



## 3-Formylchromones as diverse building blocks in heterocycles synthesis

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### ABSTRACT

This review covers the chemical reactivity of 3-formylchromones towards condensation reactions with a variety of carbon and nitrogen nucleophiles. Some chromone derivatives linked a variety of heterocyclic systems were prepared from the direct condensation of 3-formylchromones with heterocyclic compounds containing active methylene groups. A diverse number of fused heterocyclic systems were prepared from the reaction of 3-formylchromones with some bifunctional nucleophiles, these reactions mainly proceed via condensation with the aldehydic function followed by nucleophilic attack at C-2 position of the chromone moiety.

### 1. Introduction

Chromone derivatives are widely known as an important class of biologically active compounds. The chemical reactivity of 3-substituted chromones is widely different depending on the nature of the functional group present at the position 3 and the reaction conditions. Among the 3-functionalized chromones, their 3-formyl derivatives are widely used in heterocyclic synthesis. 3-Formylchromones are also known as 4-oxo-4H-1-benzopyran-3-carboxaldehydes, 4-oxo-4H-chromene-3-carboxaldehydes and chromone-3-carboxaldehydes. Although many methods are known for the synthesis of 3-formylchromones, Vilsmeier-Haack reaction on substituted 2-hydroxyacetophenones is the most suitable among them [1-17]. In Vilsmeier-Haack reaction, the reaction takes place via double formylation of *o*-hydroxyacetophenones followed by cycloaddition with concomitant dehydration (Scheme 1). DMF-POCl<sub>3</sub> plays a dual role of a reagent as well as a solvent. A variety of substituted 3-formylchromones **1a-z** were prepared under traditional and microwave irradiation using Vilsmeier-Haack reagent and are reported herein.

### 2. Chemical reactivity of 3-formylchromones

3-Formylchromones, **1a-z**, are a versatile synthons for the synthesis of a variety of novel heterocyclic systems possessing diverse biological activities. From a synthetic view point, 3-formylchromones, **1a-z**, occupy an important position in the synthesis of various heterocyclic systems, due to the availability of three electron deficient sites, the aldehydes carbon, C-2 carbon, and the C-4 carbon of the carbonyl group. Also, 3-formylchromones are able to serve as a heterodiene as well as a dienophile or a Michael acceptor. Moreover, a variety of fused heterocycles were prepared directly from the reaction of compounds **1a-z** with some bifunctional nucleophiles. The present review aims to study the chemical reactivity of 3-

formylchromones towards a variety of carbon and nitrogen nucleophiles under different reaction conditions.

#### 2.1. Chemical reactivity of 3-formylchromones towards active methyl and methylene compounds

##### 2.1.1. Condensation reactions with active methyl compounds

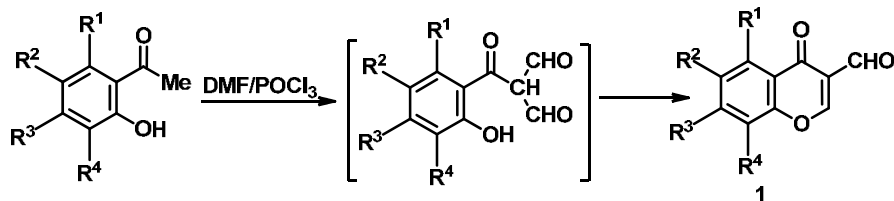
Condensation reactions of 3-formylchromone **1a-c, h** with substituted acetophenone **2** in freshly distilled pyridine or glacial acetic acid containing perchloric acid afforded substituted 3-(3-oxo-3-arylprop-1-enyl)chromones, **3** (Scheme 2) [18-21].

4-Hydroxy-1-alkyl-3-[3-(4-oxo-4H-chromen-3-yl)]quinolin-2(1H)-ones (**5**) were smoothly obtained via a Knoevenagel condensation of 3-formylchromone (**1a**) with 3-acetyl-4-hydroxy-1-alkylquinolin-2(1H)-one (**4**) in ethanol containing piperidine as basic catalyst (Scheme 3) [22,23].

Interaction of equimolar amount of 3-formyl-6-chlorochromone (**1c**) with 4-acetyl-5,6-diphenylpyridazin-3(2H)-one (**6**) in sodium ethoxide afforded 4-[3-(6-chloro-4-oxochromen-3-yl)prop-2-enyl]-5,6-diphenylpyridazin-3(2H)-one (**7**) in 87% yield. When this reaction was carried out in ethanol containing few drops of piperidine, 7-(6-chloro-4-oxochromen-3-yl)-3,4-diphenyl-6,7-dihydropyrano[2,3-c]-pyridazin-5-one (**8**) was obtained in 55% yield, via intramolecular cycloaddition reaction in compound **7** (Scheme 4) [24].

Treating 3-formylchromones **1a-c** with 5-acetylbarbituric acid (**9a**) and 5-acetylthiobarbituric acid (**9b**), in ethanol containing pyridine or water-Zn(L-proline)<sub>2</sub>, gave the corresponding  $\alpha,\beta$ -unsaturated ketones, **10a,b** (Scheme 5) [25].

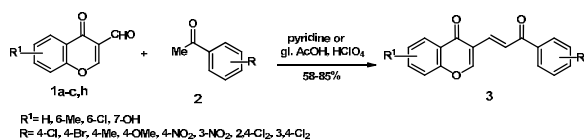
Similarly, treating 3-formylchromones **1a,b** with 5-acetyl-1,3-dimethylbarbituric acid (**11**) under the same reaction conditions afforded 1-(1,3-dimethyl-2,4,6-pyrimidinetrione-5-yl)-3-(4-oxo-4H-chromen-3-yl)-2-propen-1-one (**12a**) and



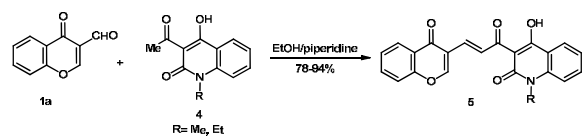
1	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>	R <sup>4</sup>	Yield (%)	M.p.(°C)
a	H	H	H	H	63	150-151
b	H	Me	H	H	65	174-175
c	H	Cl	H	H	72	166-168
d	H	Br	H	H	57	190-191
e	H	F	H	H	76	155-160
f	H	OH	H	H	55	210-212
g	H	NO <sub>2</sub>	H	H	82	253-254
h	H	H	OH	H	41	269
i	H	H	OMe	H	62	188-190
j	H	OMe	H	H	65	174-175
k	H	H	H	allyl	42	73-74
l	H	Me	Me	H	63	192-193
m	Me	H	Me	H	68	145-147
n	H	Me	H	Me	61	187-190
o	H	Me	H	Cl	69	170-171
p	H	Me	Cl	H	45	166-167
q	H	Cl	H	Cl	46	169-174
r	H	Br	H	Br	40	177-178
s	H	Cl	H	NO <sub>2</sub>	67	108
t	H	Cl	H	Br	65	155
u	H	Me	H	NO <sub>2</sub>	69	180
v	H	Me	H	Br	66	145
w	H	NO <sub>2</sub>	H	Br	63	81
x	H	OH	H	H	68	198
y	H	H	OH	NO <sub>2</sub>	64	162
z	H	H	OH	Br	74	210

Scheme 1

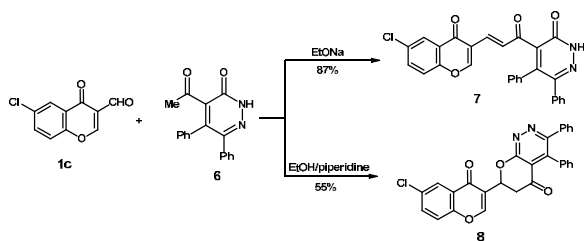
1-(1,3-dimethyl-2,4,6-pyrimidinetrione-5-yl)-3-(6-methyl-4-oxo-4H-chromen-3-yl)-2-propen-1-one (**12b**), respectively (Scheme 6) [26].



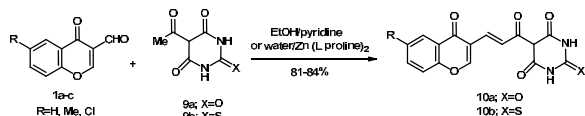
Scheme 2



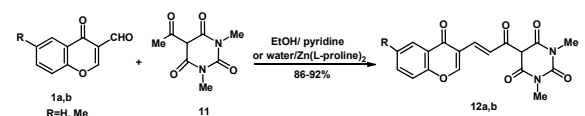
Scheme 3



Scheme 4

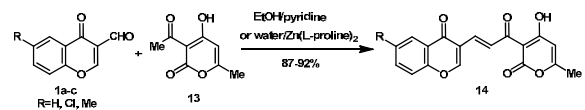


Scheme 5



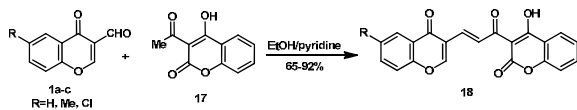
Scheme 6

Condensation of 3-formylchromones **1a-c** with dehydroacetic acid (**13**) in ethanol containing pyridine or water-Zn(L-proline)<sub>2</sub> gave α,β-unsaturated ketones **14** in high yields (87-92%) (Scheme 7) [26].



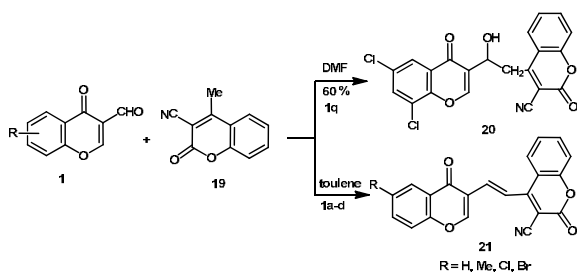
Scheme 7

Refluxing 3-formylchromones **1a-c** with 3-acetyl-4-hydroxycoumarin (**17**) in ethanol containing pyridine as a basic catalyst gave 1-(4-hydroxycoumarin-2-one-3-yl)-3-(chromen-4-one-3-yl)-2-propen-1-ones (**18**) in 65-92% yields (Scheme 8) [26,27].



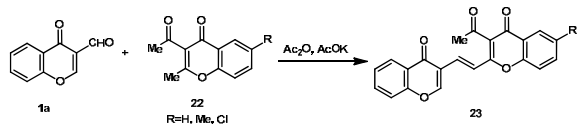
Scheme 8

Reaction of 6,8-dichloro-3-formylchromone (**1q**) with 4-methyl-2-oxo-2H-chromone-3-carbonitrile (**19**) gave the addition product **20** as recently reported by El-Shaar [28]. While Melikyan *et al.* [29] isolate the condensation products **21** on the reaction of **1a-d** with compound **19** in refluxing toluene (Scheme 9).



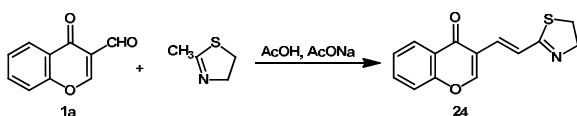
Scheme 9

Condensation of 3-formylchromone (**1a**) with 2-methyl-3-acetylchromones (**22**) in acetic anhydride-potassium acetate led to *bis*-chromonyl ethylene **23** (Scheme 10). This reaction occurred only at 2-methyl group [30].



