reaction of *N*-alkylaniline **1** (R=alkyl) with chlorocarbonylketene **4** (R^1=H) to produce 4-hydroxyquinolin-2(1*H*)-ones **2** which on further treatment with compound **4** (R^1=H, alkyl) produced the target compounds **5** [37] (Scheme 2).

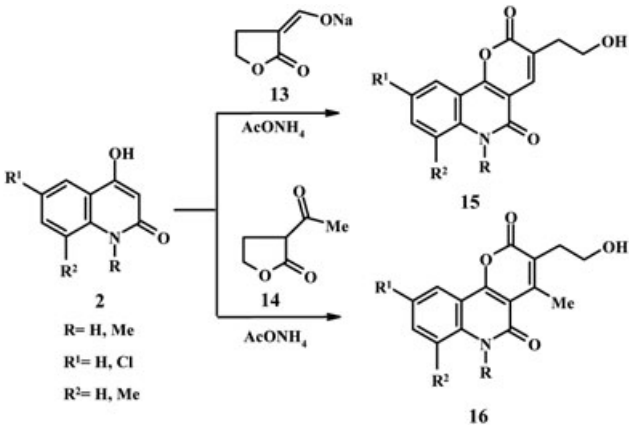
2.2. From quinoline derivatives.

2.2.1. From 4-hydroxyquinolines. 4-Hydroxyquinolin-2(1*H*)-ones possesses two nucleophilic centers (C-3 and the 4-OH group) and act as 1,3-dinucleophiles and their reactions with various 1,3-dielectrophiles represent one of the most important routes to pyrano[3,2-*c*] quinolinones. Thus, condensation of 4-hydroxy-6-methylquinolin-2(1*H*)-one **2** (R=Me) with diethyl methylpropanedioate **6** gave 3,6-dimethyl-4-hydroxypyrano [3,2-*c*] quinoline-2,5(6*H*)-dione (**7**) [21] (Scheme 3).

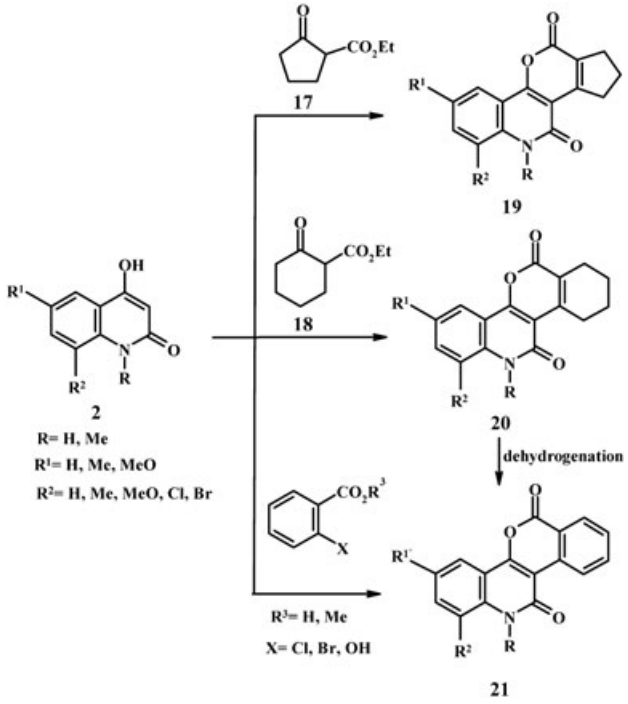
Treatment of 4-hydroxyquinolin-2(1*H*)-ones **2** with ethyl acetoacetate and/or ethyl benzoylacetate, in pyridine or benzene/diethylamine, produced 4-substituted-pyrano [3,2-*c*]quinoline-2,5(6*H*)-diones **8** (Scheme 4) [38–40].

Cyclization of 4-hydroxy-6-methylquinolin-2(1*H*)-one (**2**) with ethyl bromopyruvate, in DMF in the presence of potassium carbonate, gave 3-hydroxy-9-methylpyrano [3,2-*c*]quinoline-2,5(6*H*)-dione (**9**) [41] (Scheme 5).

Scheme 7

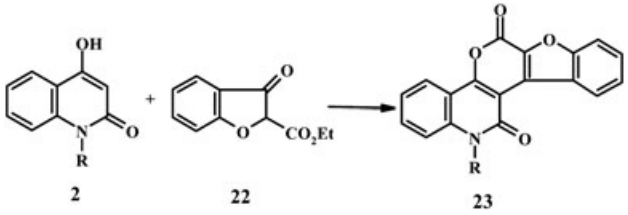


Scheme 8

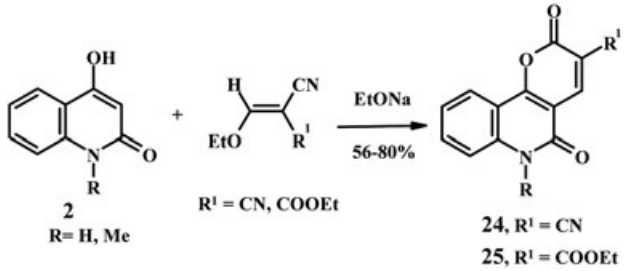


Cyclo-condensation of **2** with methyl 2-(benzoylamino)-3-(dimethylamino) propenate (**10**) and/or ethyl 2-[(2,2-dibenzoyl)ethenyl]amino-3-dimethylamino propenate (**11**) in boiling acetic acid gave 3-(benzoylamino)/3-{(2,2-dibenzoyl) ethenyl] amino} pyrano[3,2-*c*]quinoline-2,5 (6*H*)-diones **12** (Scheme 6) [42,43].

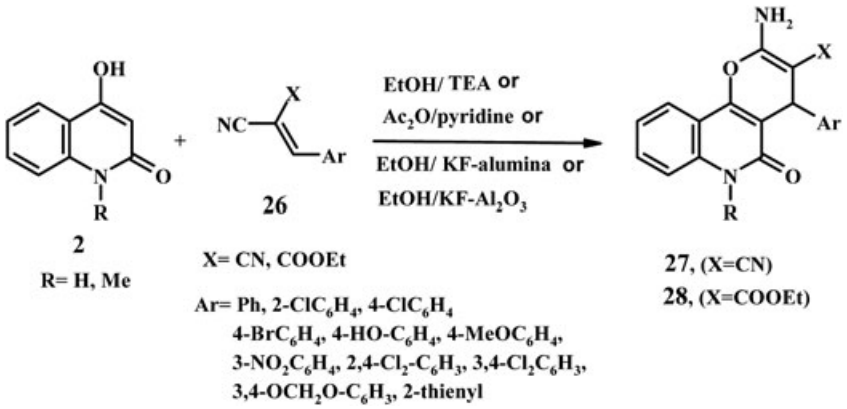
Scheme 9



Scheme 10



Scheme 11



Reaction of 4-hydroxyquinolin-2(1*H*)-ones **2** with α-formyl-γ-butyrolactone sodium salt (**13**) and α-acetyl-γ-butyrolactone (**14**) in ammonium acetate at 120°C gave 3-(2-hydroxyethyl)pyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **15** and **16**, respectively [44] (Scheme 7).

Also, condensation of **2** with ethyl cyclopentanone-2-carboxylate (**17**) and ethyl cyclohexanone-2-carboxylate (**18**) gave cyclopenta-pyrano[4,3-*c*]quinoline-6,10(11*H*)-diones **19** and tetrahydro-6*H*-[2]benzopyrano[4,3-*c*]quinoline-6,11(12*H*)-diones **20**, respectively. Dehydrogenation of **20** gave 6*H*-[2]benzopyrano[4,3-*c*] quinoline-6,11 (12*H*)-diones **21**. Compounds **21** were confirmed by an alternate synthesis *via* condensation of **2** with methyl benzoate or 2-halobenzoic acid, in an alkaline medium using copper sulfate as a catalyst [45,46] (Scheme 8).

Cyclo-condensation of compounds **2** with ethyl-2,3-dihydro-3-oxo-benzofuran-2-carboxylate (**22**) afforded the penta-cyclic systems identified as benzo[4′,5′]furo [2′,3′:5,4]pyrano[3,2-*c*]quinoline-6,12(5*H*)-diones **23** [47] (Scheme 9).

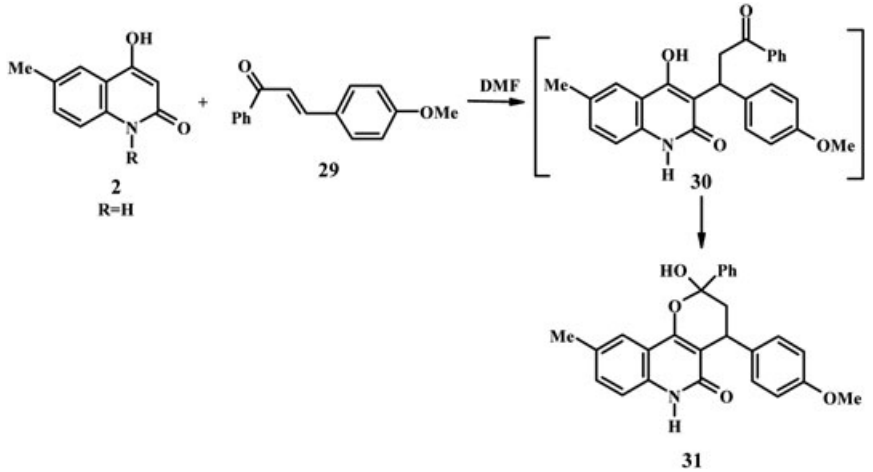
Schmidt and Junek [48] reported the formation of pyrano[3,2-*c*]quinoline-3-carbonitrile **24** (R^1=CN) and ethyl pyrano[3,2-*c*]quinoline-3-carboxylate **25** (R^1=COOEt) from the cyclo-condensation of **2** (R=H, Me) with ethoxymethylene malononitrile and ethoxymethylene ethyl cyanoacetate, respectively (Scheme 10).

4-Hydroxyquinolin-2(1*H*)-ones **2** reacted with arylidene-malononitrile **26** (X=CN) and arylidene-cyanoacetate **26** (X=COOEt), in different solvents (EtOH/TEA, Ac_2O/pyridine, EtOH/KF-Al_2O_3 or EtOH/KF-alumina), to afford 2-amino-4-arylpyrano[3,2-*c*]quinolin-5(6*H*)-ones **27** (X=CN) and **28** (X=COOEt), respectively, in slightly high yields [19,49–51] (Scheme 11).

