HETEROCYCLES, Vol. 106, No. 9, 2023, pp. 1443 - 1477.
Received, 22nd April, 2023, Accepted, 7th June, 2023,
DOI: 10.3987/REV-23-1009

RECENT TRENDS IN THE SYNTHESIS OF ENAMINONES

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Abstract – Enaminones are very useful intermediates for building different categories of heterocyclic compounds. Formation of enaminone derivatives was achieved using a variety of compounds such as alcohols, aldehydes, active methyl ketones, active methylene ketones, *β*-alkoxy ketones, *β*-diketones, acetoacetamides, Grignard reagents, diazocarbonyl compounds, acetylene derivatives, nitrile derivatives, cyclic compounds, acyl chloride and other enaminones. The present review covers the methods developed for the synthesis of enaminone derivatives.

CONTENTS

1. INTRODUCTION

The open-chain enaminones are active substrates containing functional group N–C=C–C=O as a part of a flexible chain. They are typical push-pull ethylenes in which the amine groups push electrons and the carbonyl groups pull electron densities. The presence of π-electron delocalization as well as *α*,*β*-unsaturated ketone are the two specific characteristic of enaminones which make them reactive.

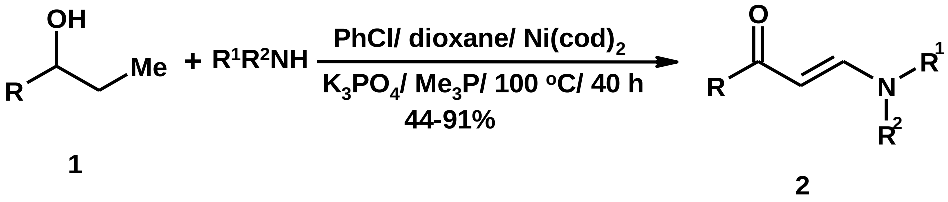
Enaminone compounds possess two electrophilic centers and three nucleophilic centers. So, enaminones are useful in variable scientific branches including synthetic intermediates in organic synthesis,[1,2] in pharmaceutical development[3] and in heterocyclic synthesis.[4,5] Also, they have been used as biological active agents such as antibacterial,[6] ant-inflammatory,[7] antioxidant, antimicrobial,[8] anticonvulsant,[9] antitumor [10] and anticancer agents.[11]

Numerous techniques have been developed for construction of enaminones using a diversity of substrates. The present review aims to collect the several methods used for the synthesis of enaminone derivatives starting from 2010 till now (April 2023).

2. SYNTHESIS OF ENAMINONES

2.1. From alcohols

Ueno *et al.*[12] reported that heating a mixture of α-substituted propanols **1** and amines, in 1,4-dioxane and chlorobenzene in the presence of potassium phosphate (4.0 equiv), Ni(cod)$_2$ (cod= cycloocta-1,4-diene) and trimethylphosphine, gave enaminones **2** (Scheme 1).

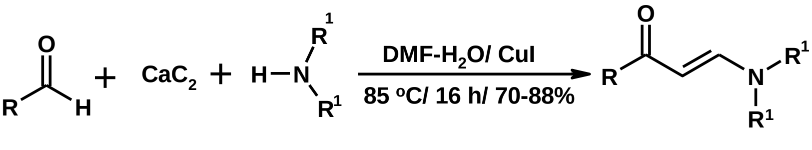


R= Et, *i*-Bu, *c*-hex, Ph, 4-ClPh, 4-F_3CPh, 4-MePh
R^1R^2NH= $HNBu_2$, H_2NBz, $HNBz_2$, piperidinyl, morpholinyl,
***N*-Boc-piperazinyl, 1,2,3,4-tetrahydroisoquinolinyl**

Scheme 1

2.2. From aldehydes

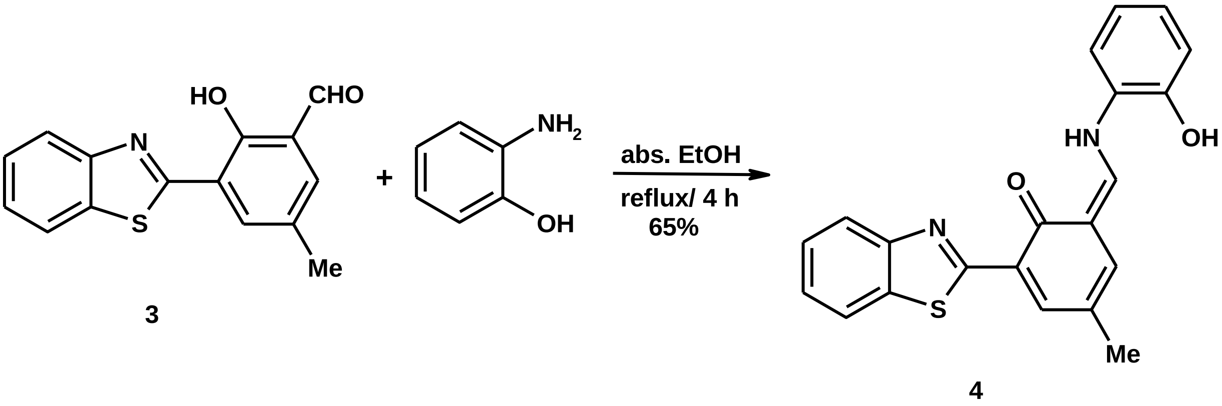
Various enaminones **2** were synthesized through three-component coupling reaction of aryl aldehyde, amines and calcium carbide, in aqueous dimethylformamide (DMF) containing copper(I) iodide as a catalyst (Scheme 2).[13]



R= Ph, 4-ClPh, 4-BrPh, 4-MePh, 4-EtPh,
4-MeOPh, 4-NO_2Ph, 3,5-$(MeO)_2$Ph,
4-CF_3Ph, 4-CNPh, 4-PhPh
R^1= *i*-Pr, *c*-hex

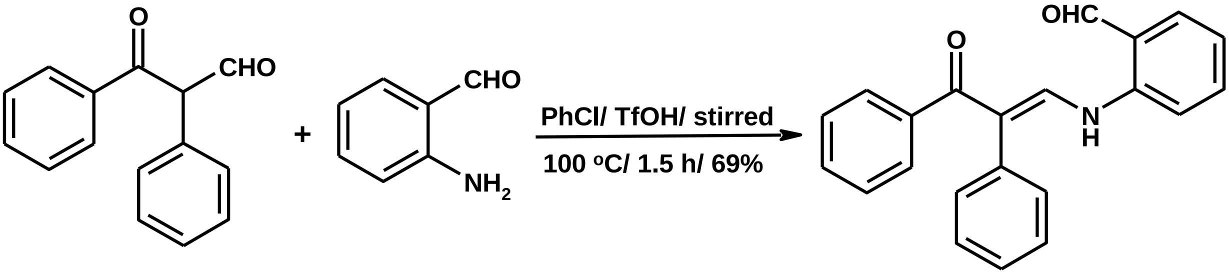
Scheme 2

2-(1,3-Benzothiazol-2-yl)-6-{[(2-hydroxyphenyl)amino]methylidene}-4-methylcyclohexa-2,4-dien-1-one (**4**) was prepared from nucleophilic addition of 2-aminophenol to 3-(benzo[*d*]thiazol-2-yl)-2-hydroxy-5-methylbenzaldehyde (**3**), in boiling ethanol (Scheme 3).[14]



Scheme 3

Luo *et al.*[15] demonstrated that 2-((*E*)-3-oxo-2,3-diphenylprop-1-enylamino)benzaldehyde (**6**) was formed, in good yield, from condensation reaction of 3-oxo-2,3-diphenylpropionaldehyde (**5**) with 2-aminobenzaldehyde, in chlorobenzene in the presence of triflic acid (TfOH) (Scheme 4).



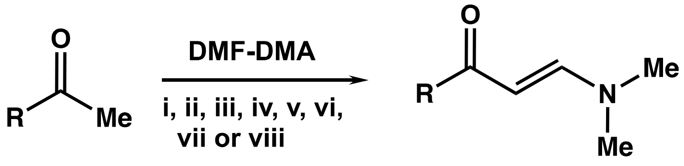
5

6

Scheme 4

2.3. From active methyl ketones

Acetyl derivatives represent one of the most common sources for formation of enaminones. Thus, treatment of acetyl derivatives **7** with dimethylformamide dimethyl acetal (DMF-DMA), under different reaction conditions, afforded (3-(dimethylamino)-1-substituted)prop-2-en-1-one **8** as shown in Scheme 5. Table 1 refers to the types of substituents (R) and their references.