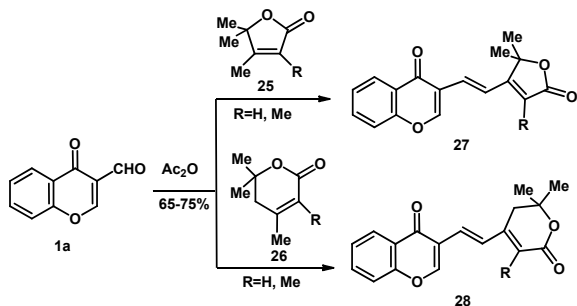
Scheme 10

2-(4-Oxo-4H-chromen-3-yl)vinylthiazoline (**24**) was synthesized by condensation of 3-formylchromone **1a** with 2-methylthiazoline in glacial acetic acid containing sodium acetate (Scheme 11) [31].



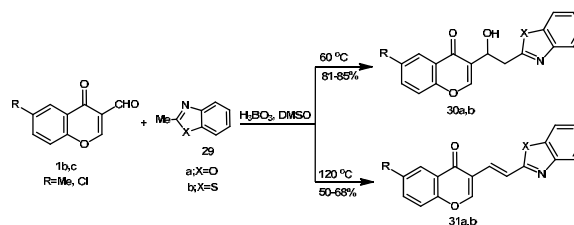
Scheme 11

Condensation of 3-formylchromone (**1a**) with 4,5,5-trimethyl-2,5-dihydrofuran-2-ones **25** and 4,6,6-trimethyl-5,6-dihydropyran-2-ones **26** in acetic anhydride yielded the condensation products **27** and **28**, respectively (Scheme 12) [29].



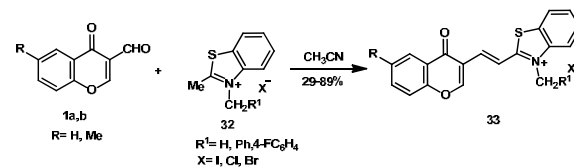
Scheme 12

3-Formylchromones **1b,c** reacted with 2-methylbenzimidazole **29a** and 2-methylbenzothiazole **29b** in dry DMSO and boric acid to give the addition products **30a,b** when the reaction took place at 60 °C, but when the reaction took place at 120 °C afforded the condensation products **31a,b** (Scheme 13) [32].



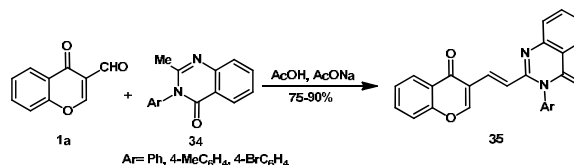
Scheme 13

Condensation of aldehydes **1a,b** with 2-methylbenzothiazolium halides **32** in boiling acetonitrile gave chromenylbenzothiazolium derivatives **33** (Scheme 14) [33].



Scheme 14

Treatment of 3-formylchromone **1a** with 3-aryl-2-methyl-4(3H)-quinazolinones (**34**) in glacial acetic acid containing fused sodium acetate led to the condensed product **35** (Scheme 15) [33].

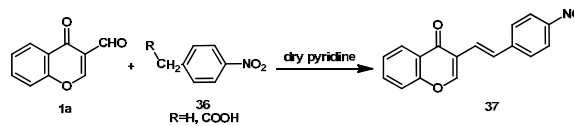


Scheme 15

As a result of the above reactions, a variety of chromone derivatives bearing various heterocyclic systems were obtained from the condensation of 3-formylchromones with some active methylene compounds either under acidic or basic conditions.

### 2.1.2. Condensation reactions with acyclic active methylene compounds

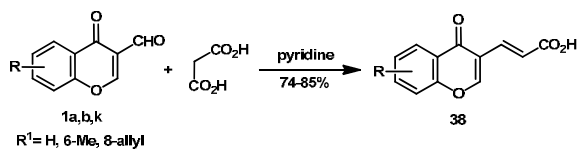
3-Styrylchromone **37**, which is associated with important biological activities, was obtained by the condensation of 4-nitrotoluene or 4-nitrophenylacetic acid **36** with 3-formylchromone **1a** in dry pyridine (Scheme 16) [34,35].



Scheme 16

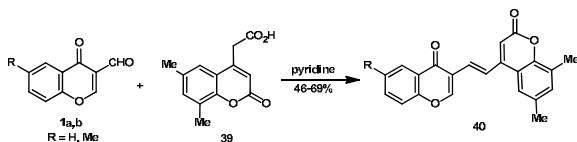
Synthesis of *trans*β-(chromon-3-yl)acrylic acids **38** were made by simple Knoevenagel condensation of 3-formyl-

chromones **1a,b,k** with malonic acid in dry pyridine under reflux (Scheme 17) [17,36-38].



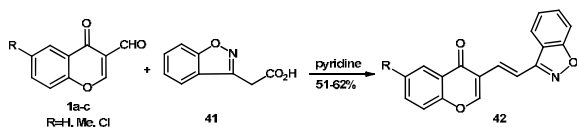
Scheme 17

6,8-Dimethylcoumarin-4-acetic acid (**39**) gave 3-styrylchromones **40** when reacted with 3-formylchromones **1a,b** in boiling pyridine, via condensation followed by decarboxylation under the reaction conditions (Scheme 18) [39].



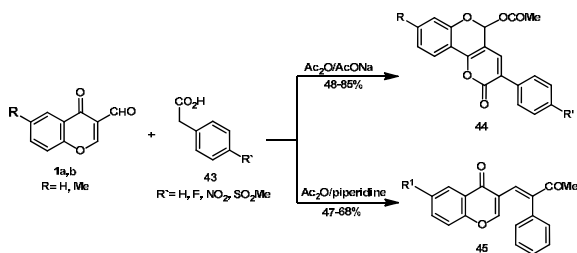
Scheme 18

In the same manner, 3-formylchromones **1a-c** reacted with benzo[d]isoxazol-3-ylacetic acid (**41**) in dry pyridine under reflux to give 3-(2-benzo[d]isoxazol-3-ylvinyl)-chromon-4-ones **42** in 51-62% yields (Scheme 19) [40].



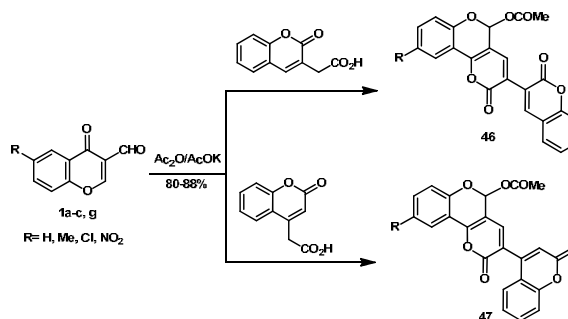
Scheme 19

Heating equimolar quantities of 3-formylchromones **1a,b** and phenylacetic acids **43** in acetic anhydride containing catalytic amounts of sodium acetate or potassium carbonate gave methyl 2-oxo-2*H,5H*-pyrano[3,2-*c*]chromen-5-yl acetates **44** in 48-85% yields (Scheme 20), [41-43] while Shingare *et al.* [44] reported the formation of compounds **45** in 47-68% yields in acetic anhydride containing piperidine as a catalyst, the spectral data showed the presence of acetyl instead of the carboxy group, which could be explained by decarboxylation followed by acetylation *in situ* (Scheme 20).



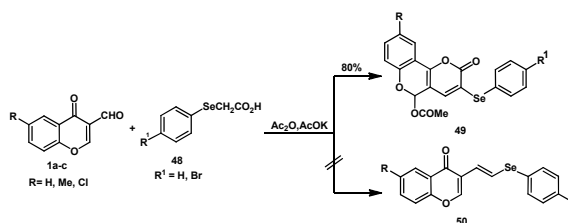
Scheme 20

Condensation of 3-formylchromones **1a-c,g** with 3- or 4-coumarinylacetic acids in acetic anhydride in the presence of potassium acetate either by heating at 90-100 °C or by microwave irradiation produced compounds **46** and **47**, respectively (Scheme 21) [41].



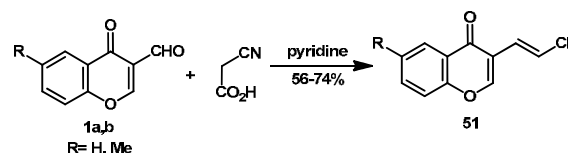
Scheme 21

Reactions of 3-formylchromones **1a-c** with 4-substituted phenylselenenylacetic acids **48** in acetic anhydride containing potassium acetate yielded 3-(phenylselenenyl)-2-oxo-2*H,5H*-pyrano[3,2-*c*]chromen-5-yl acetates **49** and not the other expected products **50** (Scheme 22) [45].



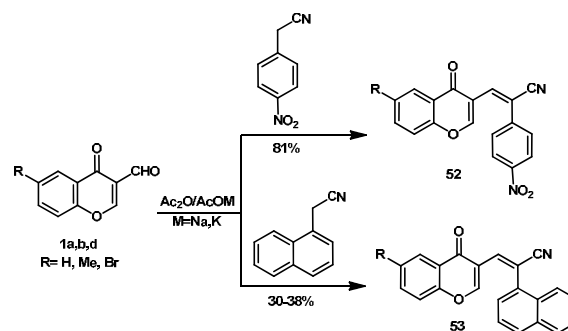
Scheme 22

Synthesis of *trans*- $\beta$ -(4-oxo-4*H*-chromen-3-yl)acrylonitrile **51** was made by simple condensation of 3-formylchromones **1a,b** with cyanoacetic acid in dry pyridine under reflux (Scheme 23) [46,47].



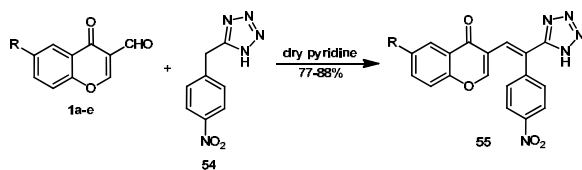
Scheme 23

Condensation of 3-formylchromones **1a,b,d** with *p*-nitrobenzyl cyanide and 1-naphthyl-acetonitrile in the presence of  $\text{Ac}_2\text{O/AcOK}$  or  $\text{Ac}_2\text{O/AcONa}$  afforded the corresponding condensation products **52** and **53**, respectively (Scheme 24) [48].



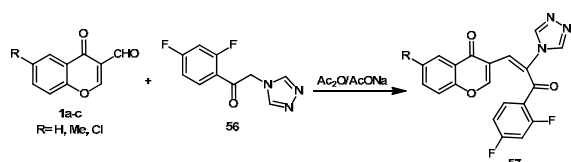
Scheme 24

Similarly, condensation of 3-formylchromones **1a**, with tetrazolyl-*p*-nitrotoluene (**54**) in dry pyridine gave 3-[2-(4-nitrophenyl)-2-(1*H*-tetrazol-5-yl)vinyl]-4*H*-chromen-4-one (**55**) (Scheme 25) [49].



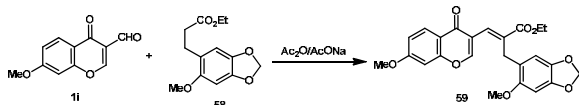
Scheme 25

3-Formylchromones **1a-c** condensed with 1-(2,4-difluorophenyl)-2-[1,2,4]triazol-4-yl)ethanone (**56**) in acetic anhydride containing anhydrous sodium acetate to afford 3-[3-(2,4-difluorophenyl)-3-oxo-2-[1,2,4]triazol-4-yl]propenyl]chromen-4-ones (**57**) (Scheme 26) [50].