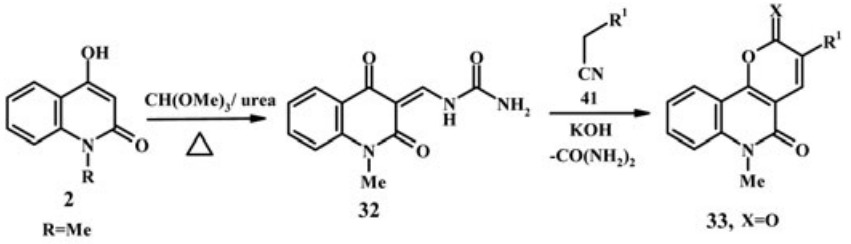
Cyclo-addition reaction takes place when compound **2** was refluxed in DMF with *p*-methoxybenzylideneacetophenone (**29**) to produce 4-(*p*-anisyl)-2-hydroxy-9-methyl-2-phenyl-2,3,4-trihydropyrano[3,2-*c*]quinolin-5(6*H*)-one (**31**) in 43% yield, *via* the nonisolable intermediate **30** [40] (Scheme 12).

Treating 4-hydroxy-1-methylquinolin-2(1*H*)-one (**2**) with trimethylortho- formate and urea gave 1-methyl-3-

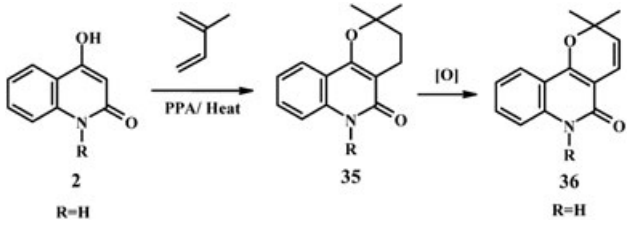
Scheme 12



Scheme 13



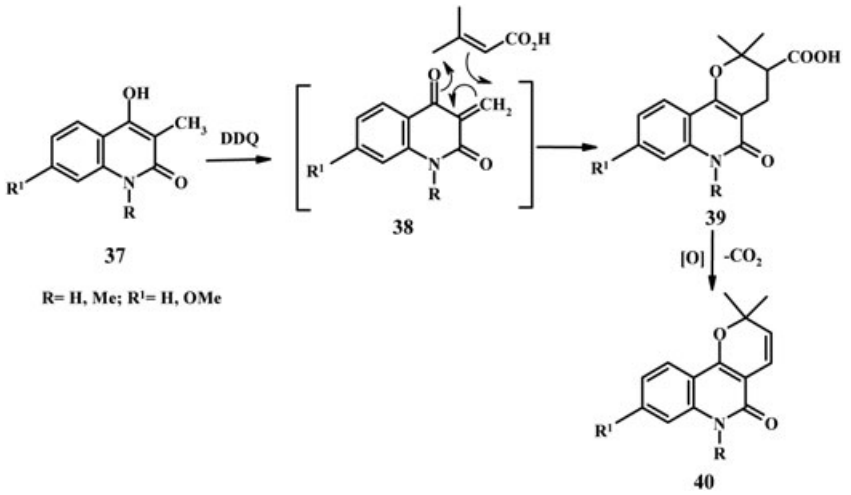
Scheme 14



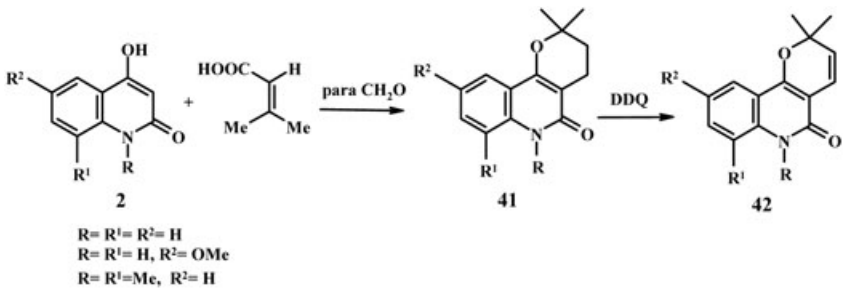
ureidomethylene-1,2,3,4-tetrahydroquinoline-2,4-dione (**32**). Condensation of **32** with acetonitrile derivatives in potassium hydroxide gave 3-substituted-6-methylpyrano[3,2-*c*]quinoline-2,5-(6*H*)dione (**33**) as published by Bratulescu [52] or its corresponding 2-imino derivative **34** [53] (Scheme 13).

Thangavel *et al.* found that, treating **2** with isoprene, in the presence of polyphosphoric acid (PPA), furnished dihydroflindersine **35** in good yield, which on

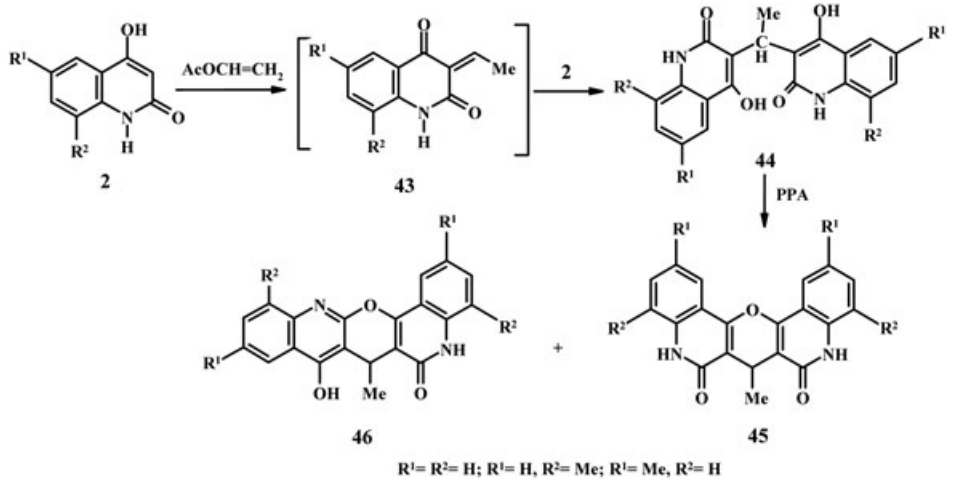
Scheme 15



Scheme 16



Scheme 17



dehydrogenation led to new synthetic route of flindersine **36** [54] (Scheme 14).

Treatment of 4-hydroxy-3-methylquinolin-2(1*H*)-ones **37** with 3-methyl-2-butenoic acid, in the presence of 2,3-dichloro-5,6-dicyano-ρ-benzoquinone (DDQ), gave the corresponding 2,2-dimethylpyrano[3,2-*c*]quinolin-5(6*H*)-ones **39** *via* the nonisolable enone intermediates **38** which were formed, in situ, and underwent cycloaddition with 3-methyl-2-butenoic acid. The cyclization was accomplished by decarboxylation and oxidation to give compounds **40** as depicted in Scheme 15 [55].

Also, pyranoquinoline based alkaloids like flindersine **41** (R=R^1=R^2=H), heplamine **41** (R=R^1=Me, R^2=OMe) and *N*-methylflindersine **41** (R=Me, R^1=R^2=H) were obtained from respective reaction of 4-hydroxyquinolin-2(1*H*)-ones **2** with dimethylacrylic acid and para-formaldehyde through generation of enone intermediates and subjecting [4+2] cycloaddition. Further, the dihydro intermediates **41** were oxidized by DDQ yielding the corresponding oxidized products **42** [56] (Scheme 16).

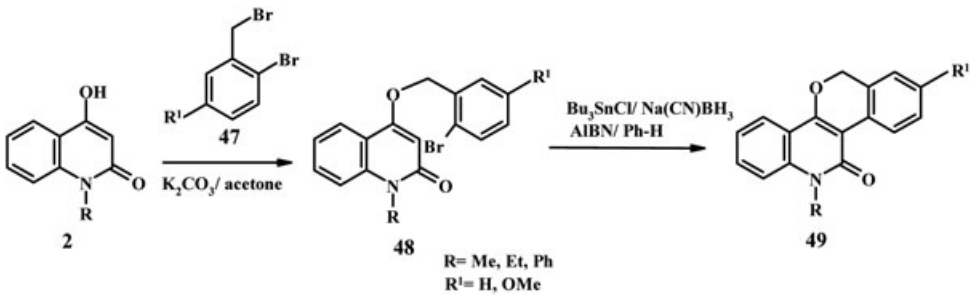
Reaction of 4-hydroxyquinolin-2(1*H*)-ones **2** with vinyl acetate led to the formation of 1,1-diquinolinylethane derivatives **44**, *via* enone intermediates **43**, which underwent dehydration by PPA yielding two isomeric pyranodiquinolines; 7-methyl-5,6,7,8,9-pentahydropyrano[3,2-*c*:5,6-*c*]diquinoline-6,8-diones **45** and 8-hydroxy-7-methyl-5,6,7-trihydropyrano[2,3-*b*:5,6-*c*]diquionlin-6-ones **46** [57] (Scheme 17).

Majumdar and Mukhopadhyay [58] reported the formation of 4-(2-bromobenzyloxy)quinolin-2(1*H*)-ones **48** in 80–85% yields by the reaction of 2-bromo benzyl bromides **47** with 4-hydroxyquionlin-2(1*H*)-ones **2**, in refluxing acetone in the presence of anhydrous potassium carbonate. The benzyloxyquinolinones **48** when refluxed, in benzene containing tributyltin(IV) chloride and sodium borohydride in the presence of azoisobutyronitrile (AIBN), gave the tetracyclic systems identified as 8-substituted-2*H*-[2]benzopyrano[4,3-*c*]quinolin-7(8*H*)-ones **49** (Scheme 18).

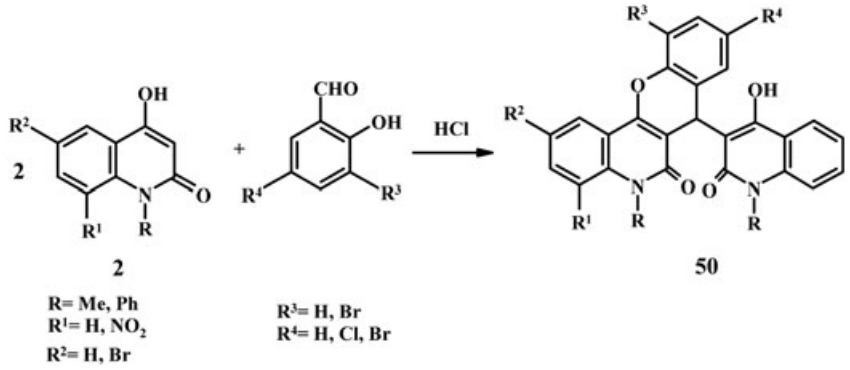
Synthesis of 7-(1,2-dihydro-4-hydroxy-2-oxo-3-quinolinyl)-5,7-dihydro-6*H*-[1]benzopyrano[3,2-*c*]quinolin-6-ones **50** was achieved by treating 4-hydroxyquinolin-2(1*H*)-ones **2** with salicylaldehyde derivatives in the presence of hydrochloric acid [59] (Scheme 19).