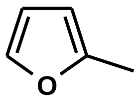
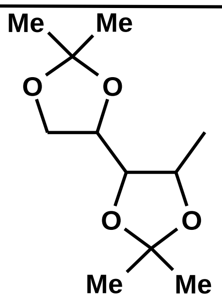
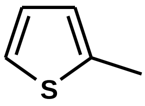
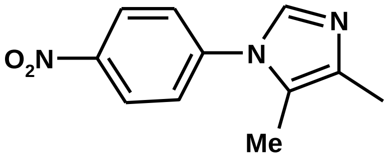
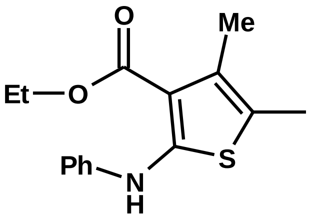
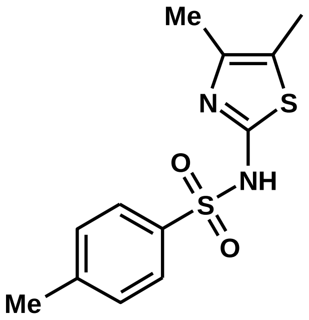
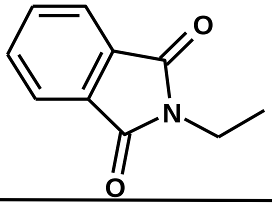
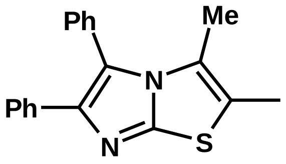
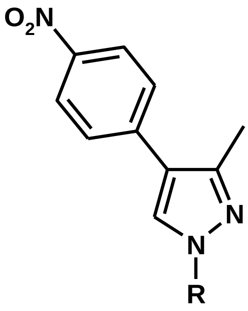
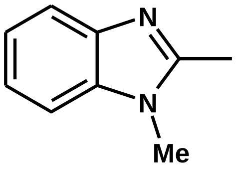
7

8

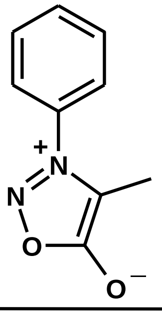
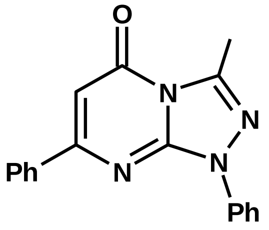
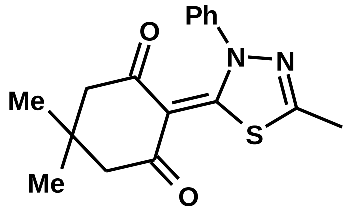
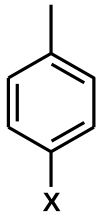
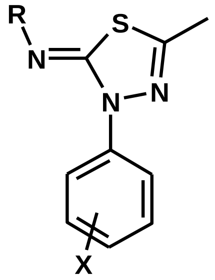
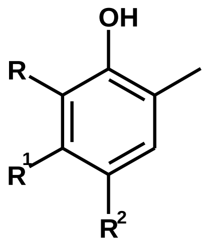
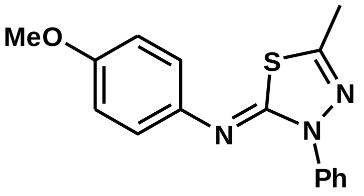
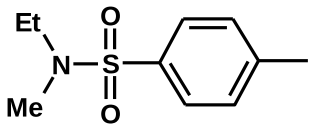
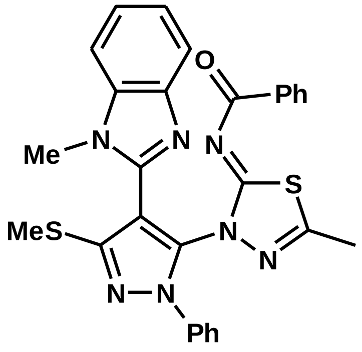
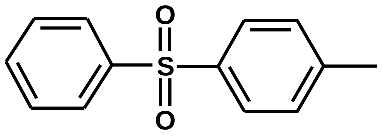
i) EtOH/ TEA/ reflux/ 6 h/ 58%[34]
ii) DMF/ reflux/ 0.5-3 h/ 82-97%[35]
iii) benzene/ reflux/ 5 h/ 77%[27]
iv) toluene/ reflux/ 5-24 h/ 46-90%[26,29,31,33,38]
v) fusion/ 85-120 °C/ 0.5-20 h/ 75-86%[17,19,21,24,40]
vi) dioxane/ reflux/ 5-6 h/ 90-93%[25,30,32,36]
vii) xylene/ reflux/ 2-7 h/ 58-93%[20,22,23,28,37,39]
viii) MW/ 120-180 °C/ 5-45 min/ 77-95%[1,2,16,18,33]

Scheme 5

Table 1:

R	Ref	R	Ref
	[16]		[20]
	[1]		[21]
	[17]		[22]
	[18]		[23]
 R= Ph, 4-MePh	[19]		[24]

Continued Table 1

	[21]		[29]
	[25]	 X= Cl, NO_2, MeO, CO_2Me	[1,16,21,30]
 X= H, 2-Cl, 4-Cl, 4-Br, 4-NO_2 2-Me, 4-Me, 4-MeO R= 4-MeOPh, PhOC	[26,27]	 R= H, I, Br R^1= H, Me R^2= H, I, Br, Cl, Me	[31]
	[27]		[32]
	[28]		[33]

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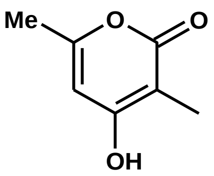
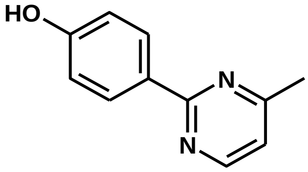
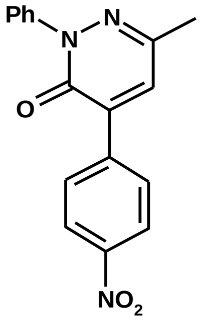
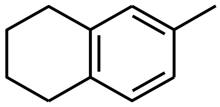
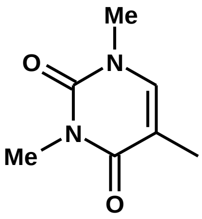
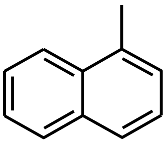
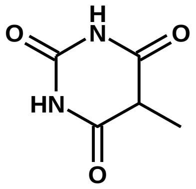
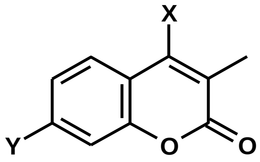
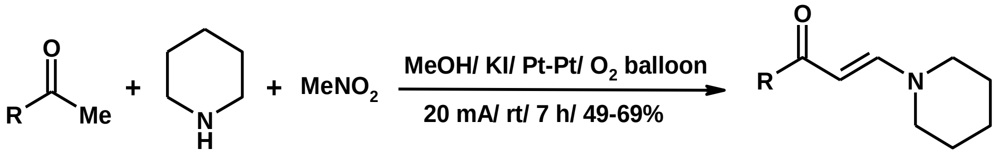
	[34,35]		[37]
	[36]		[38]
	[35]		[2]
	[35]	 X= H, OH Y= H, OH	[35,39,40]

Xu *et al.*[41] showed that enaminones **9** were obtained from electro-synthesis of acetyl derivatives **7**, piperidine and nitromethane. This method proceeded at room temperature to give enaminones **9** in one step, utilizing nitromethane as the carbon source (Scheme 6).



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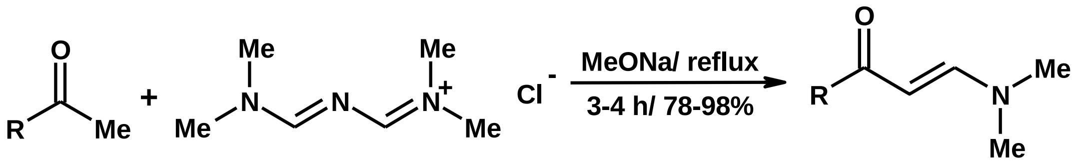
9

R= Ph, 2-FPh, 4-FPh, 4-ClPh,
4-BrPh, 4-IPh, 4-F_3CPh, 2-MePh,
3-MePh, 4-MePh, 3-MeOPh, 4-MeOPh

Scheme 6

Enaminones **8** have been prepared by Gupton method through reaction of acetyl derivatives **7** with Gold's reagent [3-(dimethylamino)-2-azaprop-2-en-1-ylidene]dimethylammonium chloride (**10**), in a good yields as shown in Scheme 7.[42,43]



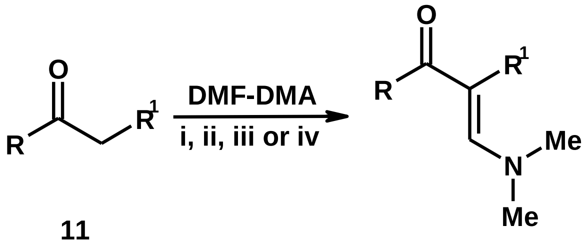
7 **10** **8**

R= 2-pyrrolyl, Ph, 4-BrPh, 4-ClPh,
4-NO_2Ph, 4-MeOPh, 2-naph,
2-(3-methylthiazolo[3,2-*a*]benzimidazolyl

Scheme 7

2.4. From active methylene ketones

Reaction of active methylene ketones **11** with DMF-DMA under various conditions [boiling in DMF or xylene, fusion or under microwave irradiations] donated enaminones **12**, in good to excellent yields (Scheme 8). Table 2 indicates to types of substituent R, R^1 and their references.