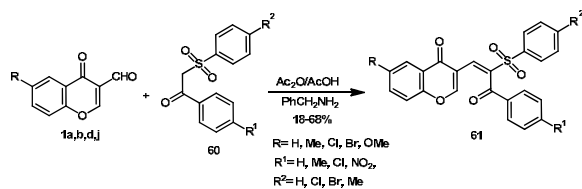
Scheme 26

Knoevenagel condensation of 3-formyl-7-methoxychromone **1i** with ethyl 3-(6-methoxy-1,3-benzodioxol-5-yl)propanoate **58** gave ethyl ester **59** (Scheme 27) [51].



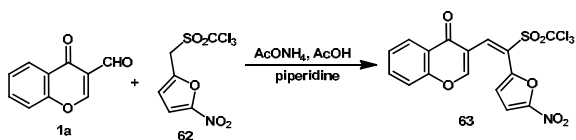
Scheme 27

Condensation of 3-formylchromones **1a,b,d,j** with phenacyl aryl sulfones **60**, in glacial acetic acid and acetic anhydride in the presence of benzylamine as catalyst, yielded 1-(aryl)-1-(arylsulfonyl)-2-[4-oxo-4*H*-chromen-3-yl]ethenes **61** (Scheme 28) [52].



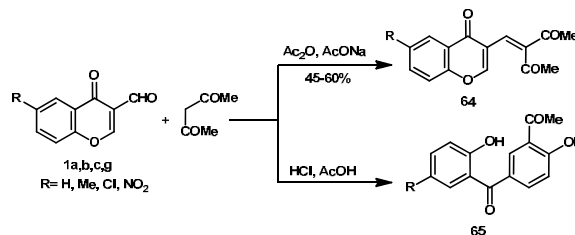
Scheme 28

When 3-formylchromone (**1a**) was treated with 5-nitro furyl trichloromethyl sulfone (**62**), in glacial acetic acid in the presence of ammonium acetate and piperidine, gave the condensation product **63** (Scheme 29) [52].



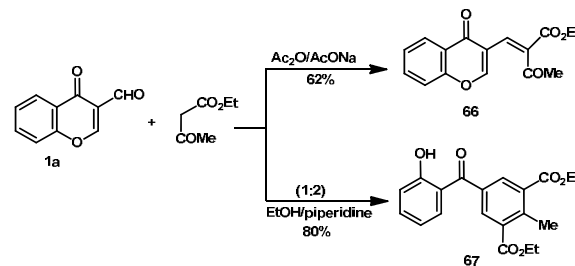
Scheme 29

Condensation product **64** was synthesized by reaction of 3-formylchromones **1a,b,c,g** and 2,4-pentanedione in acetic anhydride containing sodium acetate. While, acid catalyzed 1,4-addition of the enol form of 2,4-pentanedione to 3-formylchromones **1a,b,c,g** followed by ring opening and enolization afforded 5-benzoyl-2-hydroxyacetophenones **65** (Scheme 30) [53,54].



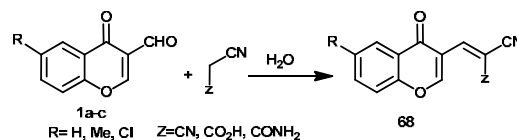
Scheme 30

Treatment of 3-formylchromone (**1a**) with ethyl acetoacetate in acetic anhydride containing sodium acetate yielded 3-(4-oxo-4*H*-1-chromen-3-yl)-2-(1-oxoethyl)-2-propenoic acid ethyl ester (**66**) in 62% yield. However, when the reaction was carried out with a (1:2) excess of the reagent using piperidine in ethanol, 5-(2-hydroxybenzoyl)-2-methylbenzene-1,3-dicarboxylic acid diethyl ester (**67**) was isolated in 80% yield (Scheme 31) [54,55].



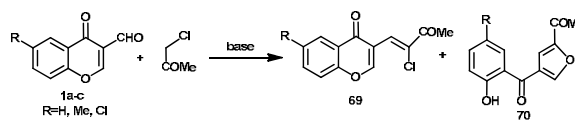
Scheme 31

Knoevenagel condensation of 3-formylchromones **1a-c** with malononitrile, cyanoacetic acid and cyanoacetamide were carried out in various solvents such as water, ethanol, methanol, DMF, DMSO and toluene (Scheme 32). Among these solvents, water was found to be the best solvent for the reaction to produce ylidenenitriles **68** in excellent yields [56].



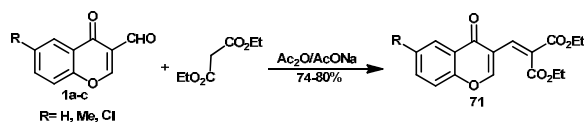
Scheme 32

Reaction of aldehyde **1a-c** with chloroacetone in basic medium gave a mixture of arylidene **69** (22-32%) and *o*-hydroxyphenyl furyl ketone **70** (Scheme 33) [57].



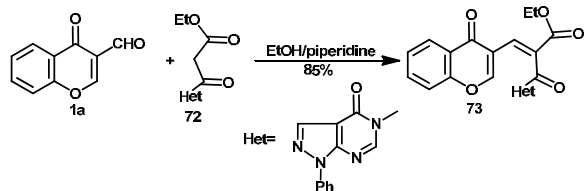
Scheme 33

Condensation of 3-formylchromones **1a-c** with diethyl malonate in acetic anhydride containing sodium acetate gave diethyl arylidene ester **71** in 74-80% yield (Scheme 34) [58].



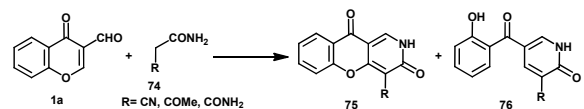
Scheme 34

Treatment of 3-formylchromone (**1a**) with 3-(4',5'-dihydro-4'-oxo-1'-phenylpyrazolo[3,4-*d*]pyrimidin-5'-yl)-3-oxopropionic acid ethyl ester (**72**) in ethanol containing piperidine afforded 3-((4-oxo-4*H*-chromen-3-yl)-2-(4,5-dihydro-4-oxo-1-phenylpyrazolo[3,4-*d*]pyrimidin-5'-carbonyl)acrylic acid ethyl ester (**73**) (Scheme 35) [59].



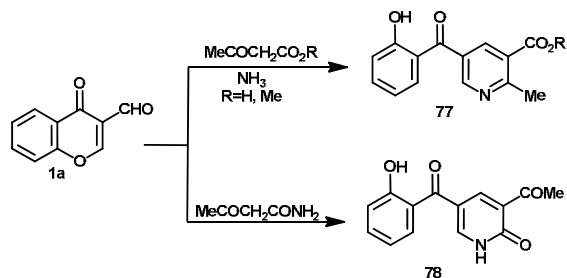
Scheme 35

3-Formylchromone (**1a**) reacted with acetamides **74** in dry pyridine to afford a mixture of chromenopyridones **75** and pyridone derivatives **76** (Scheme 36) [60,61].



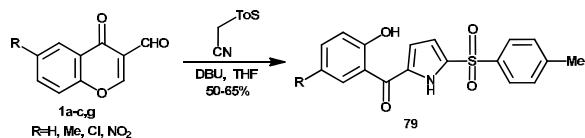
Scheme 36

3-Formylchromone **1a** condensed with alkyl acetoacetate or its acid in the presence of ammonia to give the pyridine derivatives **77** (46-50%). While, aldehyde **1a** condensed with acetoacetamide to produce the 2-pyridone derivative **78** (Scheme 37). The reaction involves ring opening of pyrone moiety at C2 [62-64].



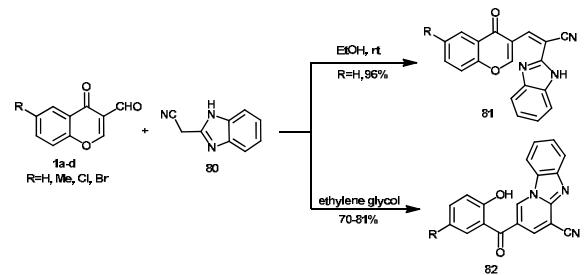
Scheme 37

When 3-formylchromones **1a-c,g** were allowed to react with equimolar amounts of tosylmethyl isocyanide (TOSMIC) in THF in the presence of mild base as 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) at room temperature, the 2-tosyl-5-(2-hydroxybenzoyl)pyrroles **79** were isolated in good yields (Scheme 38) [65].



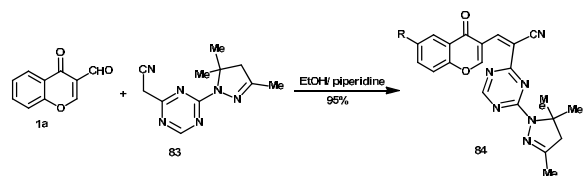
Scheme 38

Condensation of 3-formylchromones **1a-d** with 1*H*-benzimidazole-2-acetonitrile (**80**) in ethanol at room temperature gave the carbonitrile derivative **81** in 96%. When the reaction was carried out with in boiling ethylene glycol, 4-cyano-2-(2-hydroxybenzoyl)pyrido[1,2-*a*]benzimidazoles **82** were obtained in 70-81% yields (Scheme 39) [66,67].



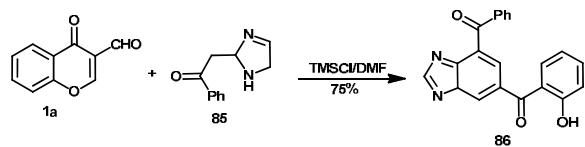
Scheme 39

Refluxing **1a** with pyrazolynltriazinylacetonitrile (**83**) in ethanol containing few drops of piperidine gave the condensation product **84** in 95% yield, as antitumor agent (Scheme 40) [68].



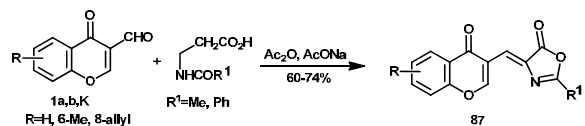
Scheme 40

Condensation of **1a** with imidazole derivative **85** in  $\text{Me}_3\text{SiCl}/\text{DMF}$  produced the imidazo[1,2-*a*]pyridine **86** in 75% yield (Scheme 41) [69].



Scheme 41

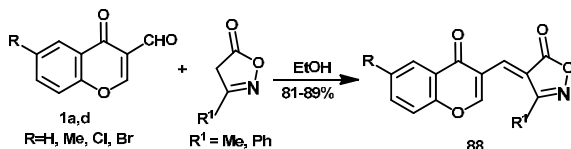
Oxazolones **87** were readily obtained from the reaction of 3-formylchromones **1a,b** and *N*-acetyl/ benzoylglycine in acetic anhydride containing freshly fused sodium acetate (Scheme 42) [17,70,71].