An efficient synthesis of 2,2-disubstituted-2*H*-pyrano [3,2-*c*]quinolin-5(6*H*)-ones **52** were achieved by ytterium

Scheme 18



Scheme 19



(III) triflate catalyzed reaction of **2** with α,β-unsaturated aldehydes **51** in acetonitrile under reflux for 12 h [60]. Also, the same reaction could be catalyzed by indium(III) trichloride in acetonitrile for only 4 h [61] (Scheme 20).

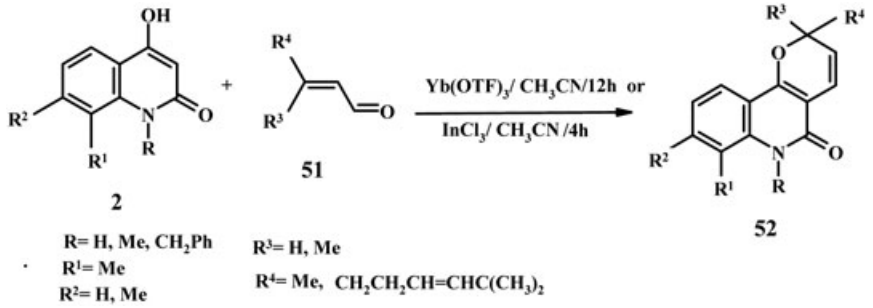
Phase-transfer catalyzed reaction of 4-hydroxyquinolin-2(1*H*)-one **2** with 3-chloro-3-methylbut-1-yne (**53**) gave pyrano[3,2-*c*]quinoline **42**, beside furo[3,2-*c*] quinolinone **54** as by-product [62] (Scheme 21).

When 3-prenyl-4-hydroxyquinolin-2(1*H*)-ones **55** treated with mercuric diacetate, pyrano[3,2-*c*]quinolinones **56** were obtained. Compounds **56** were reductively demercurated with sodium borohydride and dehydrogenated with DDQ to give pyrano[3,2-*c*]quinolinones **42** [63]. On the other hand, reaction of compound **55** (R=R[1]=H) with iodine and mercuric oxide afforded directly compound **57** [64] (Scheme 22).

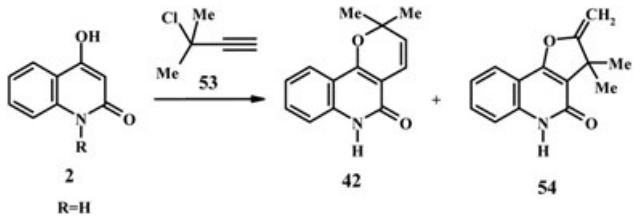
Alkylation of **2** with a number of different 1-aryloxy-4-chlorobut-2-ynes **58** in acetone/K_2CO_3 or under phase transfer catalysis (PTC) alkylation using tetrabutylammonium bromide (TBAB) as a catalyst in NaOH/chloroform gave 1-alkyl-4-[(4′-aryloxybut-2-ynyl)oxy]quinolin-2(1*H*)-ones **59**. Compounds **59** are prone to undergo [3,3] sigmatropic rearrangement followed by rapid enolization and [1,5] hydrogen shift to give intermediates **60** followed by 6π electocyclic ring closure to give final products; 6-alkyl-4-aryloxymethyl-2,6-dihydropyrano[3,2-*c*]quinolin-5-ones **61** [65] (Scheme 23).

6-Alkyl-2*H*-pyrano[3,2-*c*]quinolin-5(6*H*)-ones **63** were obtained in excellent yields (84-91%) by simply refluxing 4-(prop-2-ynyloxy)quinolinones **62** in chlorobenzen for 10h (Scheme 24). On the other hand, thermal cyclization of 2,4-di(prop-2-ynyloxyl)quinolinone **64** under similar

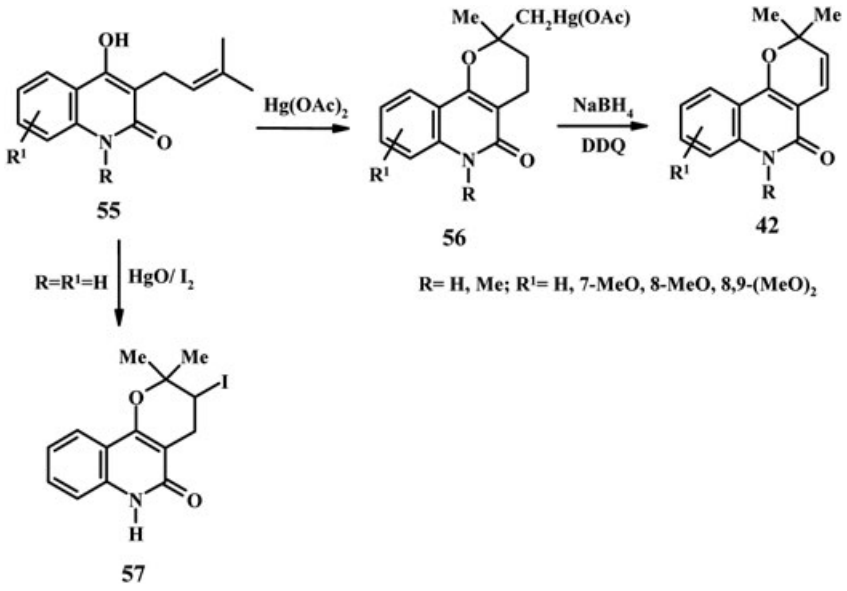
Scheme 20



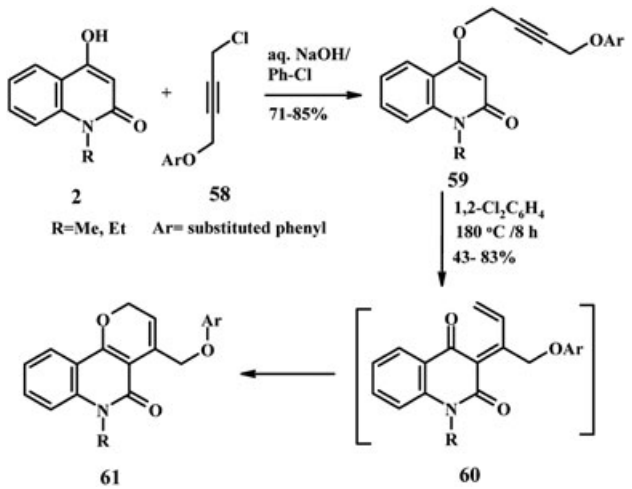
Scheme 21



Scheme 22



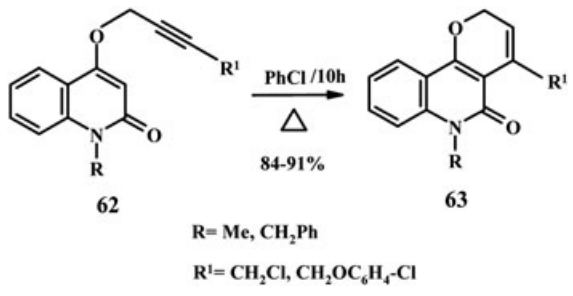
Scheme 23



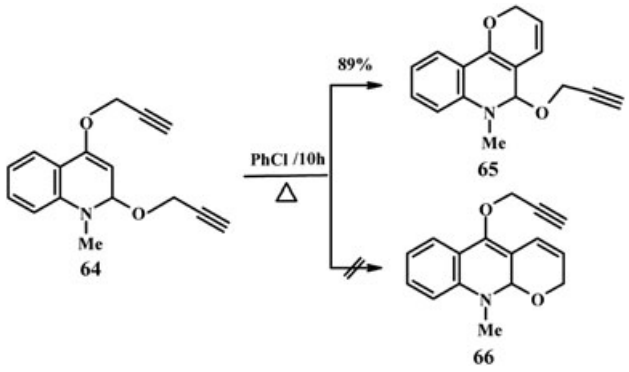
conditions led to 5-(prop-2-ynyloxyl)-2*H*-pyrano[3,2-*c*] quinoline **65** and not the corresponding isomer **66** [66] (Scheme 25). Formation of products **63** and **65** may be rationalized by [3+3] sigmatropic shift of the propynyl ether to form allene followed by enolisation, 1,5-hydrogen shift and electrocyclic ring closure to give finally the cyclic products.

Treatment of 2-methyl-4-hydroxyquinoline (**67**) with dimethyl-acetylene dicarboxylate and PPh_3 in toluene under reflux gave, *via* the intermediate **68**, a mixture of dimethyl 2-(4-hydroxy-2-methyl-3-quinolinyl)-2-butene-dioate (**69**) in 65% yield, as well as trace amounts of methyl 5-methyl-2-oxo-2*H*-pyrano[3,2-*c*]quinoline-4-carboxylate (**70**). When compound **69** was heated at 200-205 °C for additional days, it transformed to compound **70** in 10% yield [67] (Scheme 26).

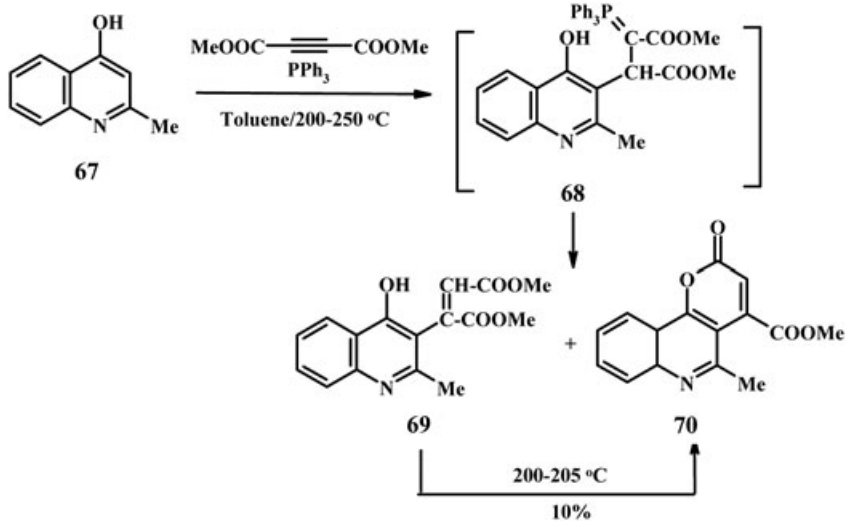
Scheme 24



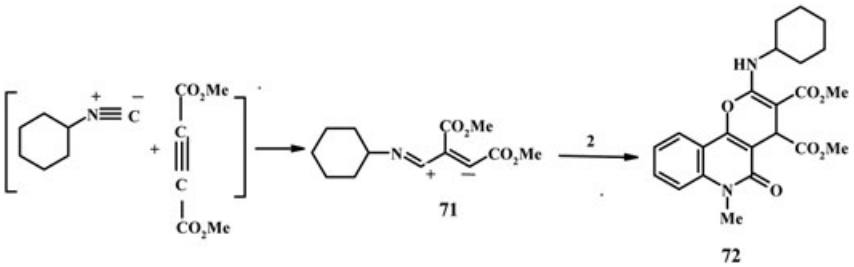
Scheme 25



Scheme 26



Scheme 27

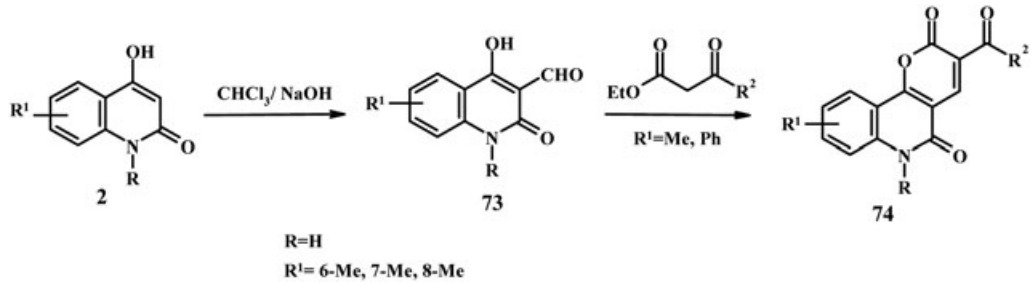


The 1:1 zwitter ionic adduct intermediate **71**, generated from dimethyl acetylene dicarboxylate and cyclohexylisocyanide, was intercepted with compound **2** in one pot to give dimethyl-2-cyclohexylamino-6-methyl-5-oxo-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-3,4-dicarboxylate (**72**) in good yield [68] (Scheme 27).