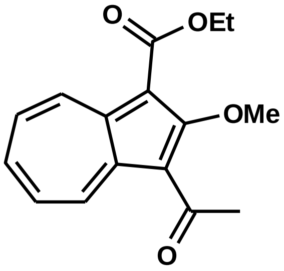
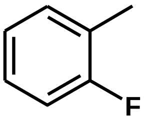
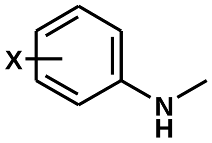
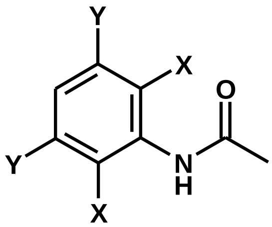
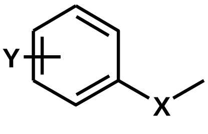
11

12

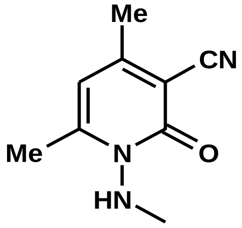
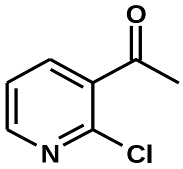
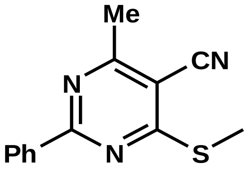
i) DMF/ 80-100 ^{o}C/ 0.5-3 h/ 82-97%[45,46,51]
ii) xylene/ reflux/ 4-5 h/ 66-87%[48,50,52]
iii) fusion/ 90-120 ^{o}C/ 0.5-24 h/ 55-98%[47,49]
iv) MW/ 5 min/ 90%[44]

Scheme 8

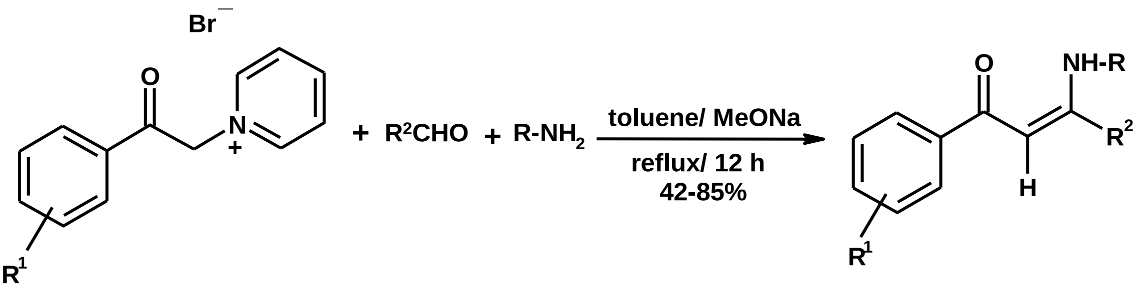
Table 2:

R	R^1	Ref
Me	CO_2Et	[44]
	CN	[45]
	CO_2Et	[46]
 X= Ph, 4-ClPh, 4-MePh, 2-MeOPh, 2,6-$(MeO)_2$Ph, 1-naph, 2-naph	Me, Ph, 4-FPh	[47]
 X=Y= H, Me	CN, COPh	[48]
 X = O, S Y= H, 4-Cl, 3-MeO, 2,5-$(MeO)_2$, 3,4-$(MeO)_2$	Me, 2-BrPh, 3-MePh, 4-MePh, 3-MeOPh, 4-MeOPh, 3-Br-4-MeOPh	[49]

Continued Table 2

	CN	[50]
	CO_2Et	[51]
	Ph	[52]

Enaminones **14** were prepared *via* multicomponent reaction of phenacylpyridinium bromides **13**, primary amines, and phenylglyoxals or ethyl glyoxalate, in moderate to good yields (Scheme 9).[53]



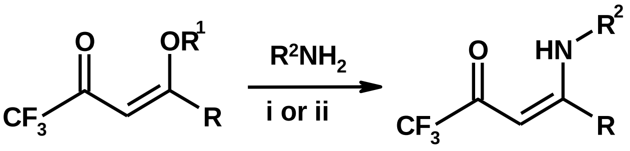
13 **14**

R= 4-ClPh, 3-MePh, 4-MePh, 3,4-$(Me)_2$Ph
R^1= H, 4-Me, 4-Br, 4-Cl
R^2= CO_2Et, PhCO, 4-ClPhCO, 4-MePhCO

Scheme 9

2.5. From *β*-alkoxy ketones

Trifluoromethylated *β*-aminovinyl ketones **16** were prepared, in good to excellent yields, by reaction of (*E*)-4-alkoxy-1,1,1-trifluorobut-3-en-2-one **15** with various amines (slight excess), in anhydrous acetonitrile (Scheme 10). Table 3 illustrates the types of substituent R, R^1, R^2 and their references.



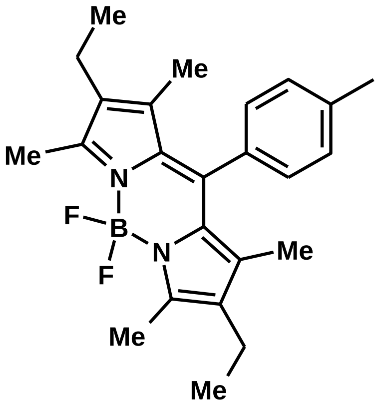
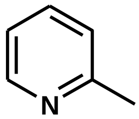
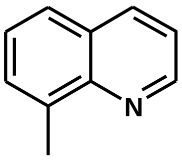
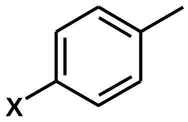
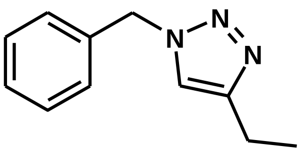
15 **16**

i) MeCN/ stirred/ rt/ 5 h/ 72-94%[54,55,57]

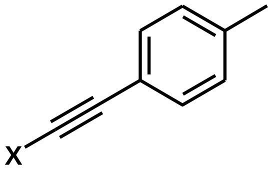
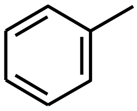
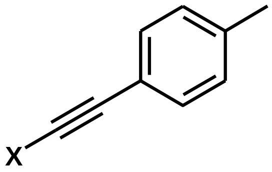
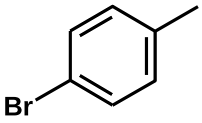
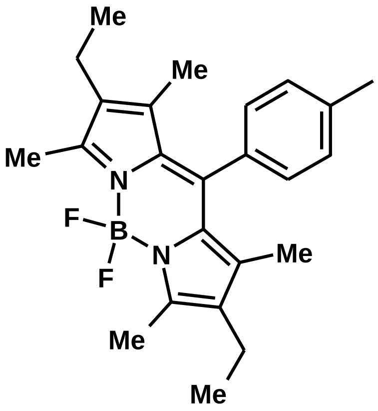
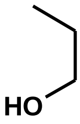
ii) MeCN/ TEA/ stirred/ rt/ 24 h/ 44-80%[56]

Scheme 10

Table 3:

R	R^1	R^2	Ref
H	Et		[54]
H	Et		[55]
H	Et		[55]
 X= H, Br, NO_2	Me		[56]

Continued Table 3

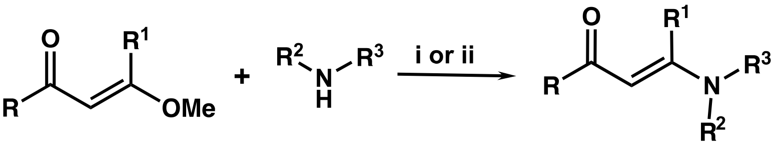
 X= TMS, Ph	Me		[57]
 X= TMS, Ph	Me		[57]
	Me		[54]

Reaction of *β*-methoxyvinyl ketones **17** with secondary amines, under different conditions [thermal or ultrasound irradiation (US), afforded enaminones **18**, in 70-93% yields as illustrated in Scheme 11.[58,59]

2.6. From *β*-diketones

Reacting acetylacetone (**19**) with ammonium salts in the presence of catalytic amount of 1-butyl-3-methylimidazolium hydrogen sulphate [bmim]HSO_4 under solvent free conditions led to 4-aminopent-3-en-2-one (**20**) (Scheme 12).[60]

Enaminones **21** were prepared, in quantitative yields, by the reaction of primary amines with *β*-diketones **19** under different conditions (Scheme 13). The substituents and their references are depicted in Table 4.