Scheme 42

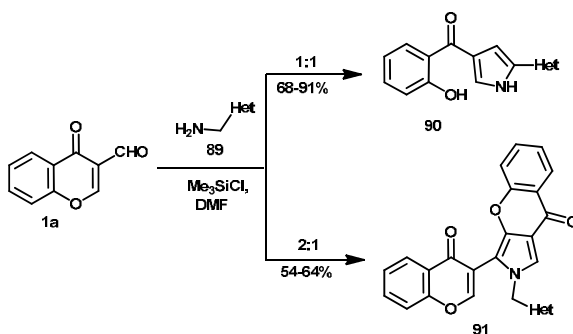
On the other hand, the isoxazolone derivatives **88** were obtained in good yields (81-89%) from the Knoevenagel condensation of 3-formylchromones **1a-d** with 3-methyl (or

phenyl)-5-isoxazolone in ethanol at room temperature (Scheme 43) [72].



Scheme 43

3-Formylchromone **1a** reacted with hetarylmethylamines **89** in DMF under heating by molar ratio 1:1 in the presence of 4 molar equivalents of Me<sub>3</sub>SiCl (trimethylsilyl chloride) to give 5-hetaryl-[1*H*-pyrrol-3-yl](2-hydroxyphenyl)methanones **90** in 68-91% yields. When the reaction carried out in molar ratio 2:1 (3-formylchromone: amines), the fused chromonopyrrolyl chromones **91** were obtained in 54-64% yields (Scheme 44) [73].



Het= benzimidazolyl, pyridyl, methylthiazolyl, pyridotriazolyl, benzothiazolyl, hydroxypyridyl, *N*-methylbenzimidazolyl, *N*-methyltriazolyl

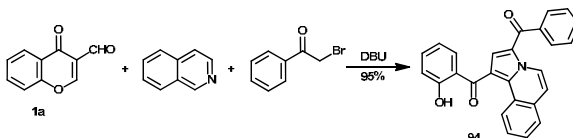
Scheme 44

Cyclocondensation of 3-formylchromone **1a** with glycine derivatives **92** in the presence of Me<sub>3</sub>SiCl (4 equivalents) afforded pyrrole derivatives **93** in moderated yield (Scheme 45) [73].



Scheme 45

A high yield of the novel pyrrolo[2,1-*a*]isoquinoline derivative **94** was prepared from the reaction of 3-formylchromone **1a** with isoquinoline and phenacyl bromide in aqueous medium containing DBU as a catalyst as reported by Naskar *et al.* (Scheme 46) [74].

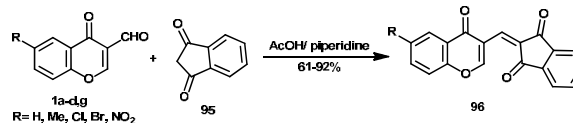


Scheme 46

Therefore, condensation of 3-formylchromones with acyclic active methylene compounds produced the corresponding condensation products or a variety of products depending on the reagent used and the reaction conditions.

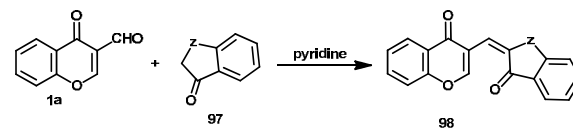
### 2.1.3. Condensation reactions with cyclic active methylene compounds

Condensation of 3-formylchromones **1a-dg** with 1,3-indandione **95** in glacial acetic acid containing piperidine afforded the condensation products **96** in 61-92% yields (Scheme 47) [75].



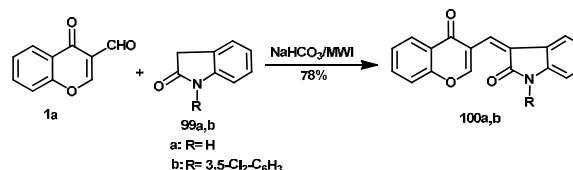
Scheme 47

Treatment of 3-formylchromone **1a** with 3-oxo-2,3-dihydrobenzo[*b*]thiophene-1,1-dioxide **97a** and oxindole (indolone) **97b** in dry pyridine afforded the corresponding condensation products **98** (Scheme 48) [76].



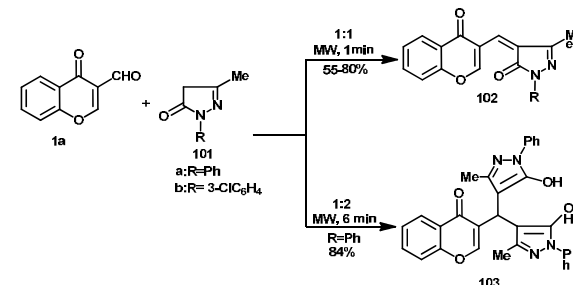
Scheme 48

Simple condensation of aldehyde **1a** with indolinone **99a,b** in the presence of sodium bicarbonate under microwave irradiation yielded (1*H*)-3-(4-oxo-4*H*-chromen-3-yl) methylene] indolin-2-one (**100a**) and 1-[(2,6-dichlorophenyl)-3-(4-oxo-4*H*-chromen-3-yl)methylene] indolin-2-one (**100b**), respectively (Scheme 49) [76,77].



Scheme 49

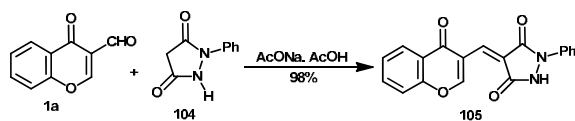
Condensation reaction of **1a** and pyrazolone derivatives **101a,b** in 1:1 molar ratio, under classical method or microwave irradiation, afforded 1-aryl-3-methyl-4-(4-oxo-4*H*-chromen-3-yl)methylene]pyrazol-5(4*H*)-ones (**102a,b**), [77,78] while using 1:2 molar ratio afforded the new 1-(chromon-3-yl)-1,1-bis(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)methane **103** (Scheme 50) [79].



Scheme 50

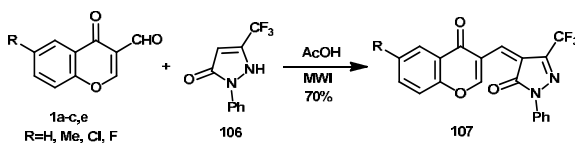
Condensation of 3-formylchromone (**1a**) with 1-phenylpyrazolidine-3,5-dione (**104**) in glacial acetic acid containing freshly fused sodium acetate, gave 4-[(4-oxo-4*H*-

chromen-3-yl)methylene]-1-phenyl-pyrazolidine-3,5-dione (**105**) (Scheme 51) [80].



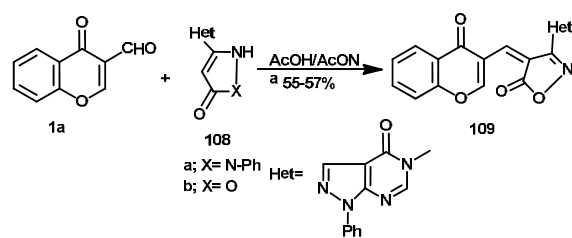
Scheme 51

Also, treatment of 3-formylchromones **1a-c,e** with 3-(trifluoromethyl)-1-phenyl-1H-pyrazol-5(4H)-one (**106**) in acetic acid yielded 3-(trifluoromethyl)-4-[(6-substituted 4-oxo-4H-chromen-3-yl)methylene]-1-phenyl-1H-pyrazol-5(4H)-ones **107** in 70% yield (Scheme 52) [81].



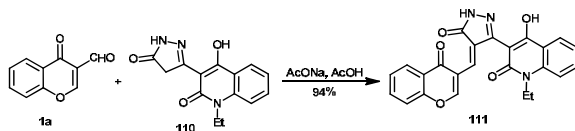
Scheme 52

Condensation of **1a** with pyrazolinone **108a** and isoxazolinone **108b** in acetic acid containing fused sodium acetate gave 5-{4-[(4-oxo-4H-chromen-3-yl)methylene-5-oxo-4,5-dihydro-1H-pyrazol-3-yl]}-1-phenylpyrazolo[3,4-d]pyrimidin-4-one **109a** and 5-{4-[(4-oxo-4H-chromen-3-yl)methylene-5-oxo-4,5-dihydroisoxazol-3-yl]}-1-phenylpyrazolo[3,4-d]pyrimidin-4-one **109b**, respectively (Scheme 53) [59].



Scheme 53

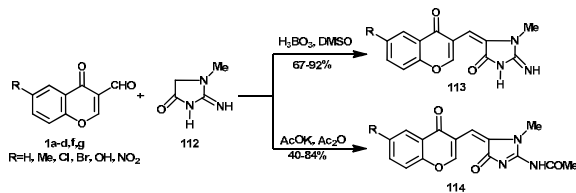
Similarly, condensation of 3-formylchromone (**1a**) with pyrazol-3-ylquinoline derivative **110** in glacial acetic acid containing sodium acetate afforded chromenylpyrazolinone **111** (Scheme 54) [82].



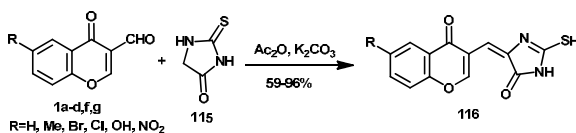
Scheme 54

Also, condensation of **1a-d,f,g** with creatinine **112** in DMSO using boric acid as a catalyst yielded 2-imino-1-methyl-5-(6-substituted 4-oxo-4H-chromen-3-yl)methylidene]imidazolin-4-one (**113**). While, 2-acetamido-1-methyl-5-[(6-substituted 4-oxo-chromen-3-yl)methylidene]-4,5-dihydroimidazol-4-one **114** was obtained when the reaction took place in acetic anhydride and potassium acetate (Scheme 55) [83].

3-Formylchromones **1a-d,f,g** condensed with thiohydantoin **115** in acetic anhydride in the presence of potassium acetate under both irradiation and classical condition yielded 2-thioxo-5-[(6-substituted-4-oxo-chromen-3-yl)methylidene]imidazolidine-4-ones (**116**) (Scheme 56) [83].

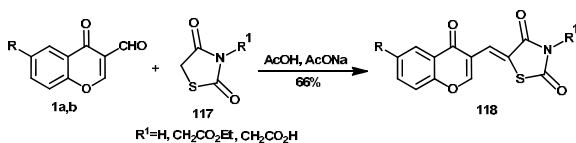


Scheme 55



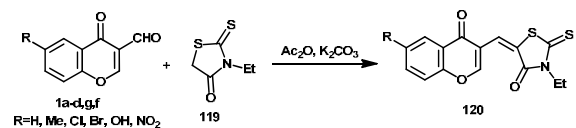
Scheme 56

Chromonyl-2,4-thiazolidinediones **118** were prepared by the Knoevenagel condensation of 2,4-TZDs (2,4-thiazolidine diones) **117** with 3-formylchromones **1a,b,n** in glacial acetic acid containing freshly fused sodium acetate (Scheme 57) [84-86].



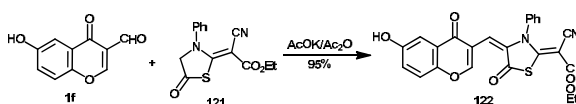
Scheme 57

Similarly, condensation of 3-formylchromone **1a-d,f,g** with 3-ethylrhodanine **119** in acetic anhydride/potassium acetate under both irradiation and classical condition, gave 2-thioxo-5-[(6-substituted-4-oxo-4H-chromen-3-yl)methylidene]thiazolidin-4-ones **120** (Scheme 58) [83].



Scheme 58

Condensation of carboxaldehyde **1f** with ethyl 2-cyano-2-(3-phenyl-5-oxo-1,3-thiazolan-2-ylidene)acetate (**121**) gave the corresponding arylidene derivative **122** (Scheme 59) [61].