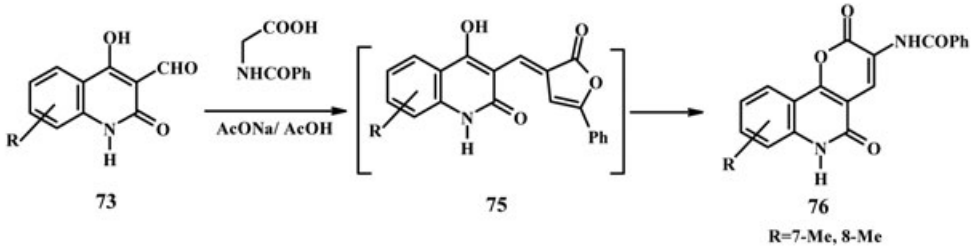
2.2.2. From 4-hydroxyquinoline-3-carbaldehydes. Reimer-Tiemann formylation of substituted 4-hydroxyquinolin-2(1*H*)-ones **2**, using chloroform in sodium hydroxide, gave the corresponding 4-hydroxyquinoline-3-carbaldehdye derivatives **73** [69,70] (Scheme 28). Quinoline-3-carbaldehydes **73** represent a good building block for pyrano[3,2-*c*]quinoline derivatives due to the presence of the nucleophilic 4-OH and the electrophilic formyl groups and their reactions with another partner containing two adjacent C-atoms, one of them being nucleophilic and the other being electrophilic, afforded the target compounds. Therefore, treatment of **73** with ethyl acetoacetate and/or ethyl benzoylacetate afforded 3-acylpyrano[3,2-*c*]quinolines **74** (R=Me, Ph) [40] (Scheme 28).

3-Formyl-4-hydroxy-7-methyl/8-methyl-quinolin-2(1*H*)-ones **73** on condensation with hippuric acid, in glacial acetic

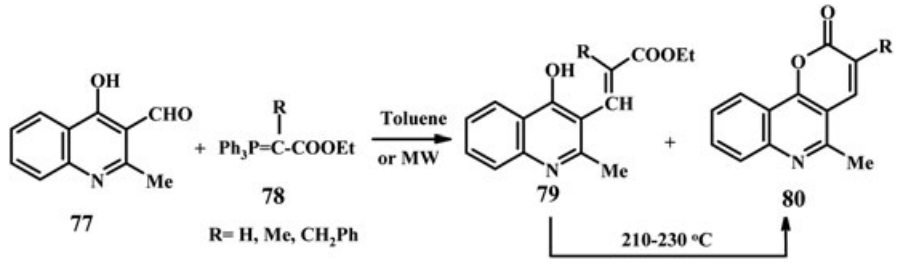
Scheme 28



Scheme 29



Scheme 30



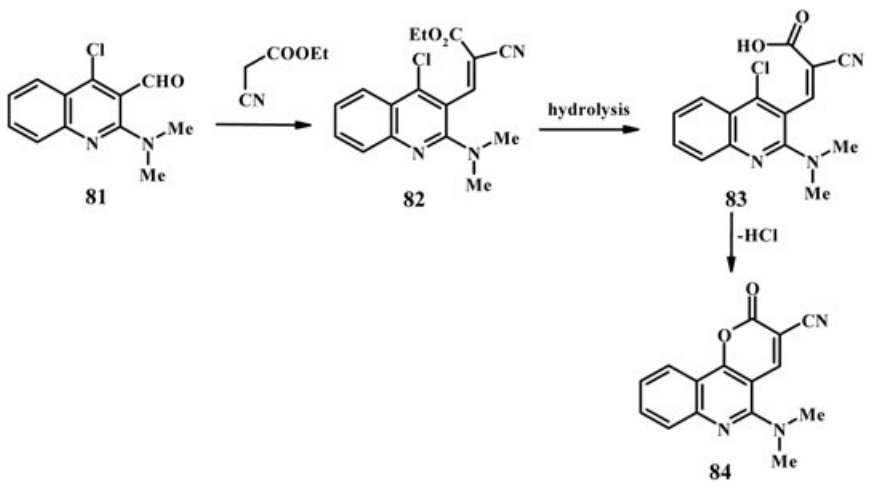
acid in the presence of sodium acetate, did not lead to the expected oxazolin-5-one derivatives **75**, instead 3-benzoyl amino-7-methyl/8-methyl-5,6-dihydropyrano[3,2-*c*] quinoline-2,5-(6*H*)-diones **76** were isolated [69,70] (Scheme 29).

Treatment of 2-methyl-4-hydroxyquinoline-3-carbaldehyde (**77**) with equimolar amount of phosphorus ylides **78**, in dry toluene at 60°C or in commercial microwave oven (800w), afforded ethyl 3-(4-hydroxy-2-methylquinolinyl)-2-propenoates **79** in 72–76% yields as well as 5-methyl-2*H*-pyrano[3,2-*c*]quinolin-2-ones **80** in 7–8% yields. Lactonization of the isolated hydroxyesters **79** to compounds **80** was achieved by heating without solvent at 210–230°C [67] (Scheme 30).

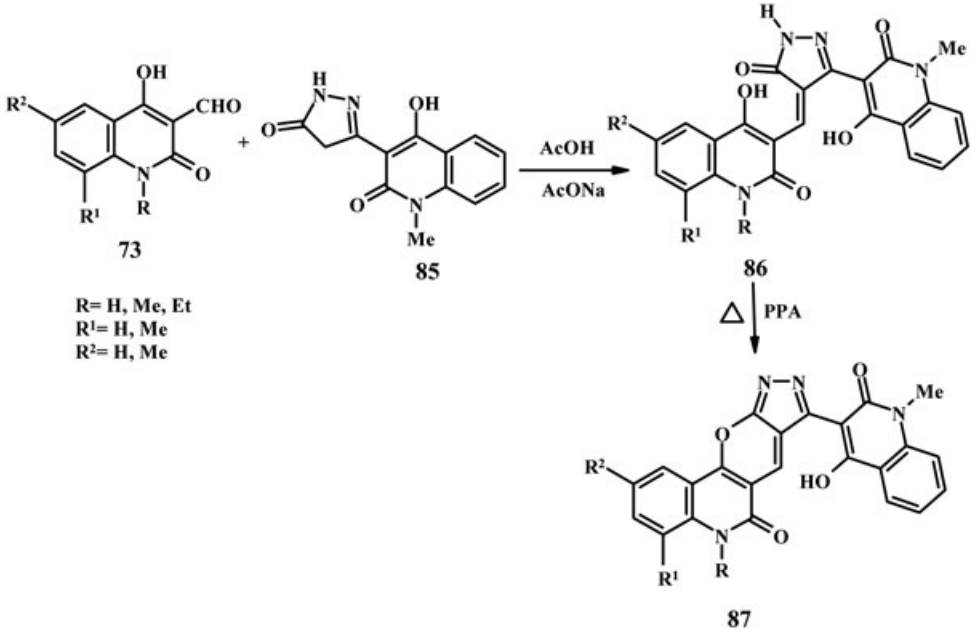
Condensation of 2-dimethylamino-4-chloroquinoline-3-carbaldehyde **(81**) with ethyl cyanoacetate gave the cyanoacrylate derivative **82**, which subsequently underwent hydrolysis giving the corresponding acid **83**. Cyclization of compound **83** gave 5-(dimethylamino)pyrano[3,2-*c*]quinolin-2-one **84** [68] (Scheme 31).

Knoevenagel condensation of 3-formyl-4-hydroxyquinolin-2(1*H*)-ones **73** with quinolinylpyrazolinone **85**, in glacial acetic acid in the presences of fused sodium acetate,

Scheme 31



Scheme 32



afforded quinolinylmethylene-pyrazolinones **86**. Dehydration of compound **86** using polyphosphoric acid (PPA) led to the formation of 8-quinolinylpyrazolo [4′,3′:5,6]pyrano [3,2-*c*]quinolin-6(5*H*)-ones **87** [71] (Scheme 32).

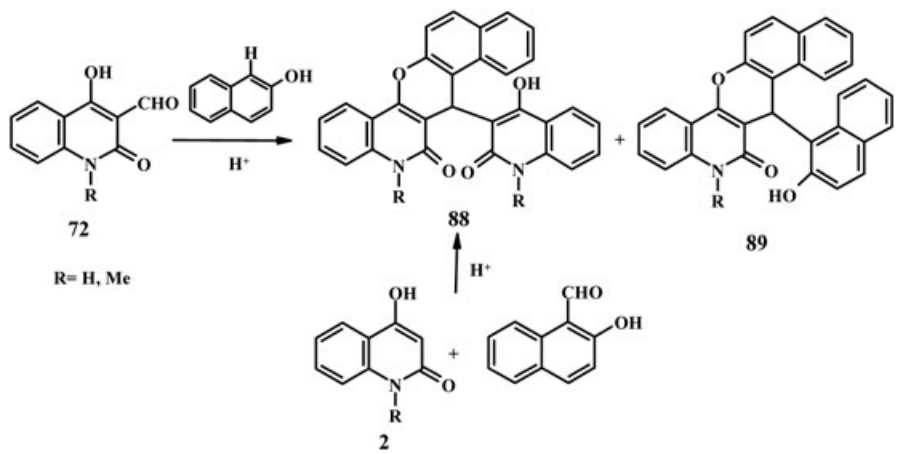
Acid catalyzed condensation of **73** with β-naphthol gave a mixture of 7-(2-oxo-4-hydroxy-3-quinolinyl)naphtho [1′,2′:5,6]pyrano[3,2-*c*]quinolines **88** and 7-(2-naphthyl)-5,7-dihydro-6*H*-benzo[5,6]chromeno[3,2-*c*]quinolin-6-ones **89**. Compound **88** was also obtained authentically from condensation of compound **2** with 2-hydroxy-1-naphthaldehyde under the same reaction conditions [72] (Scheme 33).