17

18

i) MeCN/ US/ 70-74 °C/ 20 min/ 70-93%[58]

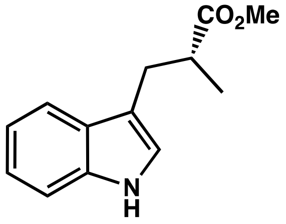
ii) AcOH/ MeCN/ 0-75 °C/ 15 h/ 91%[59]

R= CF_3, CCl_3, CO_2Et, CH_2CO_2Me

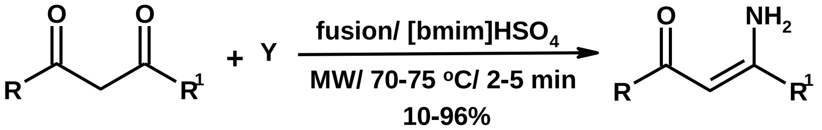
R^2= Pr, Bz

R^1= H, Me, Et, Pr, Bu, *i*-Pr, $CH_2CH_2CO_2Me$

R^3= HC≡CH-CH_2,



Scheme 11



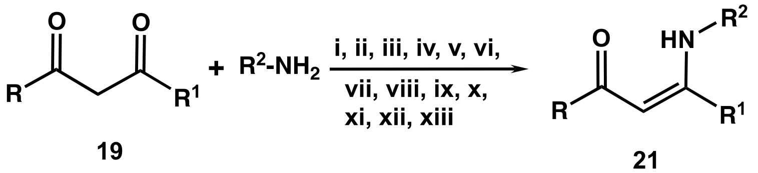
19

20

R= R^1= Me

Y= $(NH_4)_2CO_3$, $(CO_2NH_4)_2$, NH_4OAc, NH_4Cl

Scheme 12



19

21

i) toluene/ *p*-TsOH/ reflux/ 10-48 h/ 40-52%[69]

ii) $CHCl_3$/ AcOH/ 80 °C/ 1 h/ 40-58%[70]

iii) THF/ CuCl/ rt/ 12-48 h/ 75-98%[61]

iv) MeCN/ 40% phosphotungstic acid/ TiO_2/ rt/ 1-30 min/ 65-98%[65]

v) EtOH/ stirred/ rt/ 1 h/ 74-89%[63]

vi) MeOH/ HCO_2H/ 85 °C/ 4 h/ 53-98%[67]

vii) EtOH/ AcOH/ reflux/ 3 h/ 45%[63]

viii) fusion/ $ZrOCl_2{\cdot}8H_2O$/ stirred/ rt/ 10-300 min/ 81-95%[64]

ix) fusion/ $[VO(acac)_2]$/ stirred/ rt/ 15-60 min/ 86-91%[68]

x) fusion/ $Fe(HSO_4)_3{\cdot}SiO_2$/ rt/ 7-25 min/ 81-95%[66]

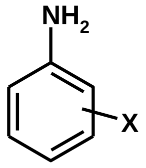
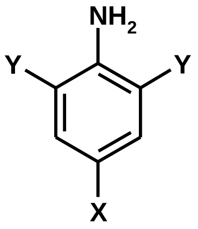
xi) fusion/ 40% phosphotungstic acid/ TiO_2/ rt/ 1-13 min/ 90-98%[65]

xii) fusion/ $[bmim]H_2SO_4$/ MW/ 70-75 °C/ 2-3 min/ 70-98%[60]

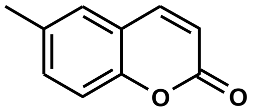
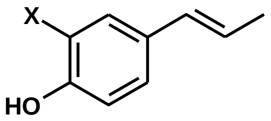
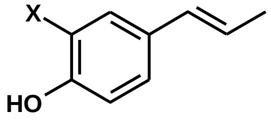
xiii) fusion/ $Zn(X)_2$/ ultraviolet/ 1h/ 65-99% X= Gly, Pro[62]

Scheme 13

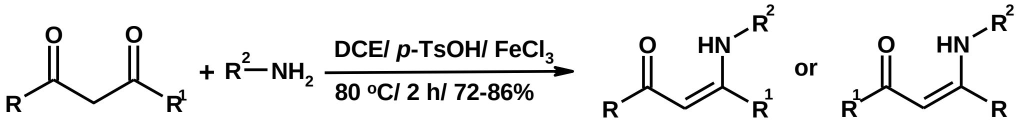
Table 4:

R	R^1	R^2	Ref
Me	Me	Bu, Ph, 2-ClPh, 3-ClPh, 4-ClPh, 4-BrPh, 3HOPh, 2-MePh, 4-MePh, 2-MeOPh, 4-MeOPh, Bz, 1-naph	[61]
Me	Me	Me, *i*-Pr, Pe, hex, 4-ClPh 4-MePh, Bz, 1-naph	[60]
Me	Me	*c*-hex, Ph, Bz, 4-MePh 4-MeOPh	[62]
Me	Me	 X= 2-CO_2H, 4-CO_2H	[63]
Me, Ph	Me	Bn, Ph, 2-MePh , 4-ClPh, 4-MePh, 4-MeOPh, 4-EtOPh	[64]
Me, Ph	Me	$(CH_2)_2NH_2$, *n*-Bu, Ph, 4-ClPh 4-NO_2Ph, 4-MePh, 4-MeOPh, Bz, PhCO	[65]
Me, Ph	Me	Ph, 4-ClPh, 4-NO_2Ph, 4-MePh, 4-MeOPh,	[66]
Me, Ph	Me	 X= H, Me, OMe, Cl, NO_2, CN Y= H, Me, *i*-Pr	[67]
Ph	Me	2-MeOPh, 2,6-$(Me)_2$Ph, Bz	[68]

Continued Table 4

Me, Et, Ph	**Me, Et**		**[69]**
 **X= H, OMe**	 **X= H, OMe**	***c*-hex, *n*-Pr, *n*-Bu, *i*-Bu, *sec*-Bu, allyl**	**[70]**

On the other hand, treatment of 1,3-dicarbonyl compounds **19** with some primary amines using $FeCl_3$/*p*-TsOH as a catalyst gave *β*-*N*-substituted enaminones **22** or **23** (Scheme 14).[71]

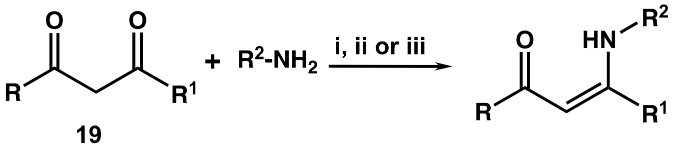


19 **22** **23**

R= Me
R^1= CF_3, Ph
R^2= hex, 2-py, 3-MePh, 4-MePh, 4-MeOPh, Bz, 1-naph

Scheme 14

Also, acyclic enaminones **24** were prepared, in good to excellent yields, from the condensation reaction of various diketones **19** with different amines, using copper, silver or copper oxide nanoparticles (NPs) as catalyst (Scheme 15).[72-74]



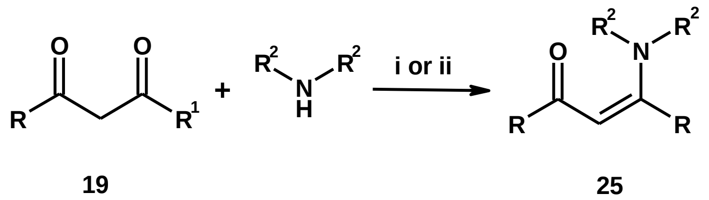
19 **24**

i) MeOH/ NPs Ag/ 60 °C/ 1-8 h/ 70-93%[72]
II) MeOH/ NPs Cu/ 45-50 °C/ 1.15-3.20 h/ 85-94%[73]
iii) fusion/ CuO NPs/ 60 °C/ 9 h/ 70-98%[74]

R= Me, Ph
R^1= Me
R^2= Me, Et, C_2H_2, *i*-Pr, Bu, *c*-hex,
2-pyrrolyl, Ph, 4-BrPh, 4-NO_2Ph,
4-MePh, 4-MeOPh, 2,4-F_2Ph, Bz