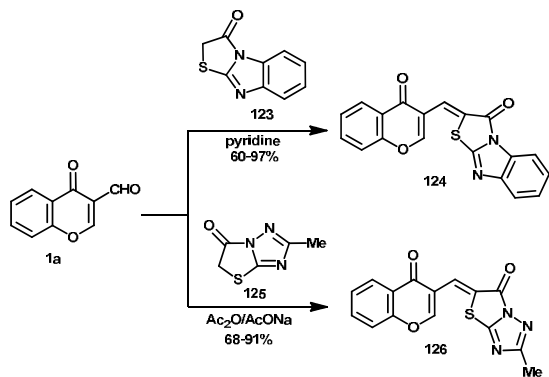


Scheme 59

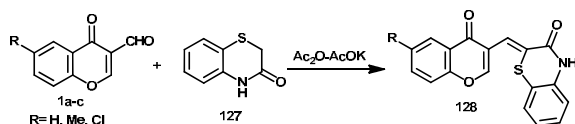
Reaction of 3-formylchromone (**1a**) with [1,3]thiazolo[3,2-a]benzimidazol-3(2H)-one (**123**) in glacial acetic acid containing fused sodium acetate afforded the condensation product **124** in high yield (60-97%) [87]. Similarly, treating **1a** with 2-methyl[1,3]thiazolo[1,2,4]triazol-5(6H)-one (**125**) in acetic anhydride/sodium acetate yielded the condensation product **126** in 68-91% yields (Scheme 60) [88].

Knoevenagel products **128** were obtained in low yields (15-43%) by heating 3-formylchromones **1a-c** with 2H-1,4-benzothiazin-3(4H)-one (**127**) in acetic anhydride/potassium acetate medium for 6-10 h. Using microwave the yields were increased (33-62%) in short time (7-20 min) (Scheme 61) [87].



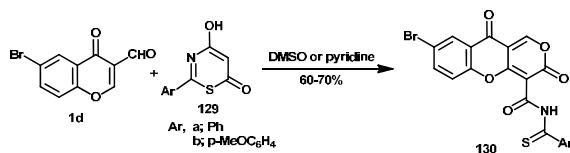


Scheme 60



Scheme 61

Reaction of 6-bromo-3-formylchromone **1d** with 2-aryl-4-hydroxy-6*H*-1,3-thiazin-6-ones **129a,b** in DMSO or pyridine gave 8-bromo-3,10-dioxo-*N*-(arylcarbonothioyl)-4,4a-dihydro-3*H*,10*H*-pyrano[4,3-*b*]-chromene-4-carboxamides (**130a,b**) in 60-70% yields [Scheme 62] [89].



Scheme 62

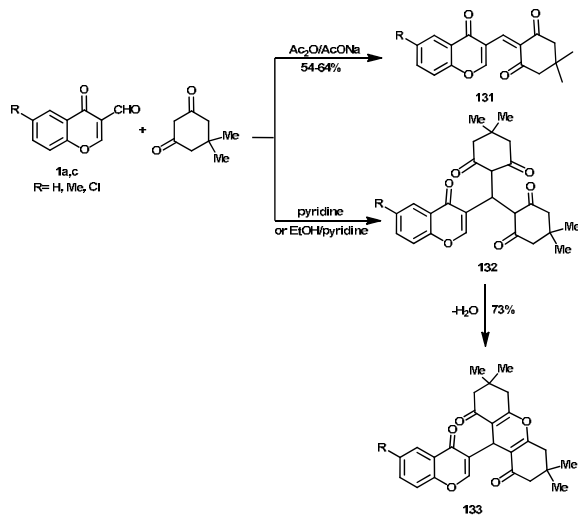
Also, condensation of 3-formylchromone **1a** with dimedone (5,5-dimethylcyclohexane-1,3-dione) in acetic anhydride containing sodium acetate yielded the condensation products **131** in 54-64% [87]. When the reaction took place in aqueous ethanol containing pyridine or aqueous pyridine by molar ratio (1:2) afforded the adduct **132** which dehydrated to form xanthane **133** in 73% yield [Scheme 63] [60,76,90].

Meldrum's acid (2,2-dimethyl-1,3-dioxane-4,6-dione) undergoes Knoevenagel condensation with 3-formylchromone **1a-c,e** producing the corresponding condensation product derivatives **134** in 90-95% yields [Scheme 64] [91-93].

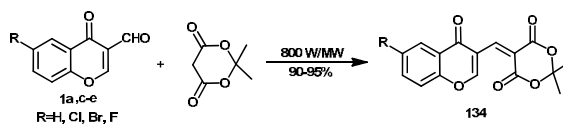
Reaction of 3-formylchromones **1a,c** with 4-chromanone **135** in ethanol containing triethylamine gave benzopyrano-2,3-dihydrobenzopyranones **136** in 67-80% yields [Scheme 65] [94].

Treatment of 3-formylchromone (**1a**) with barbituric acid **137a**, thiobarbituric acids **137b** and 1,3-dimethylbarbituric acid **138** gave 5-[(4-oxo-4*H*-chromen-3-yl)methylene]pyrimidine derivatives **139** and **140**, respectively [Scheme 66] [76,77].

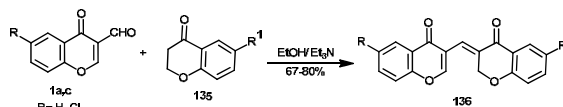
*N*-Methylpyrrole reacted with 3-formylchromones **1a,c,d,g** under solvent-free conditions exclusively via 1,4-addition followed by recyclization to form 2-hydroxy-3-(1-methylpyrrol-2-ylmethylene)chroman-4-ones **141** in good yield [Scheme 67] [95].



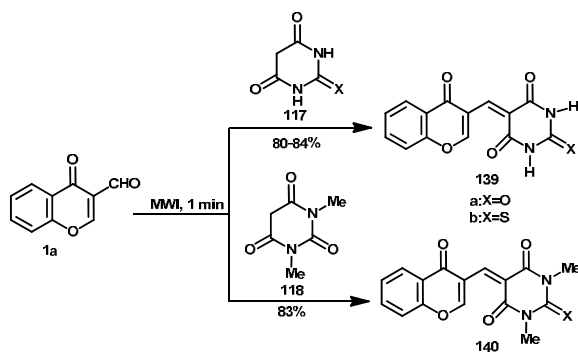
Scheme 63



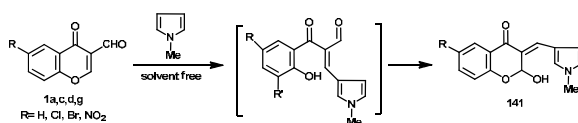
Scheme 64



Scheme 65

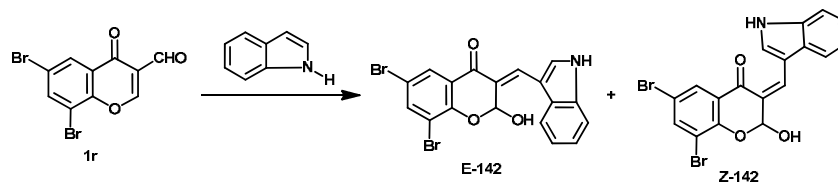


Scheme 66

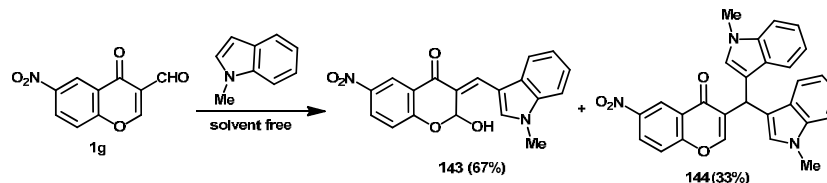


Scheme 67

A similar reaction of 6,8-dibromo-3-formylchromone (**1r**) with indole proceeded exclusively via 1,4-addition followed by recyclization to form a mixture of *E*-isomer **142** (91%) and *Z*-isomer **142** (9%) [Scheme 68] [95].



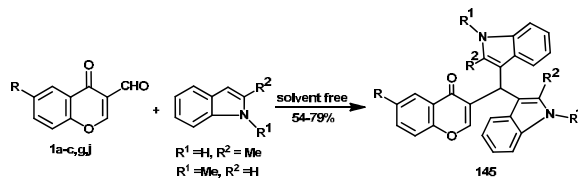
Scheme 68



Scheme 69

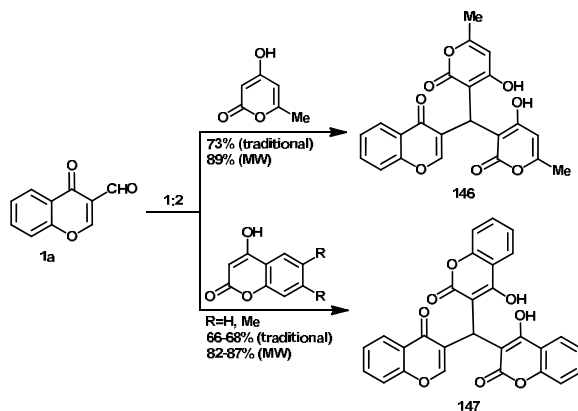
Reaction of 3-formyl-6-nitrochromone (**1g**) with *N*-methylindole afforded a mixture of *E*-(2-hydroxy-6-nitro-3-(1-methylindol-3-ylmethylene)chroman-4-one (**143**) and the *bis*-adduct **144** [Scheme 69] [95].

On the other hand, 3-formylchromones **1a-c,g,j** reacted without solvent with excess indole, 1-methyl or 2-methylindoles (2 equivalents) to give (chromon-3-yl)-*bis*-(indol-3-yl)methanes **145** [Scheme 70] [95].



Scheme 70

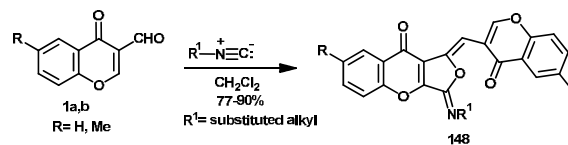
Also, 3-formylchromones **1a** reacted with triacetic acid lactone and 4-hydroxycoumarin derivatives (2 equivalents), under conventional and solvent free methods, to give (chromon-3-yl)-*bis*-(hetaryl)methanes **146** and **147**, respectively [Scheme 71] [79].



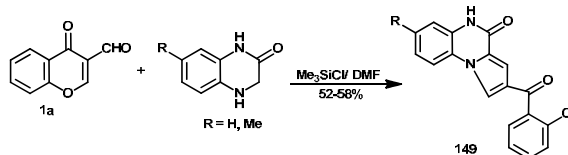
Scheme 71

Condensation of 3-formylchromones **1a,b** with alkyl isocyanides in dry dichloromethane at room temperature afforded (1*Z*)-3-(alkylimino)-1-[(chromon-3-yl)methylene]-1,3-dihydro-9*H*-furo [3,4-*b*]chromen-9-ones **148** in good yields (77-90%) [Scheme 72] [96].

The reaction of 3-formylchromone **1a** with 3,4-dihydroquinoxalin-2(1*H*)-ones gave 2-(2-hydroxy-benzoyl)pyrrolo[1,2-*a*]quinoxalin-4(5*H*)-ones **149** in 52-58% yields, respectively [Scheme 73] [73].