2.2.3. From 3-acetyl-4-hydroxyquinolin-2(1H)-ones. *Clasien* condensation of 3-acetyl-4-hydroxy-1-methylquinolin-2 (1*H*)-one (**90**) with diethyl carbonate in the presence of sodium metal led to 1,3-diketone; 3-ethoxycarbonylacetyl-4-hydroxy-1-methylquinolin-2(1*H*)-one (**91**). Cyclization of **91** upon treating with sulfuric acid gave 4-hydroxy-6-methylpyrano[3,2-*c*] quinoline-2,5(6*H*)-dione (**3**) [73] (Scheme 34).

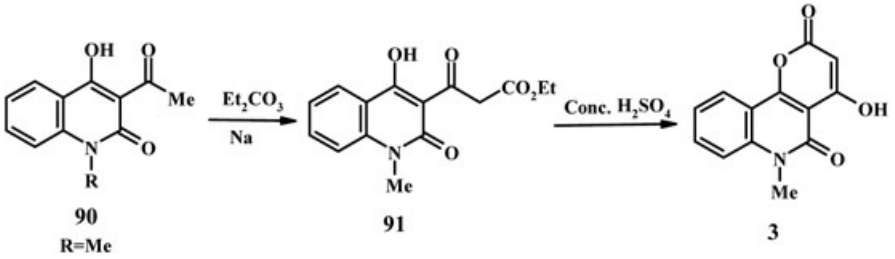
Similarly, *Clasien* condensation of compound **90** with diethyl oxalate in the presence of sodium metal led to α,γ-diketo-ester; ethyl 4-(1-ethyl-4-hydroxy-2-oxo-1,2-dihydroquinolin-3-yl)-2,4-dioxobutyrate (**92**). Cyclization of **92**, in glacial acetic acid in the presence of freshly fused sodium acetate or dry pyridine, afforded ethyl 6-ethyl-5,6-dihydro-4*H*-pyrano[3,2-*c*]quinoline-2-carboxylate (**93**) [74] (Scheme 35).

Also, *Clasien* condensation of compound **93** with ethyl propionate in the presence of sodium metal led to 1,3-

Scheme 33



Scheme 34



diketone; 3-(1,3-dioxopentanyl)-4-hydroxy-1-methylquinolin-2(1*H*)-one (**94**). Cyclization of compound **94** with sulfuric acid gave 2-ethyl-6-methylpyrano[3,2-*c*]quinoline-2,5(6*H*)-dione (**95**) [73] (Scheme 36).

3-[(2*E*)-3-(Dimethylamino)prop-2-enoyl]-4-hydroxy-1-methyl-quinolin-2(1*H*)-one (**96**) was smoothly obtained *via* thermal condensation of compound **90** (R=Me) with dimethylformamide-dimethylacetal (DMF-DMA). Enaminone **96** when boiled in glacial acetic acid underwent an intramolecular cyclo-condensation furnishing 4*H*-pyrano [3,2-*c*]quinoline-4,5(6*H*)-dione (**97**) [75] (Scheme 37).

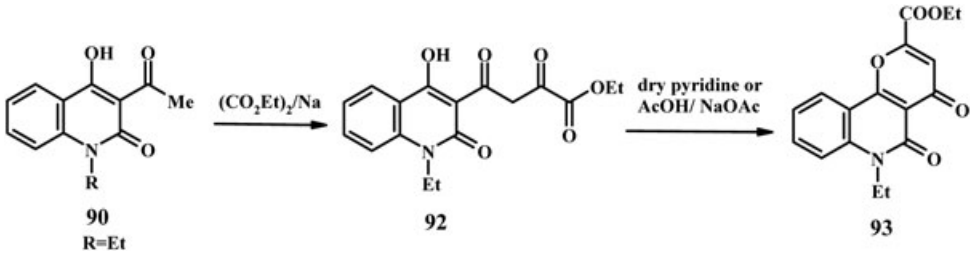
When enaminone **96** was allowed to react with some active methylene compounds such as ethyl cyanoacetate and/or malononitrile in the presence of potassium hydroxide, the pyranoquinolinone derivatives **99** were obtained, *via* the nonisolable intermediates **98** [75] (Scheme 38).

The enaminone **96** reacted with diethyl acetonedicarboxylate (**100**), in acetic acid in the presence of ammonium acetate as a catalyst, to give compound **102**. Compound **103** is thought to be formed *via* condensation of the active methylene group in compound **100** with the carbonyl function in enaminone **96** with water elimination forming intermediate **101**, which was cyclized in the presence of ammonium acetate *via* elimination of dimethylamine and ethanol to afford compound **102**. Dehydration of **102** afforded 2-ethoxycarbonylmethyl-3,7,8-trihydro-8-phenyl-pyrano[4,3-*b*]quinoline-3,7-dione (**103**) [76] (Scheme 39).

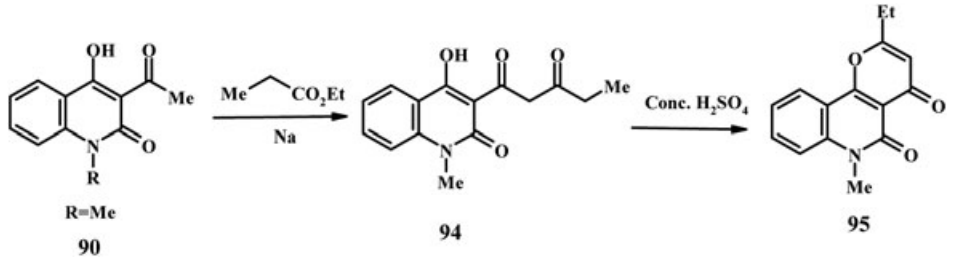
Reaction of compound **90** with arylidenenitriles **26**, in ethanol/TEA, gave 2-amino-pyrano[3,2-*c*]quinolin-5(6*H*)-ones **27** and **28** *via* intermediates **104** in which the acetyl group eliminates under the reaction conditions [77,78] (Scheme 40).

Refluxing equivalent amounts of 3-acetyl-4-hydroxy-2-methyl-6-iodo-quinoline (**105**) with malononitrile and/or ethyl cyanoacetate, in glacial acetic acid containing catalytic amounts of ammonium acetate, afforded 9-iodopyrano [3,2-*c*] quinoline-3-carbonitrile **106** and **107**, respectively.

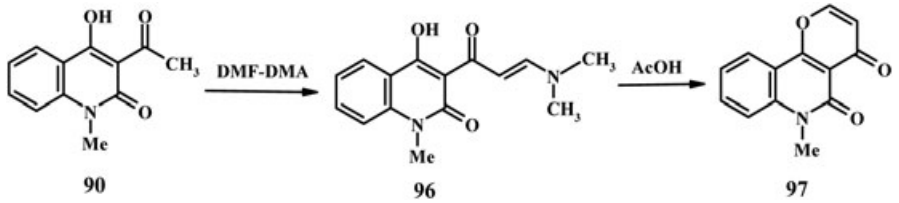
Scheme 35



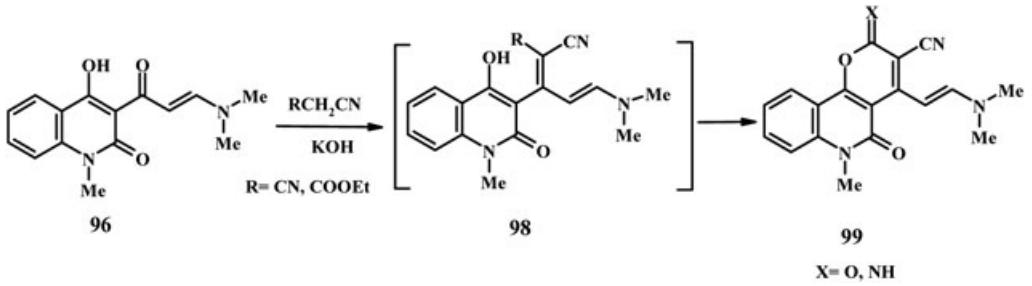
Scheme 36



Scheme 37



Scheme 38

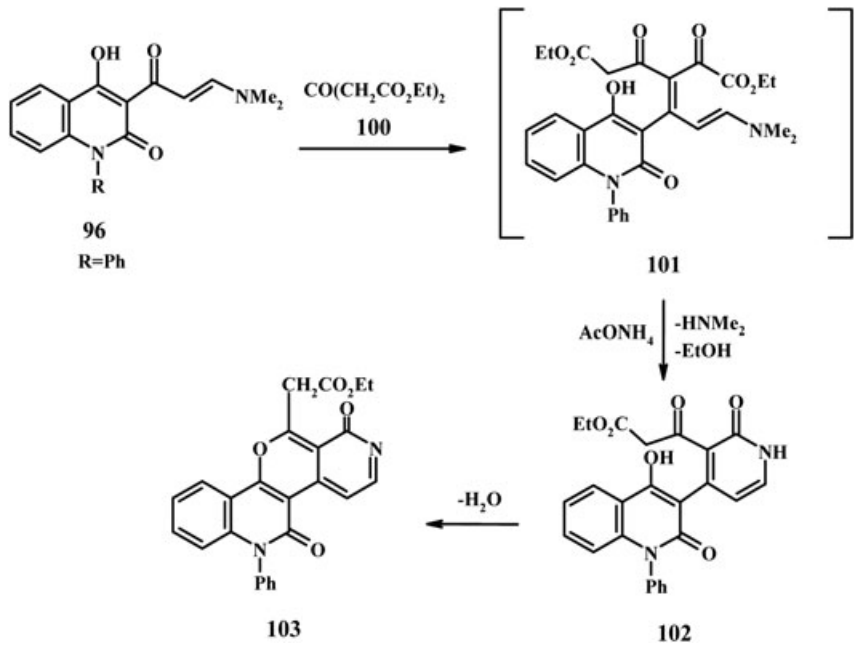


3-Formyl-5-methyl-4-oxo-9-iodo-pyrano[3,2-*c*]quinoline (**108**) was obtained *via* the reaction of acetyl derivative **105** with Vilsemier-Hacck reagent ($POCl_3$/DMF). Also, condensation of compound **105** with diethyl oxalate in sodium ethoxide yielded ethyl 9-iodo-5-methyl-4-oxo-pyrano[3,2-*c*]quinoline-2-carboxylate (**109**) [79] (Scheme 41).