Scheme 15

Similarly, 4-*N*,*N*-dialkylaminopent-3-en-2-ones **25** were obtained from reaction of acetylacetone (**19**) with secondary amines under different reaction conditions, in good yields (Scheme 16).[75]

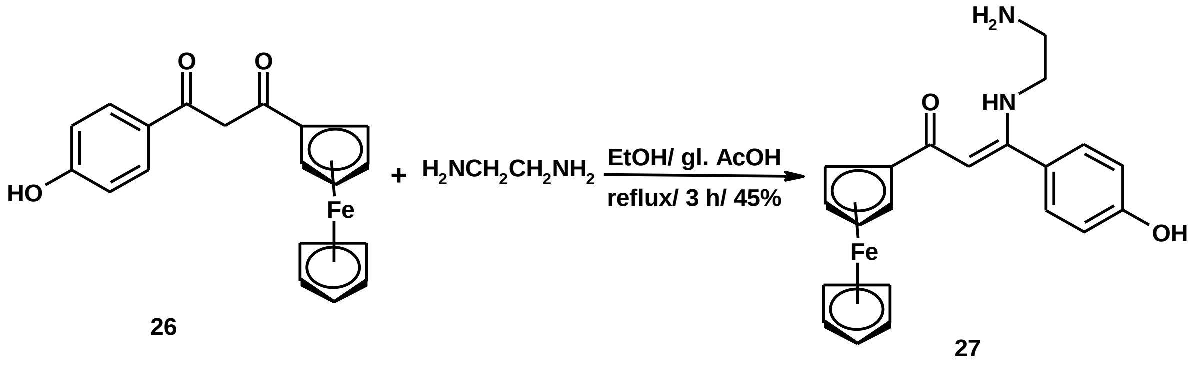


i= ice bath/ 15 min/ 66-68% R^2= Et $R=R^1$= Me

ii= benzene/ *p*-TsOH/ 85 °C/ 5 h/ 64-67% R^2= Pr, *c*-hex

Scheme 16

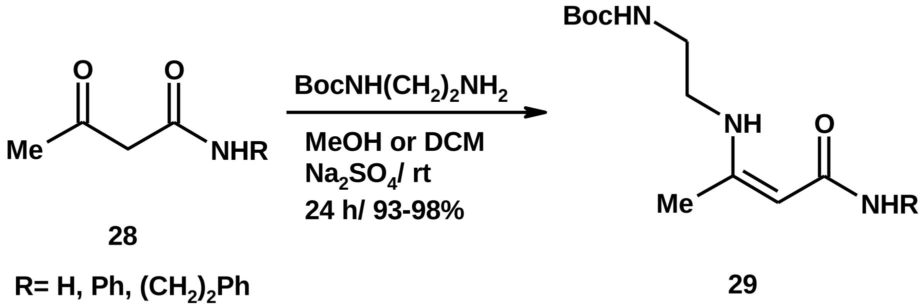
Celedon *et al.*[76] reported that formation of *O*,*N*,*N*-tridentate metalloligand enaminone **27**, in 45% yield, was achieved from organometallic *β*-diketone **26** with ethylenediamine, in ethanol containing drops of glacial acetic acid (Scheme 17).



Scheme 17

2.7. From acetoacetamides

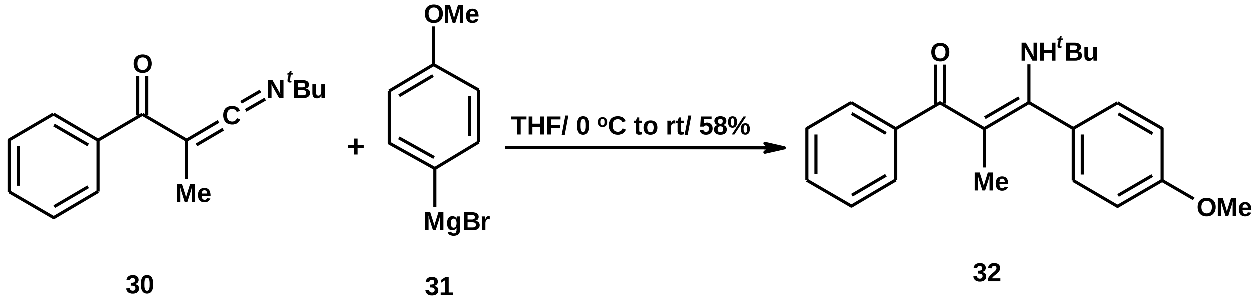
Boc-monoprotected ethylenediamines (Boc = *tert*-butyloxycarbonyl) was treated with acetoacetamides **28** to provide *β*-enaminoamides **29**, in quantitative yields (Scheme 18).[77]



Scheme 18

2.8. From Grignard reagents

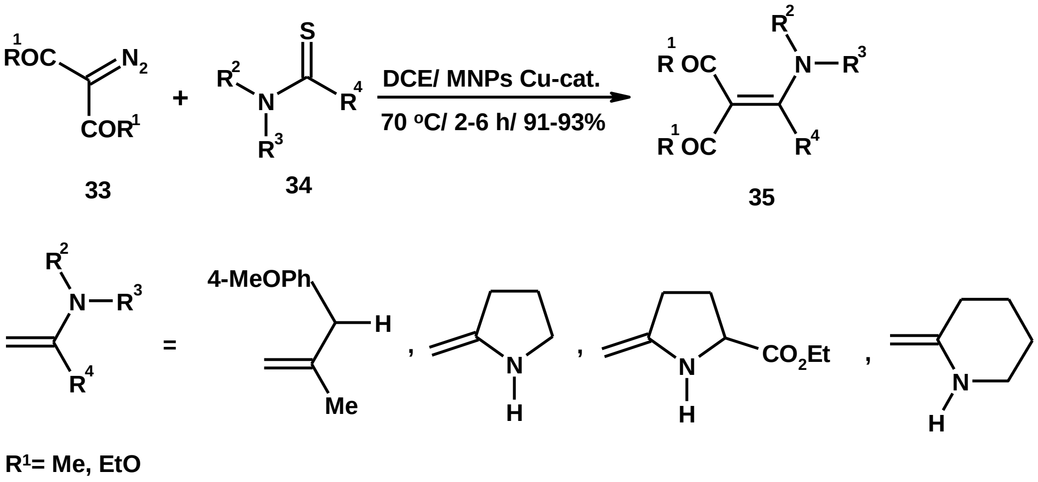
Mamboury *et al.*[78] presented that treatment of α-oxo-ketenimine **30** with 4-methoxyphenylmagnesium bromide (Grignard reagent) (**31**) provided 3-(*t*-butyl-amino)-3-(4-methoxyphenyl)-2-methyl-1-phenylprop-2-en-1-one (**32**), in 58% yield, *via* chemoselective nucleophilic addition to the ketenimine function (Scheme 19).



Scheme 19

2.9. From diazocarbonyl compounds

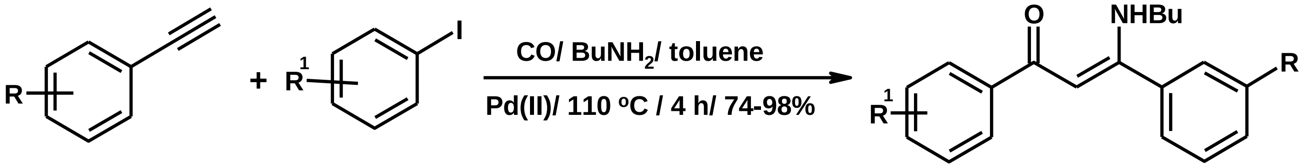
Mohammadi *et al.*[79] presented that coupling of α-diazocarbonyl compounds **33** and thioamides **34**, using a magnetic nanoparticle (MNPs)-Cu as a catalyst, in dry dichloroethane at 70 °C, gave enaminones **35** (Scheme 20).