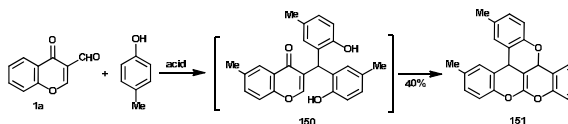


Scheme 72



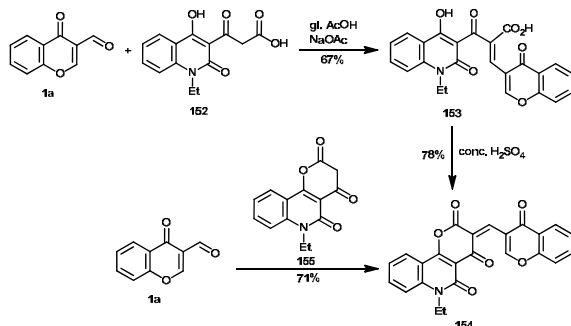
Scheme 73

Under acidic conditions, *p*-cresol underwent 1,2-addition through C-2 atom to the aldehyde function of 3-formylchromone (**1a**) to produce intermediate **150** which, converted to 2,14-dimethyl-10*aH*,15*aH*-tribenzo[*b,e,i*][1,6,7] trioxaphenylene (**151**) [Scheme 74] [97].



Scheme 74

Treating carboxaldehyde **1a** with  $\beta$ -ketoacid **152**, [98] in glacial acetic acid freshly fused sodium acetate, yielded the Knoevenagel condensation products **153** which underwent dehydration by stirring in concentrated  $H_2SO_4$  at room temperature to afford 3-chromonylmethylenepyran[3,2-*c*]quinoline derivatives **154**. Moreover, compound **153** was obtained authentically from condensation reaction of pyrano [3,2-*c*]quinoline **155** with **1a** [Scheme 75] [99].



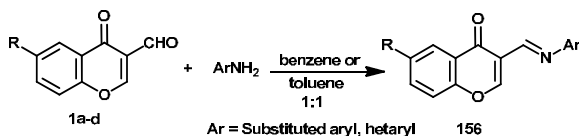
Scheme 75

Thus, a large number of chromone derivatives linked a variety of heterocyclic systems were prepared from the direct condensation of 3-formylchromones with heterocyclic compounds containing active methylene groups.

## 2.2. Chemical reactivity of 3-formylchromones towards nitrogen nucleophiles

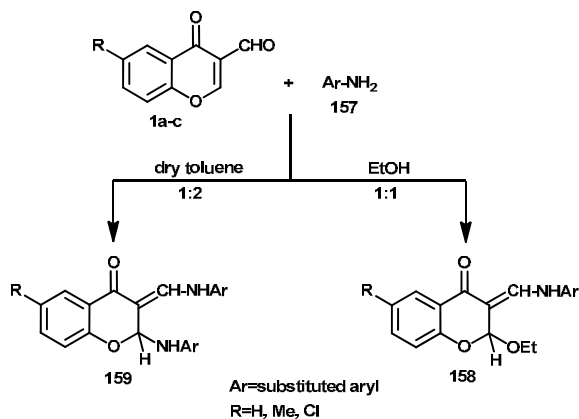
### 2.2.1. Reactions with primary amines

A large group of 3-(aryl/heteroarylaminomethyl)chromone derivatives **156** were prepared from condensation reactions of 3-formylchromones **1a-d** with a variety of aryl/hetarylamines in non polar solvents such as benzene, toluene or xylene under reflux, in the presence of *p*-toluenesulfonic acid as a catalyst (Scheme 76) [100-108].



Scheme 76

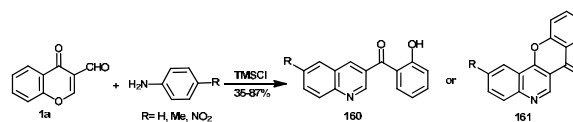
On the other hand, reaction of 3-formylchromones **1a-c** with equimolar ratio of aryl/hetarylamines **157** in boiling ethanol gave 3-(aryl/hetarylamino)methylene-2-(ethoxy)chromones **158**, but when the reaction took place with two moles of **157** in dry toluene, the 1,4-adducts **159** were obtained (Scheme 77) [105-115].



Scheme 77

Reaction of 3-formylchromone **1a** with various anilines in the presence of Me<sub>3</sub>SiCl/DMF led to either 3-(2-hydroxybenzoyl)quinolines **160** or 7*H*-chromeno[3,2-*c*]quinolin-7-ones **161** (Scheme 78), depending on the

structure of the starting aniline. Substituents in the aniline molecule that withdraw electrons favored the formation of **161**; on the contrary, electron-rich anilines gave only **160** [116,117].

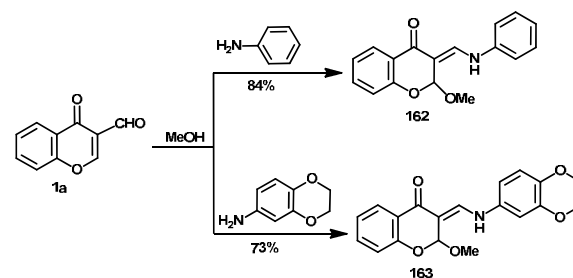


Scheme 78

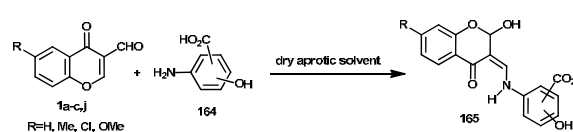
Condensation of 3-formylchromone (**1a**) with aniline and 6-amino-1,4-benzoxodioxane in methanol gave 3-(aniline methylene)-2-methoxychroman-4-one (**162**) and 3-(1,4-benzodioxane-6-aminomethylene)-2-methoxychroman-4-one (**163**), respectively (Scheme 79) [118].

Also, reaction of 6-substituted 3-formylchromone **1a-cj** with aromatic amino carboxylic acid **164** in benzene or toluene yielded only 3-(arylamino)methylene-2-hydroxychromon-4-ones **165** at room temperature or under reflux (Scheme 80) [113].

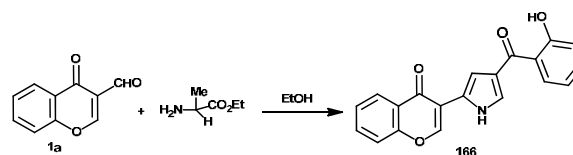
Treatment of 3-formylchromone **1a** with ethyl 2-amino-propanoate yielded pyrrole derivative **166** in high yield (Scheme 81) [119].



Scheme 79

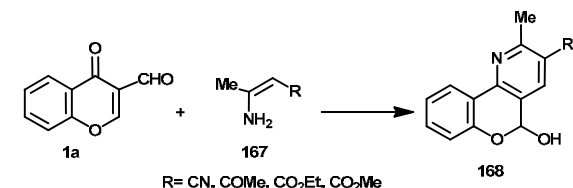


Scheme 80

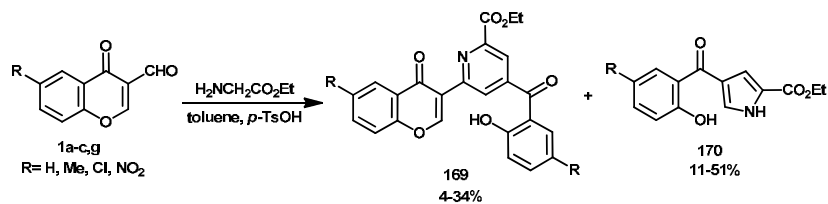


Scheme 81

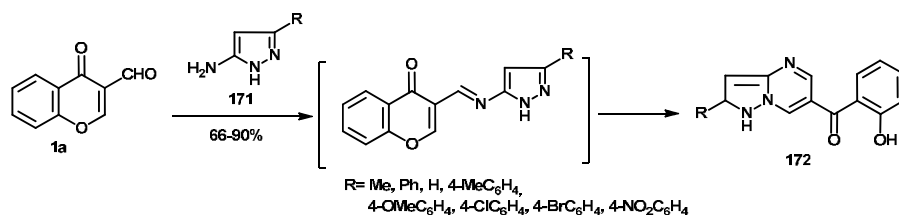
Chromenopyridines **168** were prepared by treating 3-formylchromone **1a** with enamionitriles, enamionketones or  $\beta$ -aminoesters **167** (Scheme 82) [63,64,76,120].



Scheme 82



Scheme 83

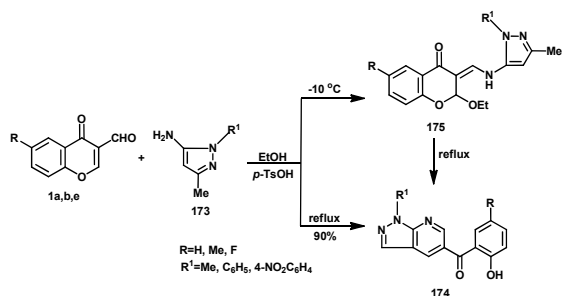


Scheme 84

Interaction of 3-formylchromones **1a-c,g** with ethyl glycinate in boiling toluene in the presence of *p*-toluenesulfonic acid gave a mixture of ethyl 4-(2-hydroxybenzoyl)-6-(4-oxochromen-3-yl)-pyridine-2-carboxylates **169** and ethyl 4-(2-hydroxybenzoyl)pyrrole-2-carboxylates **170** (Scheme 83) [62,65,76,121].

Also, the reaction of 3-formylchromone (**1a**) with equimolar amount of aminopyrazoles **171** in absolute ethanol afforded 6-(2-hydroxybenzoyl)pyrazolo[1,5-*a*]pyrimidines **172**, via iminomethyl derivative (Scheme 84) [122].

Treatment of 3-formylchromones **1a,b,e** with 5-amino pyrazoles **173** in boiling ethanol containing *p*-toluenesulfonic acid gave 2-hydroxybenzoylpyrazolo[3,4-*b*]pyridines **174**. While, when this reaction was carried out at lower temperature (-10 °C), the enamine-adducts **175** were isolated. Enamine-adducts rearrange into pyrazolo[3,4-*b*]pyridines **174** after prolonged refluxing (Scheme 85) [123].



Scheme 85

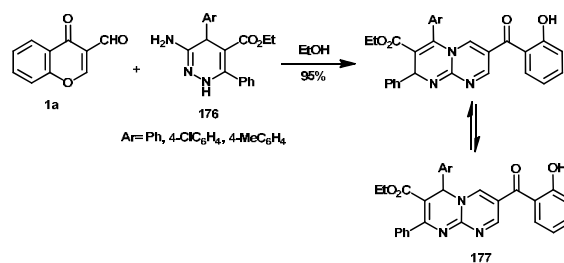
Reaction of 3-formylchromone **1a** with ethyl 2-aminopyrimidine carboxylates **176** in ethanol gave ethyl 7-(2-hydroxybenzoyl)-2*H*-(4*H*)pyrimido[1,2-*a*]pyrimidine-3-carboxylate **177** (Scheme 86) [124].

On the other hand, aminopyrimidones **178** reacted with aldehyde **1a** in refluxing ethanol to afford the unexpected pyrido[2,3-*d*]pyrimidines **179** but not the predicted pyridopyrimidines **180** (Scheme 87) [125].