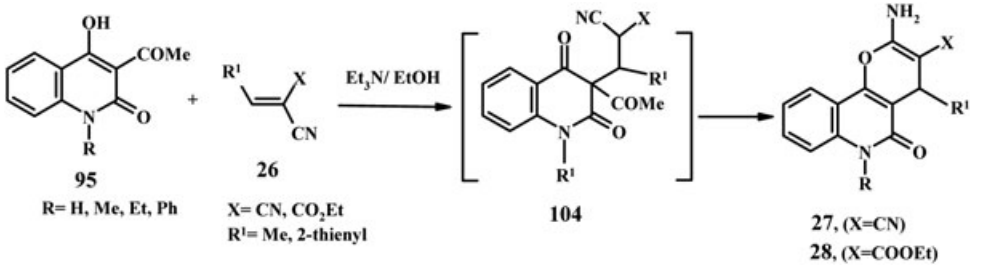
Wittig reaction of 3-acyl-4-hydroxyquinolin-2(1*H*)-ones **110** with ethyl (triphenylphosphoranylidene)acetate (**111**) proceeds with high stereo-selectivity giving pyrano[3,2-*c*] quinoline-2,5(6*H*)-diones **112** in good yield [42] (Scheme 42). While, reaction of 3-acetyl-4-hydroxy-1-methylquinolin-2(1*H*)-one **90** (R=Me) with ethyl (triphenyl-phosphoranylidene)chloroacetate (**113**) in boiling xylene gave a mixture of ethyl 3,5-dimethyl-4-oxo-5,6-dihydro-furo[3,2-*c*]quinoline-2-carboxylate (**114**) and ethyl 4,6-dimethyl-2-oxo-5,6-dihydro-2*H*-pyrano[3,2-*c*]quinolin-2-ylidene(chloro) acetate (**115**) [80] (Scheme 42).

Condensation of compound **90** with phenyl hydrazine gave the corresponding hydrazone **116** which underwent Vilsmeier-Hacck formylation to give 3-(4-hydroxy-

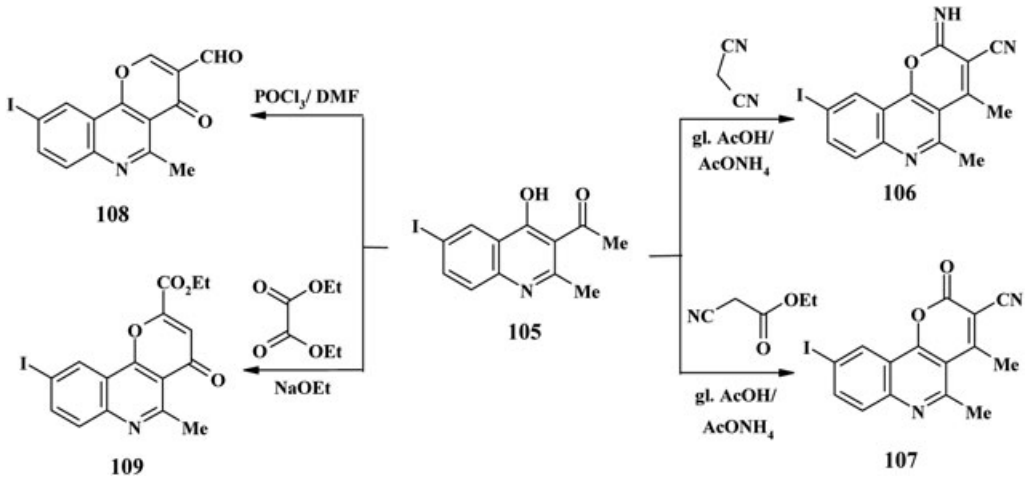
Scheme 39



Scheme 40



Scheme 41



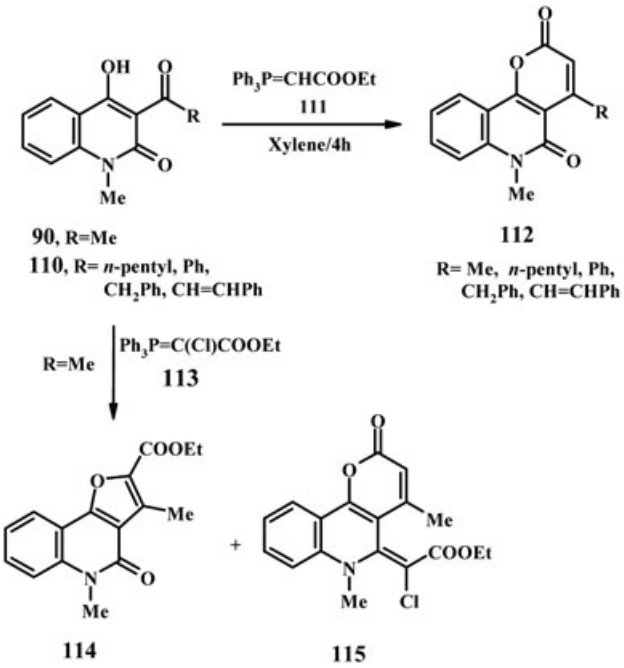
1-methyl-1,2-dihydro-2-oxoquinolin-3-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehyde (**117**). Treatment of **117** with hydroxylamine hydrochloride, in glacial acetic acid did not give the expected oxime or even the probable carbonitrile **118**. The compound that separated from the latter reaction was characterized as pyrazolo [3′,4′:4,5][3,2-*c*] quinolinedione **119** [75] (Scheme 43).

Reaction of compound **90** with a mixture of phenylisothiocyanate and glycine in acetic acid gave *N*-pyrano [3,2-*c*]quinoline-2,5(6*H*)-dion-3-yl-*N*′-phenylthiourea derivatives **121** in 70–75% yields as shown in Scheme 43 [81]. Compounds **121** were supposed to be formed *via* condensation of the carbonyl group in **90** with the active methylene group in glycine derivative to give the intermediates **120** which readily cyclized *via* water elimination to yield **121**. Condensation of the latter compound with chloroacetic acid, in the presence of fused sodium acetate and acetic anhydride, resulted in the formation of thiazolidine derivatives **122** (Scheme 44).

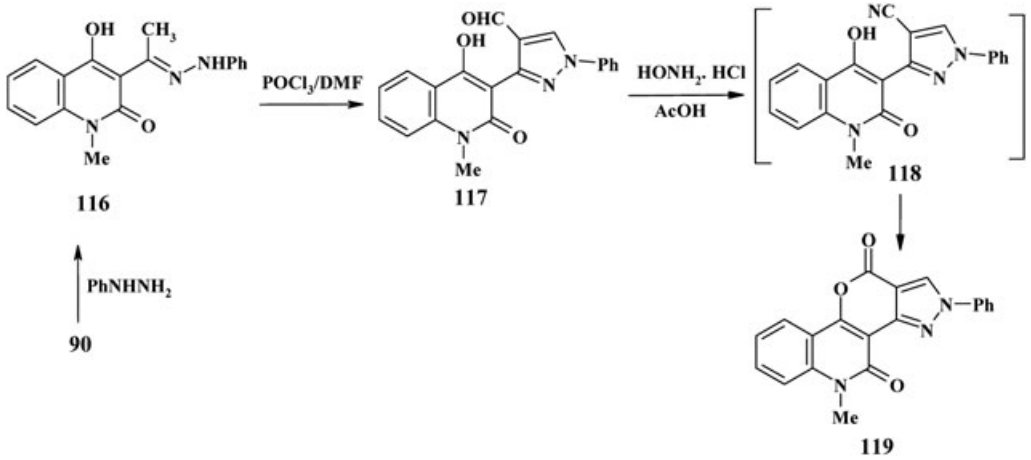
2.3 From cyclic anilines

2.3.1 From tetrahydroquinoline derivatives. Condensation of tetrahydroquinoline (**123**) with excess diethyl malonate

Scheme 42



Scheme 43



in boiling diphenyl ether led to angular fused pyrano[3,2-*c*] quinoline **124** [82] (Scheme 45).

2.3.2. From indoline derivatives. Condensation of indoline derivatives **125** with diethyl malonate in boiling diphenyl ether led to the tetracyclic systems **126** [21] (Scheme 46).

3. CHEMICAL REACTIVITY OF PYRANO[3,2-*c*] QUINOLINONES

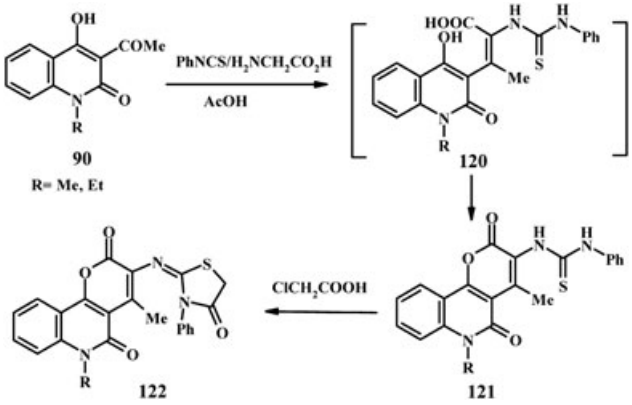
The chemical reactivity of pyrano[3,2-*c*]quinolinones was found to include α-pyrone ring opening (hydrolysis, hydrazinolysis), electrophilic and nucleophilic substitution reactions in addition to condensation reactions.

3.1. Hydrolysis

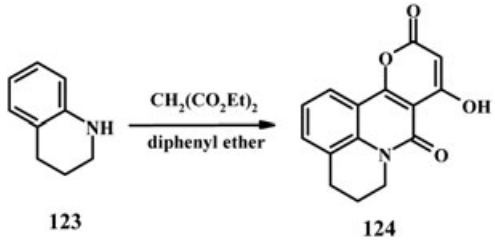
3.1.1. Basic hydrolysis. Treatment of 3-benzoylamino-7-methyl/8-methyl-pyrano[3,2-*c*]quinolin-2,5 (*6H*)-diones **76** with aqueous sodium hydroxide solution led to α–pyrone ring opening to afford 2-benzoylamino-3(1,2-dihydro-4-hydroxy-7-methyl/8-methyl-2-oxo-3-quinolinyl)acrylic acid **127** [69], [70] (Scheme 47).