Scheme 20

2.10. From acetylene derivatives

On the other hand, alkynes represent another way to synthesize enaminones. Carbonylative Sonogashira coupling of terminal alkynes **36** with iodobenzenes **37** is accompanied by Michael addition of butylamine in a one-pot method, to generate enaminones **38**. This reaction occurred in the presence of *N*,*N* bidentate palladium(II) complexes as catalyst (Scheme 21).[80]

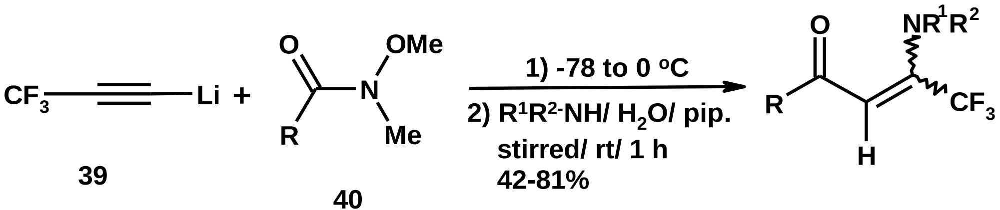


36 **37** **38**

R= H, 4-F, 4-Me, 4-Et, 4-^{t}Bu,
R^1= H, 4-Me

Scheme 21

Trifluoromethyl enaminones **41** were prepared, in good yields, from the reaction of trifluoropropynyllithium (**39**) with *N*-methoxy-*N*-methylamide **40** at -78-0 °C, followed by quenching with a variety of amines, in the presence of H_2O (Scheme 22).[81]

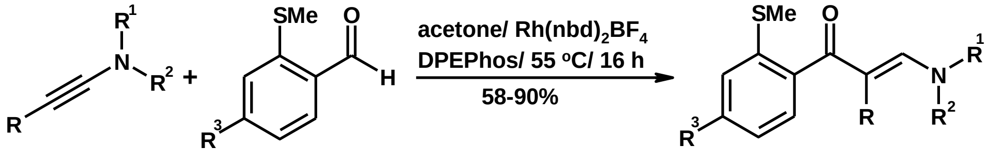


39 **40** **41**

R= Ph, 4-ClPh, 4-MePh
R^1, R^2= *c*-hex, $(Me)_2$, (Bn, Me), (H, Bn),
$((Me)_2CH)_2$, $((Me)_3Si)_2$, (H, Ph)

Scheme 22

Enaminones **44** were generated, in high yields, from the reaction of ynamide derivatives **42** and aldehydes **43**, in the presence of rhodium *bis*[(2-diphenylphosphino)phenyl] ether [DPEPhos] and [*bis*(norbornadiene)rhodium(I) tetrafluoroborate] [$Rh(nbd)_2BF_4$] as depicted in Scheme 23.[82]

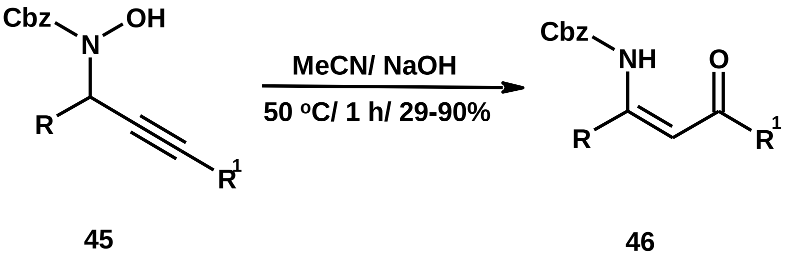


42 **43** **44**

R= 3-thienyl, *n*-hex, Ph, 4-CF_3Ph, 4-MeOPh
R^1= Ts, Cbz, Boc, Ms
R^2= Me, Ph
R^3= H, Br, F_3C, MeS, MeO

Scheme 23

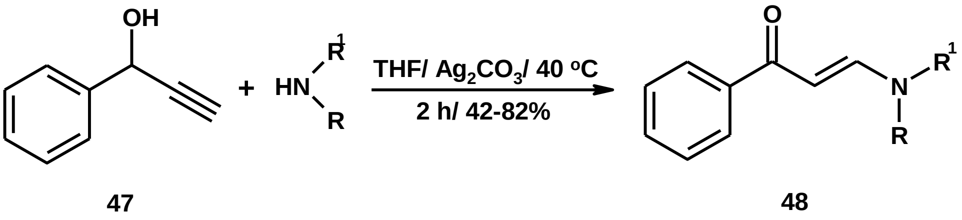
Gayon *et al.*[83] demonstrated that (*N*-carboxybenzyl) Cbz-protected enaminones **46** were obtained from catalytic isomerization of readily available propargylic hydroxylamines **45**, in acetonitrile containing NaOH for 1 h, in 29-90% yields (Scheme 24).



R= thienyl, Ph, 2-FPh, 3-BrPh, 4-FPh, 4-ClPh,
4-BrPh, 4-MePh, 3-MePh, 3-MeOPh, 4-PhPh,
1-naph, 2-naph
R^1= Me, *i*-Pr, *n*-Bu, *t*-Bu

Scheme 24

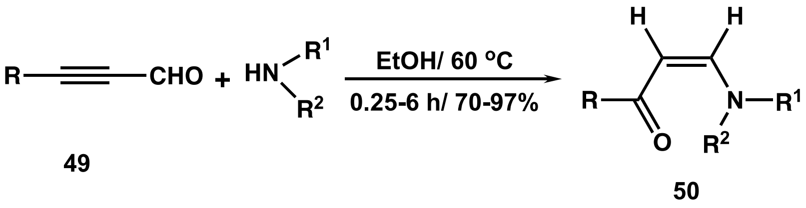
Enaminones **48** were prepared, in good yields, from treatment of propargyl alcohols **47** with various acyclic amines, under mild reaction conditions in the presence of Ag_2CO_3 (Scheme 25).[84]



R, R^1= H, Me, Et, Ph, Bz

Scheme 25

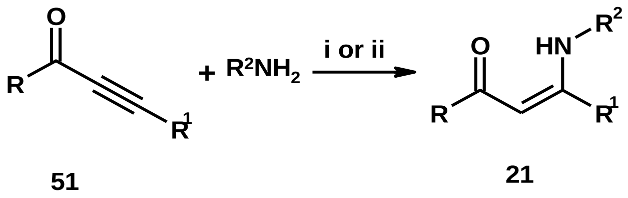
A convenient one-pot strategy for the regioselective synthesis of enaminones **50** have been developed *via* the condensation reaction of propiolaldehydes **49** with some acyclic and cyclic amines, in boiling ethanol (Scheme 26).[85]



R= 2-thienyl, Ph, 4-FPh, 4-ClPh, 4-BrPh,
2-ClPh, 4-MePh, 4-EtPh, 4-MeOPh,
R^1= R^2 = Et, Pr, *i*-Pr, *n*-Bu, *n*-Pr,
n-hex, *c*-hex, *n*-octanyl

Scheme 26

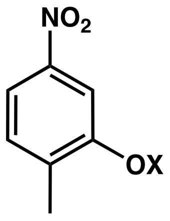
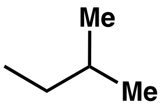
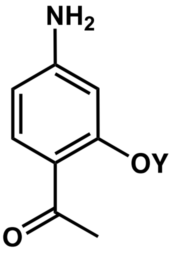
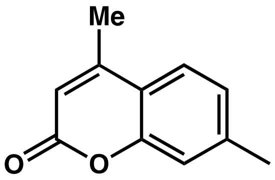
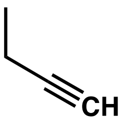
Enaminone derivatives **21** were obtained through addition reactions of primary amines with several α-ketoalkynes **51**, under various reaction conditions (a nickel homogeneous aqueous catalytic system formed from Ni(CN)$_2$/CO/NaOH/KCN or boiling methanol) (Scheme 27). Table 5 refers to substituents R, R^1, R^2 with their reference.



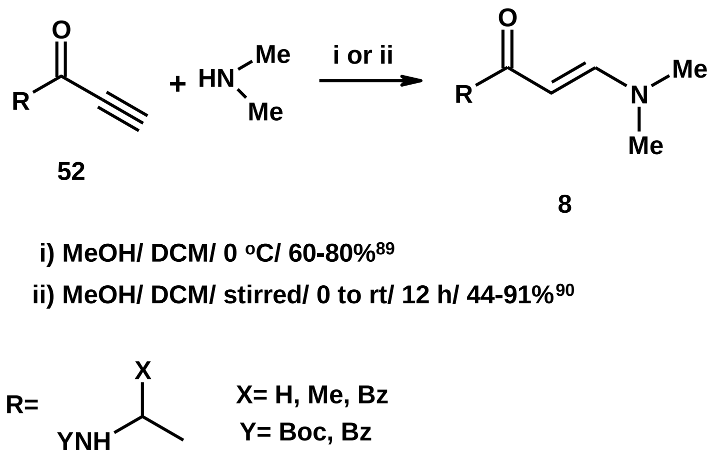
i) EtOH/ Ni(CN)$_2$/KCN/ NaOH/ CO/ H$_2$O/ rt/ 24 h/ 40-60%[87]
ii) MeOH/ 65-120 °C/ 3-6 h/ 13-98%[86,88]

Scheme 27

Table 5:

R	R^1	R^2	Ref
 X= Me, *i*-Pr, Bn		 Y= Me, *i*-Pr, Bn	[86]
3-MePh, 4-MePh 4-EtPh, 4-BrPh, 3,5-(MeO)$_2$Ph	Fc [Ferrocenyl]		[87]
Me, Ph, 4-ClPh, 4-MePh, 4-MeOPh	Bu, 3-thienyl, Ph 4-MePh, 4-MeOPh		[88]