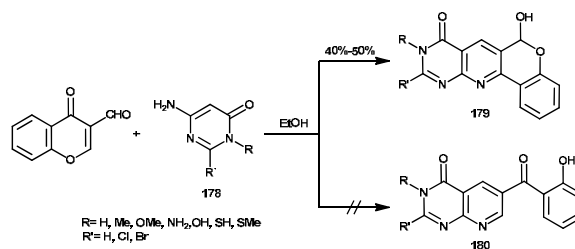
Acetic acid catalyzed condensation of aldehyde **1a** with 1-(2-aminophenyl)pyrrole (**181**) led to the pyrrolo[1,2-*a*]quinoxaline derivative **182** (Scheme 88) [126].

2-(4*H*-4-Chromen-3-yl)-3-aryl-1,2-dihydroquinazolin-4(3*H*)-ones **184** are the final products from the reaction of

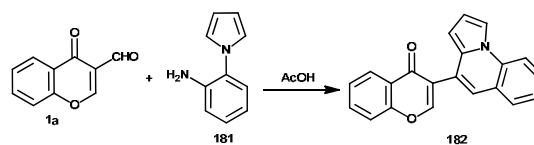
carboxaldehyde **1a** and amino amides **183** in both ethanolic and nonpolar medium (Scheme 89) [127].



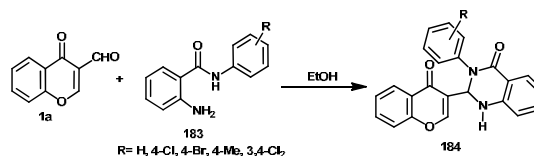
Scheme 86



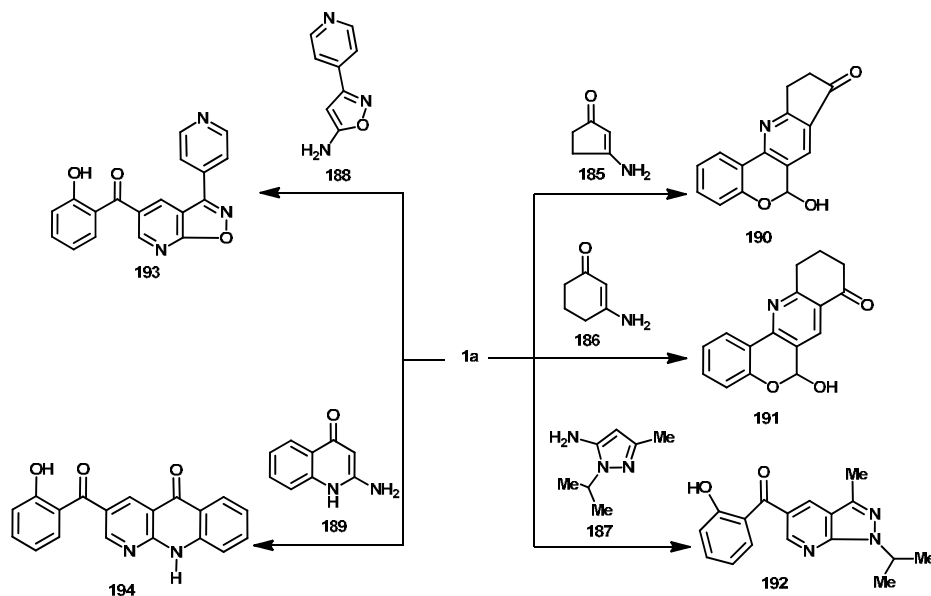
Scheme 87



Scheme 88



Scheme 89

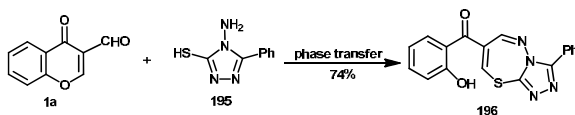


Scheme 90

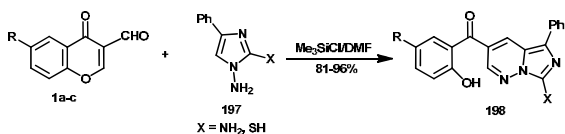
Reaction of 3-formylchromone **1a** with enamine derivatives **185-189** in pyridine or glacial acetic acid led to fused pyridine derivatives **190-194** in one step, respectively (46-82% yields) (Scheme 90) [76].

Reaction of 3-formylchromone **1a** with 4-amino-3-mercapto-5-phenyl-1,2,4-triazole (**195**) under phase transfer condition yielded ring-opened thiadiazepine **196** (Scheme 91) [128].

Condensation of **1a-c** with 4-phenyl-1*H*-imidazole-1-amines **197** in the presence of Me<sub>3</sub>SiCl/DMF gave imidazo[1,5-*b*]pyridazines **198** in 81-96% yields (Scheme 92) [129].

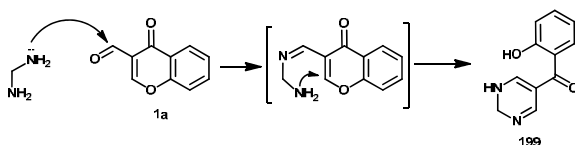


Scheme 91



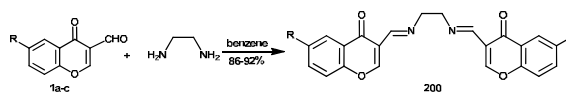
Scheme 92

Diaminomethane reacted with 3-formylchromone **1a** to yield pyrimidine derivative **199**. The reaction initially takes place on the formyl group and is followed by an intramolecular attack of the second amine function on the C-2 atom of the pyrone ring followed by the ring opening (Scheme 93) [130].



Scheme 93

Condensation of equimolar amounts of **1a-c** with ethylenediamine in benzene gave the 1,2-*bis*[(chromon-3-yl)methyleneamino]ethane **200**, using excess of ethylenediamine afforded poor yield of 1:1 condensation product (Scheme 94) [130].



Scheme 94

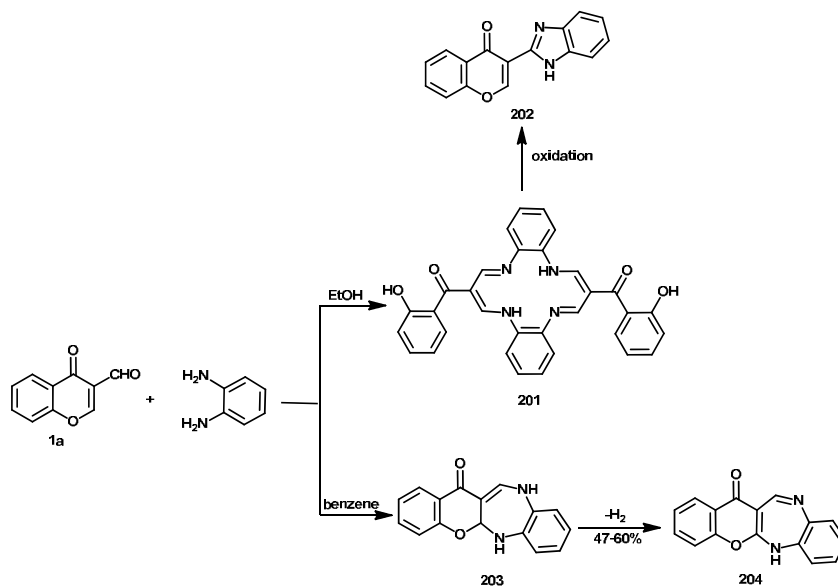
Reaction of aldehyde **1a** with *o*-phenylenediamine in boiling ethanol gave 7,16-*bis*-(2-hydroxybenzoyl)-5,14-dihydrobenzo[*b-f*][1,4,8,11]tetraazacyclotetradecine (**201**), which then oxidized to 3-(2-benzimidazolyl)chromone **202**, while in boiling benzene, benzodiazepino chromone **204** was obtained during dehydrogenation of the initially formed dihydro analogous **203**, through air oxidation or boiling in nitrobenzene (Scheme 95) [130-141].

On the other hand, cyclocondensation of 6-chloro-3-formylchromone (**1c**) with diaminopyridone derivative **205** in DMF under reflux containing few drops of piperidine afforded the 1,2,3,5-tetrahydro[1,2,4]triazolo[1,5-*a*]pyridine derivative **206** (Scheme 96) [142].

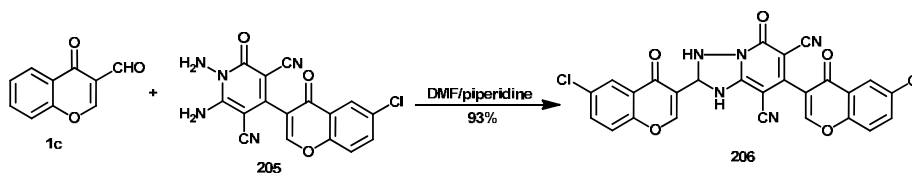
Condensation reaction of 6-chloro-3-formylchromone (**1c**) with *p*-phenylenediamine in dry benzene containing *p*-toluenesulfonic acid in 1:1 and 2:1 molar ratio gave 3-[(4-aminophenylimino)methyl]-6-chloro-4-oxo-4*H*-chromene (**207**) and the *bis* compound **208**, respectively (Scheme 97) [143].

On the other hand, condensation reaction of 3-formylchromone (**1a**) with 2-aminophenol and 2-aminothiophenol in ethanol afforded compound **209** via the formation of the simple condensation product followed by 1,4-addition of ethanol molecules (Scheme 98) [132].

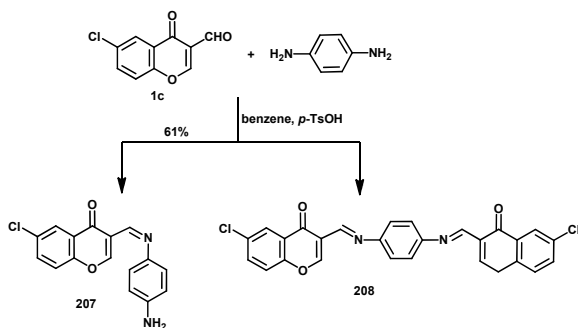
Consequently, the products obtained from the reaction of 3-formylchromones with primary amines depend mainly on the type of amine, molar ratio and the solvent used. Also, a diverse number of heterocyclic systems were obtained from the condensation of 3-formylchromones with bifunctional nucleophiles.



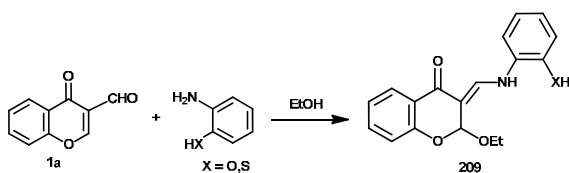
Scheme 95



Scheme 96

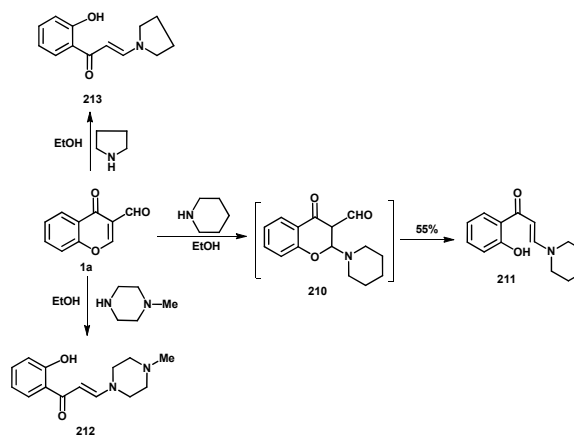


Scheme 97



Scheme 98

enaminoketone **211** in 55% yield [151]. Further, 3-formylchromones **1a** when heated with *N*-methylpiperazine in ethanol furnished 1-(2-hydroxyphenyl)-3-(4-methylpiperazin-1-yl)prop-2-en-1-one (**212**) [41]. Similarly, 1-(2-hydroxyaryl)-3-(pyrrolidin-1-yl)prop-2-en-1-one (**213**) was synthesized by the reaction of 3-formylchromones **1a** with pyrrolidine in dry ethanol (Scheme 99) [145,146].