Alkaline hydrolysis of 6-substituted-4-hydroxypyrano [3,2-*c*]quinoline-2,5(6*H*)-diones **3**, using 15% sodium

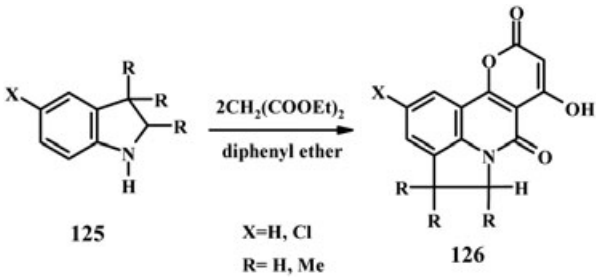
Scheme 44



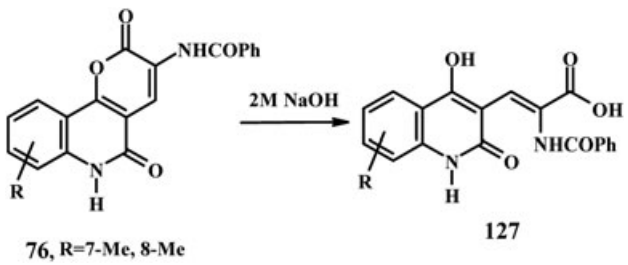
Scheme 45



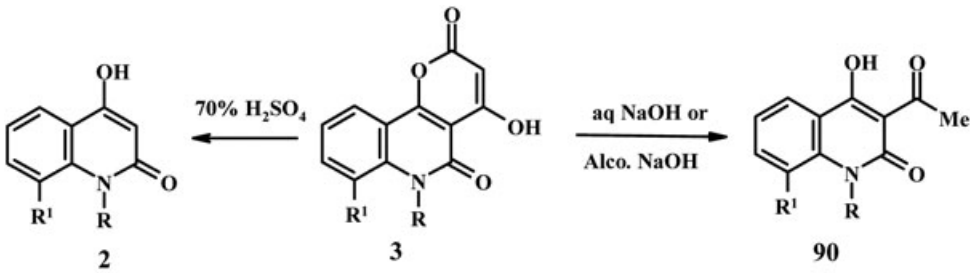
Scheme 46



Scheme 47

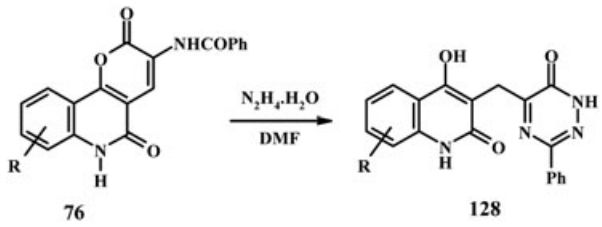


Scheme 48



Scheme 49

Scheme 50

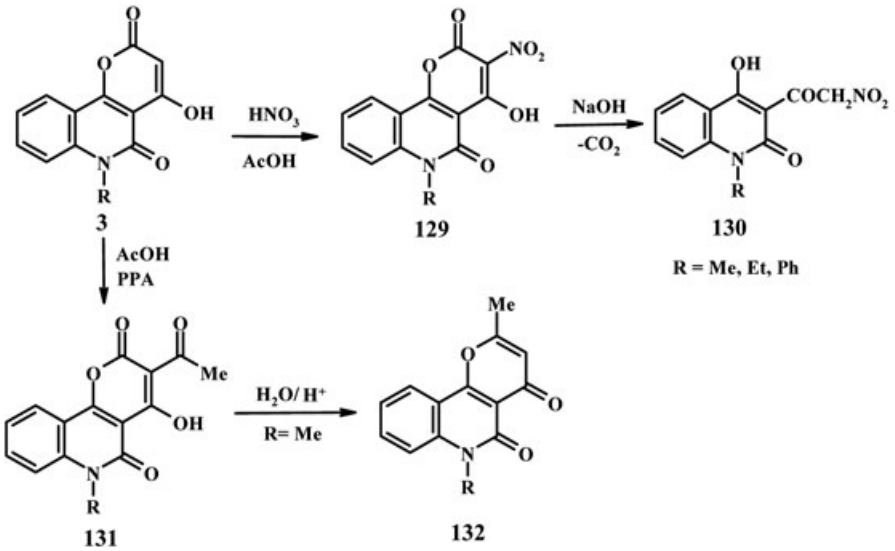


hydroxide, furnished the corresponding 3-acetyl-1-substituted-4-hydroxyquinolin-2(1*H*)-ones **90**. The reaction proceeds through α–pyrone ring opening in basic medium with subsequent decarboxylation to afford the desired products **90** [26,28,30,35,83–85] (Scheme 48).

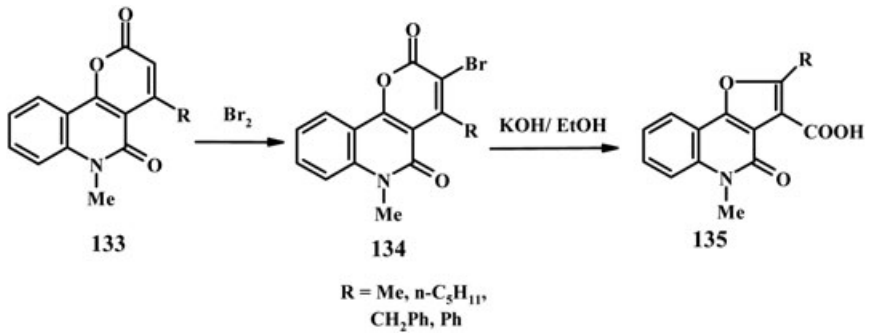
3.1.2. Acid hydrolysis. Heating pyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **3** in 70% sulfuric acid resulted in destroy of the α-pyrone ring producing the well known 4-hydroxy-1-alkylquinolines **2** [35] (Scheme 48).

3.2. Hydrazinolysis. Ring opening ring closure sequence of the tandem reaction of 4-hydroxy-6-substituted-pyrano [3,2-*c*]quinoline-2,5(6*H*)-diones **3** with hydrazine hydrate in DMF under reflux gave 4-hydroxy-1-substituted-3-(5-oxo-4,5-dihydro-1*H*-pyrazol-3-yl)quinolin-2(1*H*)ones (**85**) in good yield [29,86], (Scheme 49).

Scheme 51



Scheme 52



Reaction of the pyranoquinolinones **76** with hydrazine hydrate in boiling DMF caused successive ring opening and ring closure transformation to give 3-(1,6-dihydro-6-oxo-3-phenyl[1,2,4]triazin-5-yl)methyl-4-hydroxy-7-methyl/8-methyl- quinolin-2(1*H*)-ones **128** [69,70], (Scheme 50).

3.3. Electrophilic substitution reactions. Electrophilic substitution reactions such as nitration, acetylation, halogenation, formylation, and condensation reactions with 4-hydroxypyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **3** take place at the acidic CH at position 3.

3.3.1. Nitration. Nitration of pyrano[3,2-*c*]quinoline-2,5(6*H*)-diones **3** at 70–90°C with conc. nitric acid or conc. nitric acid in acetic acid gave 3-nitropyranoquinolines **129** in good yield. Hydrolytic cleavage of compound **129** using aqueous sodium hydroxide led to 3-nitroacetylquinolinones **130** [26], [87] (Scheme 51).

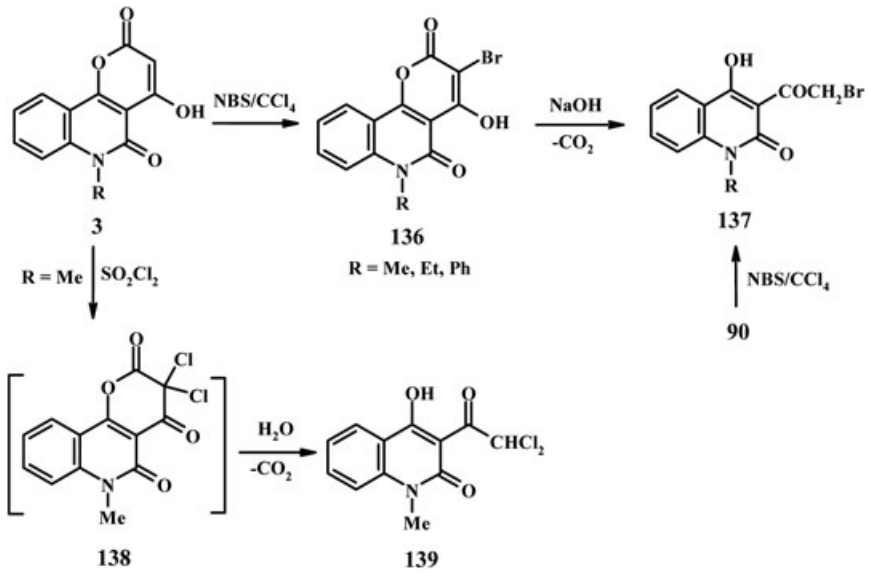
3.3.2. Acetylation. Introduction of an acetyl group into the position 3 of pyranoquinolinones **3** takes place through direct acetylation of **3** with acetic acid in polyphosphoric acid to give 3-acetylpyranoquinolinediones **131** in good yields 26. The 3-acetyl derivative **131** (R=Me) can be rearranged with mineral acids to afford 2-methylpyrano [3,2-*c*] quinoline-4,5(4*H*)-dione **132** as reported by Kappe [26] (Scheme 51).

3.3.3. Halogenation

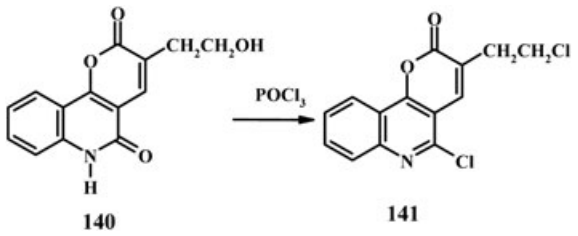
3.3.3.1. Bromination. Bromination of compounds **133**, using bromine in chloroform, gave 3-bromopyranoquinoline derivatives **134** which underwent alkaline hydrolysis to give 2-alkyl/aryl-4-oxo-furo[3,2-*c*]quinoline-3-carboxylic acids **135** [80] (Scheme 52).

On the other hand, bromination of compounds **3** with *N*-bromosuccinimide (NBS) afforded the corresponding 3-bromo derivatives **136**. Hydrolytic cleavage of **136** using aqueous sodium hydroxide led to 3-bromoacetylquinolinones **137** which were also obtained from direct bromination of 3-acetylquinolinones **90** under the same reaction conditions as published by El-Taweel *et al*. [87] (Scheme 53).

Scheme 53



Scheme 54



3.3.3.2. Chlorination. Chlorination of pyranoquinolinone **3** using sulfuryl chloride in dioxane did not give the expected 3,3-dichloropyrano[3,2-*c*]quinoline-2,4,6-trione **138**, but gave the 3-dichloroacetylquinolinone **139** (R=Me) *via* ring opening of the nonisolable intermediate **138** followed by decarboxylation [81] (Scheme 53).

Action of phosphorus oxychloride on compound **140** converted the ethanol side chain to the chloroethyl group, and the lactam carbonyl was converted to the imidoy1 chloride producing 3-chloroethyl-5-chloro-pyrano[3,2-*c*] quinolin-2-one (**141**) [44] (Scheme 54).

3.3.4. Formylation. Vilsmeier-Hacck formylation ($POCl_3$/DMF) of 4-hydroxypyrano[3,2-*c*] quinolinediones **3** resulted in the formation of 3-formylpyrano[3,2-*c*]quinoline-4,5(6*H*)-diones **142** in 60–65% yields. Compounds **142** are proposed to be formed as sequence demonstrated in Scheme 55 [87].