Enaminones **8** were prepared, in a good yields, from addition of dimethylamine to ynone derivatives **52**, under different reaction conditions (Scheme 28).[89,90]



52

8

i) MeOH/ DCM/ 0 °C/ 60-80%[89]

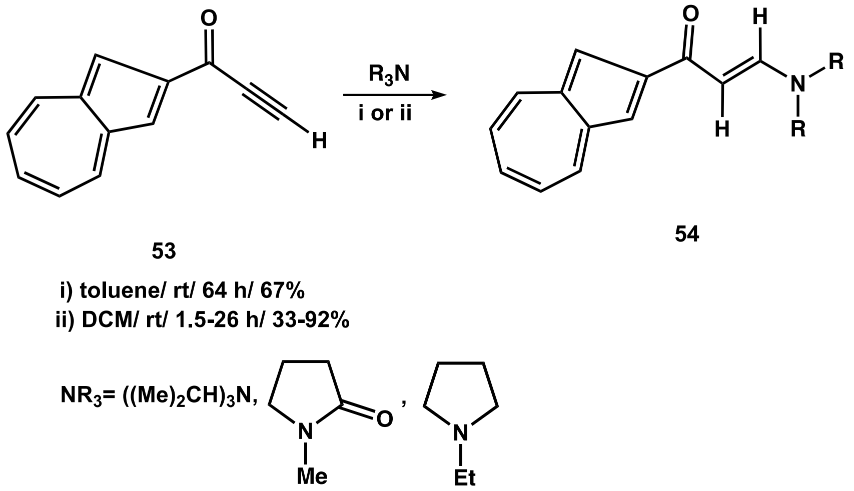
ii) MeOH/ DCM/ stirred/ 0 to rt/ 12 h/ 44-91%[90]

X= H, Me, Bz

Y= Boc, Bz

Scheme 28

Treatment of 1-(azulen-2-yl)prop-2-yn-1-one (**53**) with tertiary amines, under various reaction conditions, resulted in 1-((azulen-2-yl)prop-2-en-1-ones **54**, in high yield (Scheme 29).[91]



53

54

i) toluene/ rt/ 64 h/ 67%

ii) DCM/ rt/ 1.5-26 h/ 33-92%

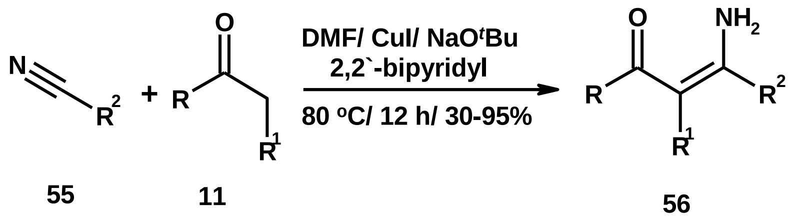
Scheme 29

2.11. From nitrile derivatives

Aldol-type addition of active methylene ketones **11** to aromatic nitriles **55** proceeded, in the presence of a simple copper catalyst system (CuI–2,2-bipyridyl–NaO^{t}Bu), produced enaminones **56**, in satisfactory to excellent yields (Scheme 30).[92]

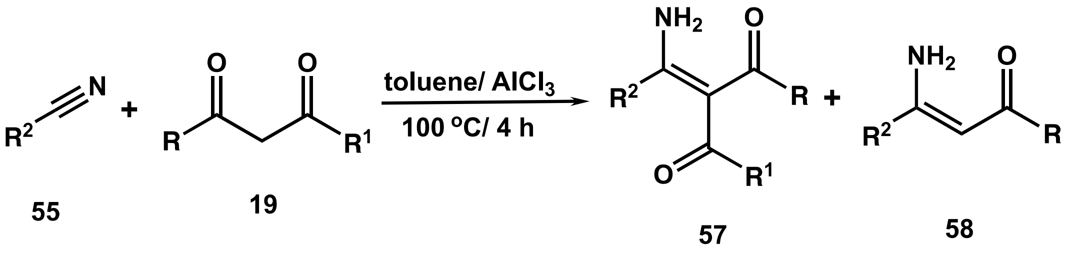
Various enaminone derivatives **57** and **58** were prepared from $AlCl_3$ catalyzed reaction of *β*-diketones **19** with nitriles **55**, in boiling toluene (Scheme 31).[93]

One-pot Ugi/nucleophilic addition reaction of isocyanides **59**, aromatic aldehydes, propiolic acid (**60**) and primary amines in the presence of secondary amines such as piperidine, morpholine and *N*-hydroxyethylpiperazine were carried out at room temperature to produce polyfunctionalized enaminones **61** as shown in Scheme 32.[94]



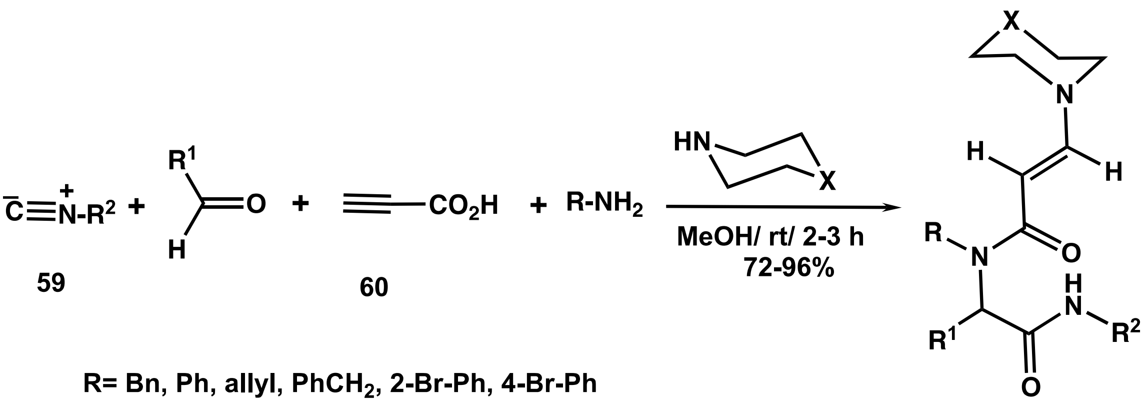
R= Pr, 2-thienyl, Ph, 2-MePh, 4-MePh,
4-MeOPh, 4-$(Me)_2$NPh, 4-BuNHPh,
4-FPh, 4-ClPh
R^1= H, Me
R^2= Ph, 4-ClPh, 4-CF_3Ph, 4-MePh,
4-MeOPh, 2-Py, 2-ClPy

Scheme 30



R= R^1= Me, *t*-Bu, Ph
R^2= Me, $Br(CH_2)_2$, 2-furyl, Ph, 2-ClPh,
2-MeOPh, 4-NO_2Ph, 2-NO_2Ph,
Bz, 4-HOBz, PhCH=CH

Scheme 31

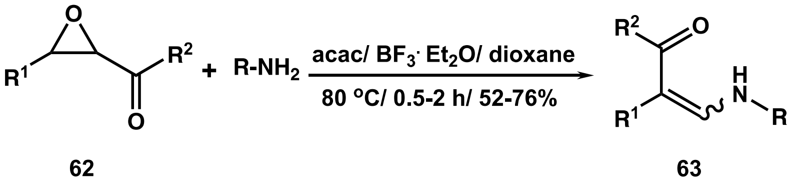


R= Bn, Ph, allyl, $PhCH_2$, 2-Br-Ph, 4-Br-Ph
R^1= 2-thienyl, Ph, 4-BrPh, 4-CNPh
R^2= *t*-Bu, *c*-hex
X= O, CH_2, NCH_2CH_2OH

Scheme 32

2.12. From cyclic compounds

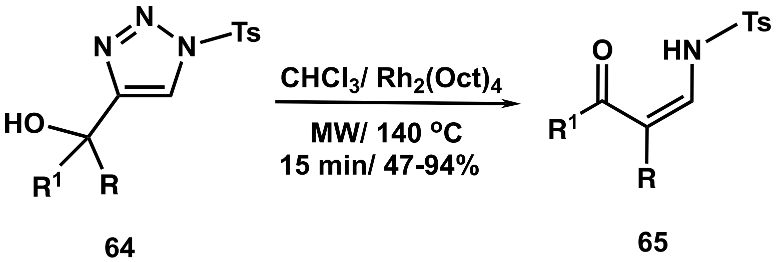
Domino reactions of different oxirane derivatives **62** with substituted arylamines, in the presence of acetylacetone (acac) and $BF_3·Et_2O$, in 1,4-dioxane, afforded enaminones **63**, in moderate yields (Scheme 33).[95]