Scheme 99

### 2.2.2. Reactions with secondary amines

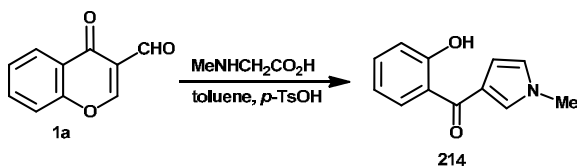
When 3-formylchromone **1a** treated with secondary amine like piperidine, an unstable 1,4-adduct **210** is formed, this adduct undergoes base catalyst deformylation to give the

Treatment of 3-formylchromone (**1a**) with *N*-methylglycine in boiling toluene in the presence of *p*-toluenesulfonic acid

produces *N*-methyl-3-salicyloyl pyrrole **214** in high yield (Scheme 100) [119].

Condensation of 3-formylchromone **1a** with *N*<sup>1</sup>,*N*<sup>2</sup>-dimethyl glycinamide (**215**) and *L*-pyrrolinamide **216** afforded chromonylimidazolone **217** (79% yield) and pyrroloimidazolone **218** (84% yield), respectively, these reactions proceed via [4+1] recyclization (Scheme 101) [73,147].

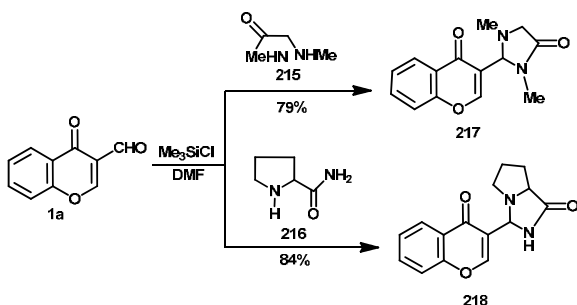
As a result, a variety of products were obtained from the reaction of 3-formylchromones with secondary amines depending on the nucleophile used.



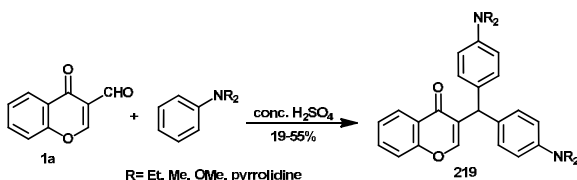
Scheme 100

### 2.2.3. Reactions with tertiary amines

The acid-catalyzed condensation of 3-formylchromone (**1a**) with a range of tertiary aromatic amines gave the 3-[bis(4-aminophenyl)methyl]chromones **219** in moderate yield (Scheme 102) [148].



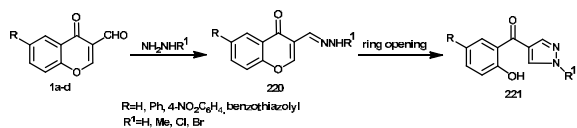
Scheme 101



Scheme 102

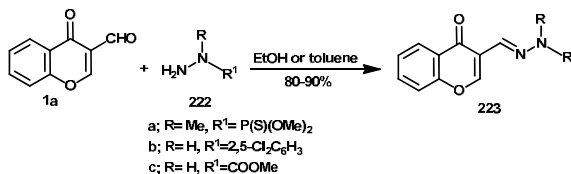
### 2.2.4. Reactions with hydrazines

The reaction of 3-formylchromones **1a-d** with hydrazines gave initially hydrazones **220** which then reacted further at the C-2 position to give 4-(2-hydroxybenzoyl)pyrazoles **212** (Scheme 103) [149-151].



Scheme 103

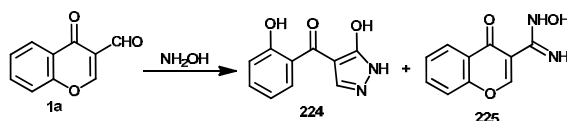
Treatment of 3-formylchromone **1a** with substituted hydrazines **222a-c** in absolute ethanol or toluene afforded the corresponding hydrazones **223a-c** (Scheme 104) [152,153].



Scheme 104

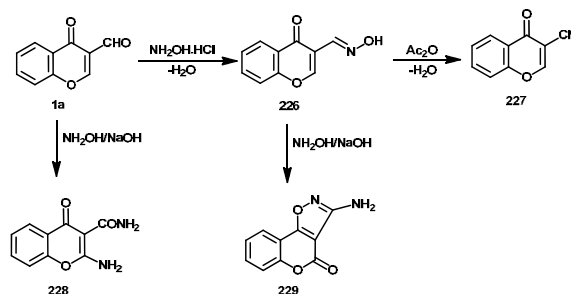
### 2.2.5. Reactions with hydroxylamine

The reaction of 3-formylchromone (**1a**) with hydroxylamine hydrochloride (1:2 molar ratio) afforded a mixture of pyrazole **224** and chromone **225** (Scheme 105) [154,155].



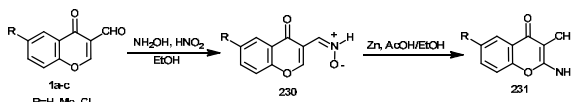
Scheme 105

Treatment of 3-formylchromones (**1a-d**) with hydroxylamine hydrochloride in ethanol gave the corresponding oximes **226** which on dehydration by acetic anhydride gave chromone-3-carbonitriles **227**. When the reaction of **1a-d** with hydroxylamine hydrochloride took place in the presence of sodium formate containing HCl, the carbonitriles **227** were obtained directly. On the other hand, Oximation of **1a** and its oxime **225** using hydroxylamine in sodium hydroxide led to 2-aminochromone-3-carboxamide (**228**) and 3-amino-4*H*-chromeno[3,4-*d*] isoxazol-4-one (**229**), respectively (Scheme 106) [156-158].



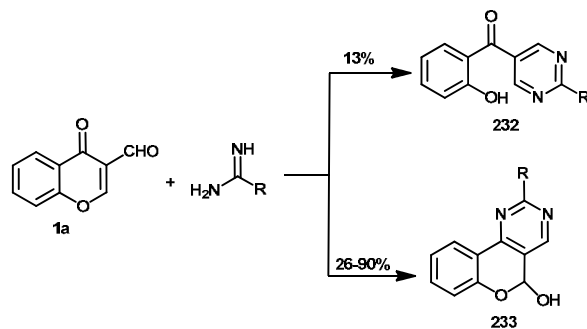
Scheme 106

Nitrones **230** were prepared from the reaction of aldehydes **1a-c** with hydroxylamine in ethanol. Nitrones **230** rearranged to 2-amino-3-formylchromones **231** in ethanol containing few drops of acetic acid in the presence of zinc (Scheme 107) [159,160].



Scheme 107

Hence, hydroxylamine on reactions with 3-formylchromones gave diverse types of products depending on the reaction conditions.



Scheme 108

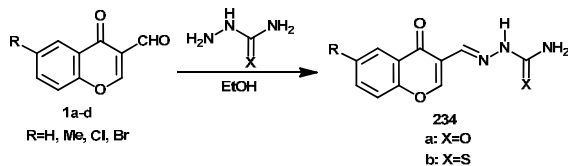
### 2.2.6. Reactions with amidines

Reaction of 3-formylchromone **1a** with formamidine gave a mixture of 5-(2-hydroxybenzoyl) pyrimidine **232** (R=H, 13%) and 5-hydroxy-5H-chromeno[4,3-d]pyrimidine **233** (R=H, 31%) as reported by Loewe [161]. While, reaction of **1a** with several C-substituted formamidines (R=alkyl, aryl, hetaryl, NH<sub>2</sub>, NHCN, SH, SMe, OH, OMe, 1-pyrrolidinyl, 1-piperidinyl, 4-morpholinyl) gave only chromeno-pyrimidines **233** in 26-90% yields (Scheme108) [161-166].

Thus, 3-formylchromones are a good precursors for the synthesis of pyrimidine derivatives via treatment with amidine derivatives as 1,3-bifunctional nucleophiles.

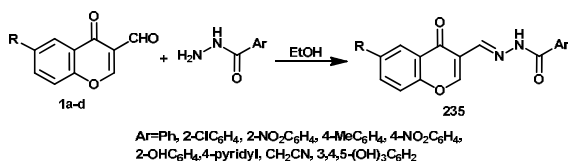
### 2.2.7. Reactions with hydrazides

3-Formylchromones **1a-d** reacted with semicarbazide and thiosemicarbazide in ethanol to give the corresponding semicarbazones **234a** (X=O) and thiosemicarbazone **234b** (X=S), respectively (Scheme109) [150,167,168].



Scheme 109

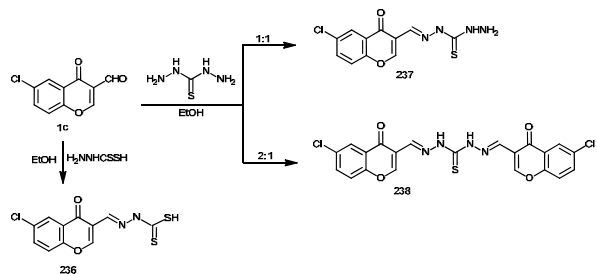
The reaction of 3-formylchromone **1a-d** with aroylhydrazines gave the corresponding aroylhydrazone **235** (Scheme110) [169-172].



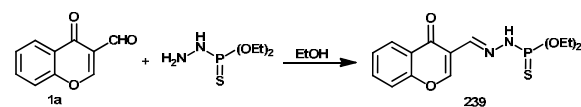
Scheme 110

3-Formylchromone **1c** condensed with equimolar amount of hydrazine carbodithioic acid and thiocarbohydrazide in ethanol to give the corresponding hydrazones **236** and **237**, respectively. Also, condensation of **1c** with two equivalents of thiocarbohydrazide gave bis thiocarbohydrazone derivative **238** (Scheme111) [173].

Phosphorohydrazone of chromone **239** was obtained from stirring 3-formylchromone **1a** with diethoxythiophosphorylhydrazide in methanol (Scheme112) [159].



Scheme 111



Scheme 112

## 3. Conclusion

In the present work, the chemical reactivity of 3-formylchromones was evaluated and summarized towards all types of carbon and nitrogen nucleophiles under different reaction conditions. 3-Formylchromones are very active toward the nucleophilic reagents, due to the availability of three electron deficient sites, the aldehydes carbon, C-2 carbon, and the C-4 carbon of the carbonyl group. A variety of fused heterocyclic systems were prepared directly from the reaction of 3-formylchromones with some bifunctional nucleophiles, these reactions mainly proceed via condensation with the aldehydic function followed by nucleophilic attack at C-2 position of the chromone moiety.

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