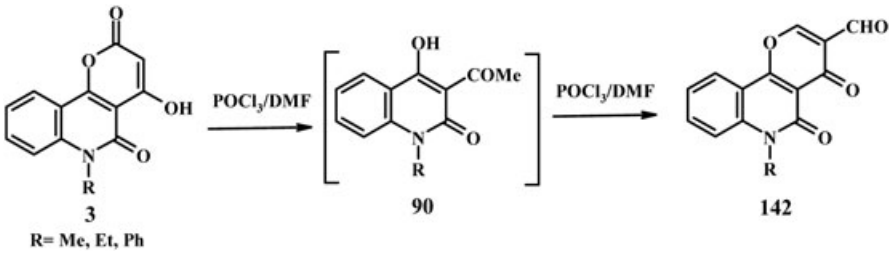
3.4. Nucleophilic substitution reactions. Reaction of 4-hydroxypyranoquinolines **3** (R^3= OH) with isopropyl amine and/or methylammonium chloride as methylamine source gave the corresponding 4-alkylamino derivatives **143** [28]. While, fusion of **3** (R^3= Me, Ph) with formamide produced the corresponding naphthopyridine derivatives **144** [40] (Scheme 56).

3.5. Condensation reactions. Condensation of 6-methylpyrano[3,2-*c*]quinoline-2,5(6*H*)-dione **3** (R=Me) with ylidenenitriles **26** or with a mixture of aromatic aldehyde and activated nitriles; malononitrile or ethyl cyanoacetate afforded 3-(4*H*-pyran-2-yl)quinolinone **149**. Compounds **149** are assumed to be formed *via* intermediates **145-148** as demonstrated in Scheme 57 [78].

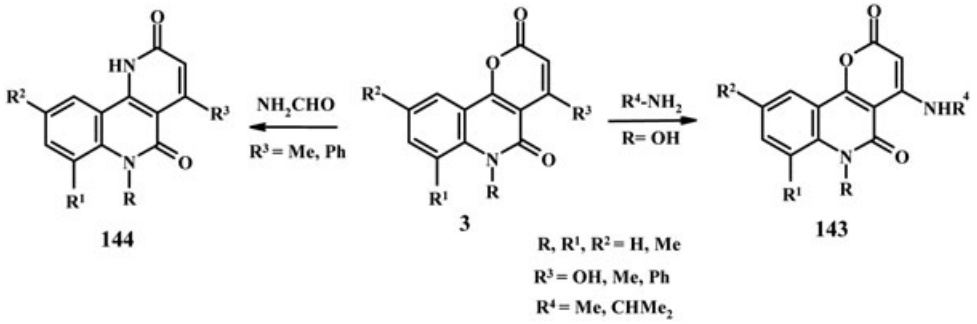
In contrary to the behavior of compound **3** (R=Me) towards nitriles **26**, the reaction of **3** (R=H, Et) with ylidenenitriles **26** afforded pyrano[3,2-*c*]quinolin-5-ones **27** and **28** (Scheme 58). Formation of compounds **27** and **28** is assumed to proceed *via* initial hydrolysis of compounds **3** (R=H, Et) under the reaction conditions to give acetylquinolinones **90** [78]. compound**s 90** were added to the π-deficient double bond in nitriles **26** to form adducts **104** which then converted into compounds **27** and **28** *via* deacetylation as has been previously reported [88–90].

On the other hand, treatment of **3** with ylidine **150** or a mixture of malononitrile and isatine in ethanol/piperidine

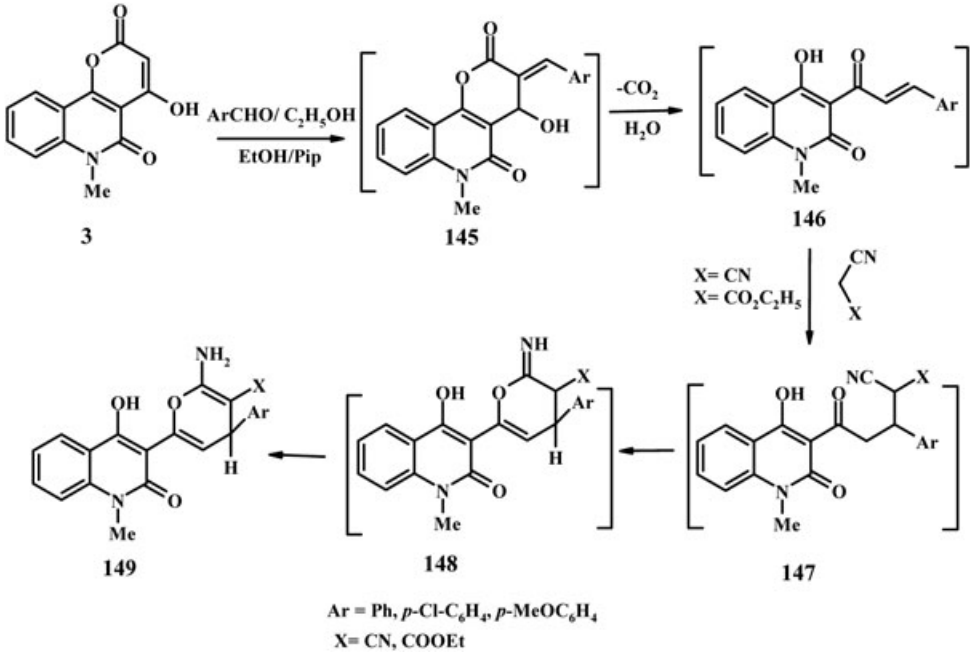
Scheme 55



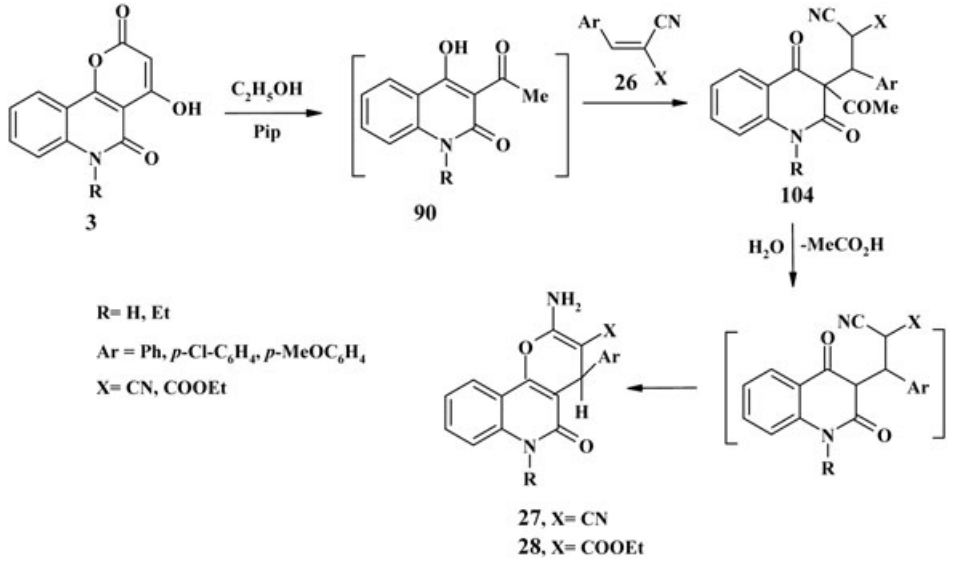
Scheme 56



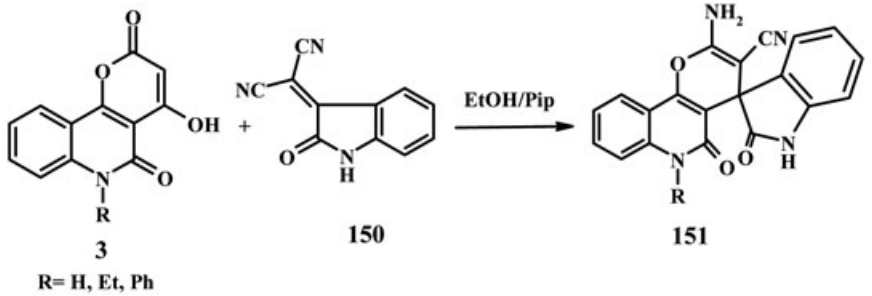
Scheme 57



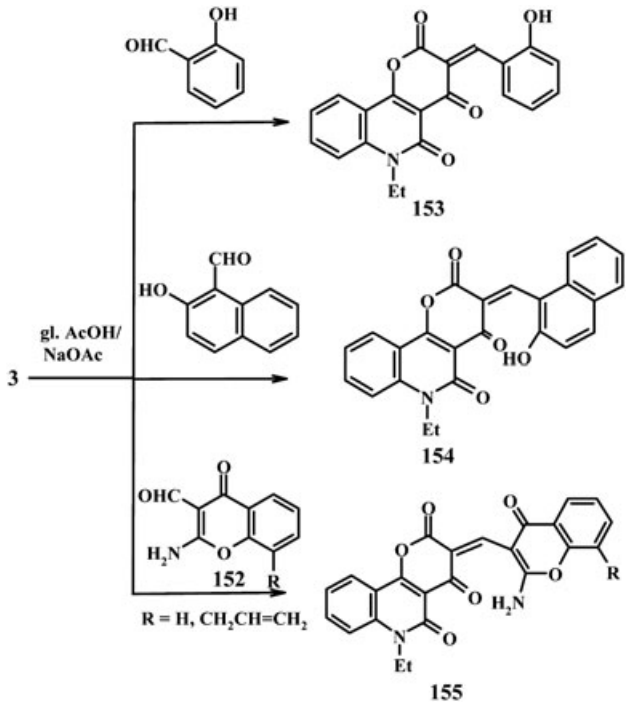
Scheme 58



Scheme 59



Scheme 60



yielded 2-amino-5,6-dihydro-5-oxo-6-substituted-4-(1′,3′-dihydro-2′*H*-indol-2-one)spiro-4*H*-pyrano[3,2-*c*]quinoline-3-carbonitrile **151** [88] (Scheme 59). Formation of compounds **151** from compounds **3** and **150** is assumed to proceed *via* initial hydrolysis of **3** under the reaction conditions to give the acetyl derivative **90** [91]. Herein again, quinolinyl C-3 in **90** was added to the π-deficenet center in compounds **150** giving **151** as has been shown before.

Recently [92], we reported that, condensation of **3** with salicylaldehyde, 2-hydroxy-1-naphthaldehyde and 2-amino-3-formylchromones **152**, in glacial acetic acid and freshly fused sodium acetate, yielded 6-ethyl-pyrano[3,2-*c*]quinoline-2,4,5-(3*H*,6*H*)-triones **153**-**155**, respectively (Scheme 60).

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