**R= Ph, 2-IPh, 3-BrPh, 4-ClPh, 4-BrPh,
4-MeOPh, 3,4-Cl_2Ph, 1-naph**
R^1= Ph, 4-BrPh
R^2= Ph, 4-MeOPh

Scheme 33

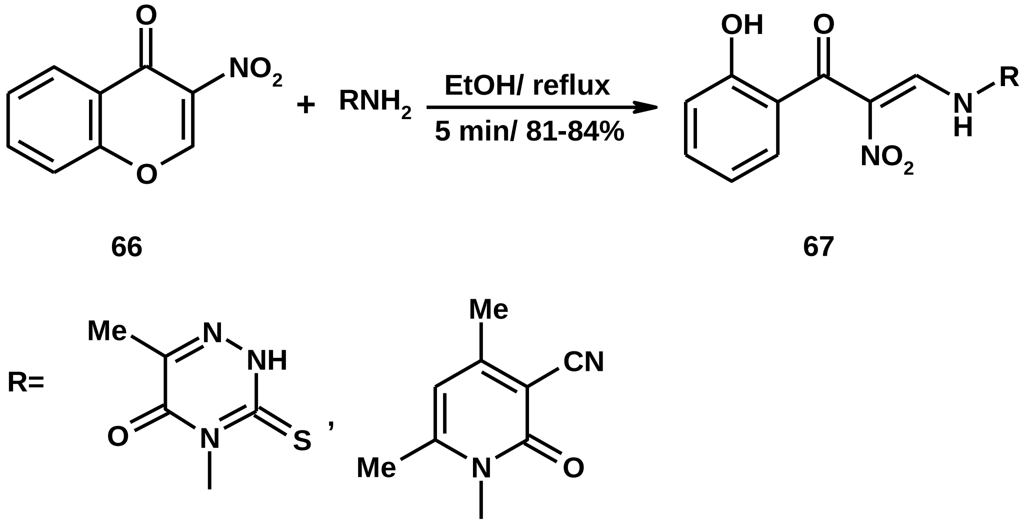
Enaminones **65** were synthesized by rhodium(II) octanoate, $Rh_2(Oct)_4$, catalyzed denitrogenative rearrangement reaction of 1-(*N*-sulfonyl-1,2,3-triazol-4-yl)alkanols **64**, in $CHCl_3$ at 140 °C under microwave irradiation for 15 min. (Scheme 34).[96]



R= H, Me, Ph
R^1= Me, *i*-Pr, *t*-Bu, Ph

Scheme 34

On the other hand, ring opening of 3-nitrochromone (**66**) with some heterocyclic amines, in boiling ethanol, furnished (2*Z*)-1-(2-hydroxyphenyl)-3-(substitutedamino)-2-nitroprop-2-en-1-ones **67** (Scheme 35).[97]

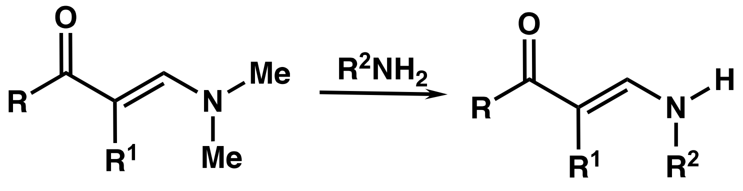


66 **67**

Scheme 35

2.13. From other enaminones

In addition to the above methods developed for the synthesise of enaminones, another method to synthesize enaminone derivatives are the reaction of enaminone itself with different primary and secondary amines. Thus, a variety of enaminones **68** were efficiently synthesized from reaction of enaminone **12** with a diversity of primary amines, under various reaction conditions (Scheme 36). Table 6 shows the types of substituent R, R^1, R^2 and their references.

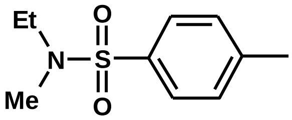
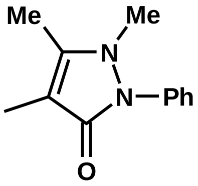
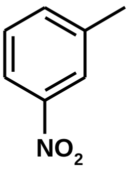
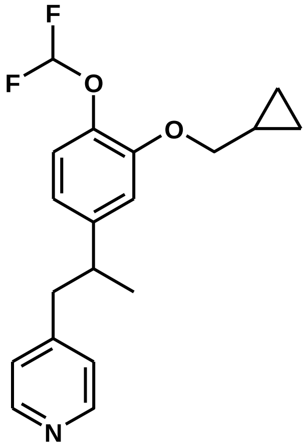
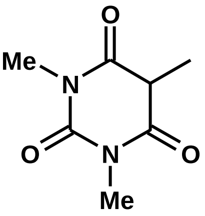
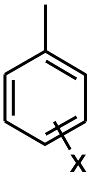
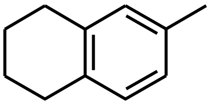
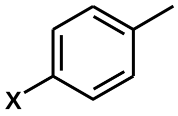
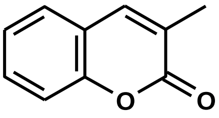
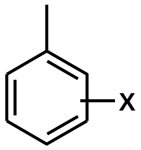


12 **68**

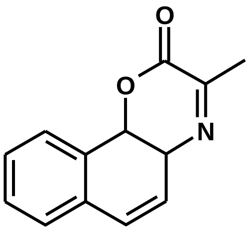
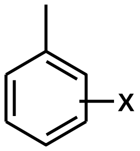
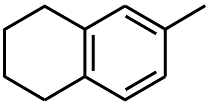
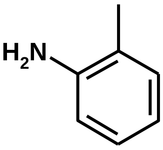
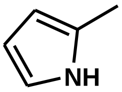
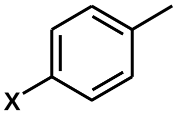
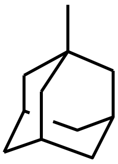
i) EtOH/ reflux/ 4-8 h/ 66-83%[38,103]
ii) EtOH/ AcOH/ reflux/ 3 h/ 66%[4]
iii) AcOH/ reflux/ 5 h/ 56-90%[39,102]
iv) AcOH/ stirred/ rt/ 3 h/ 68-78%[100,101]
v) [PyH]Cl/ EtOH/ 110 °C/ 20 min/ 74%[103]
vi) EtOH/ ultrasound/ 70 °C/ 2 h/ 83%[103]
vii) glycerol/ Lanthanum / chitosan/ 100 °C/ 2-5 h/ 62-72%[99]
viii) glycerol/ Lanthanum / chitosan/ MW/ 100 °C/ 5-10 min/ 90-96%[99]
ix) EtOH/ MW/ 15-25 min/ 71-92%[104]

Scheme 36

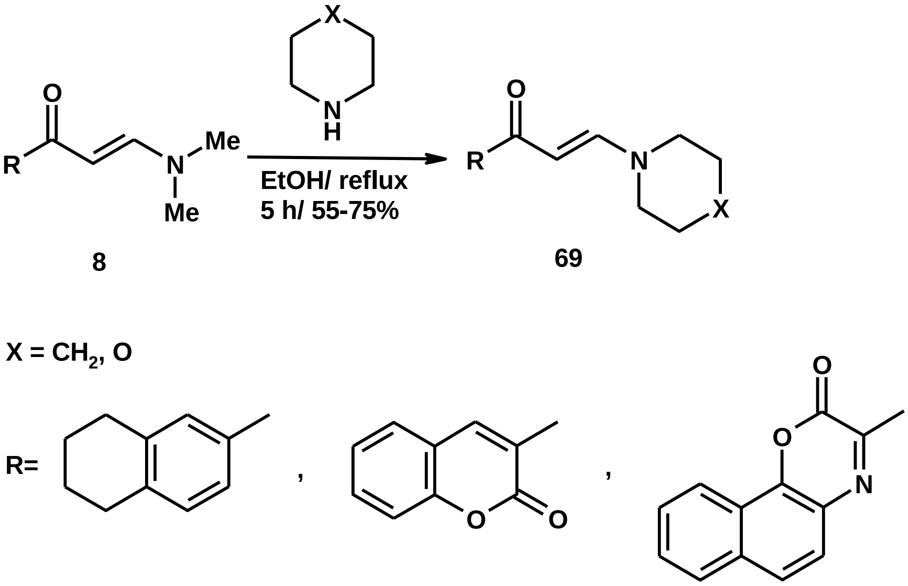
Table 6:

R	R^1	R^2	Ref
	H		[4]
	H		[98]
	H	 X= H, 4-Cl, 4-OH, 4-NO$_2$, 2-NO$_2$, 4-Me, 4-OMe	[99]
	H	 X= H, F, Cl, Br, Me, MeO, NO$_2$	[100,101]
	H	 X= H, 2-CO$_2$H	[39]

Continued Table 6

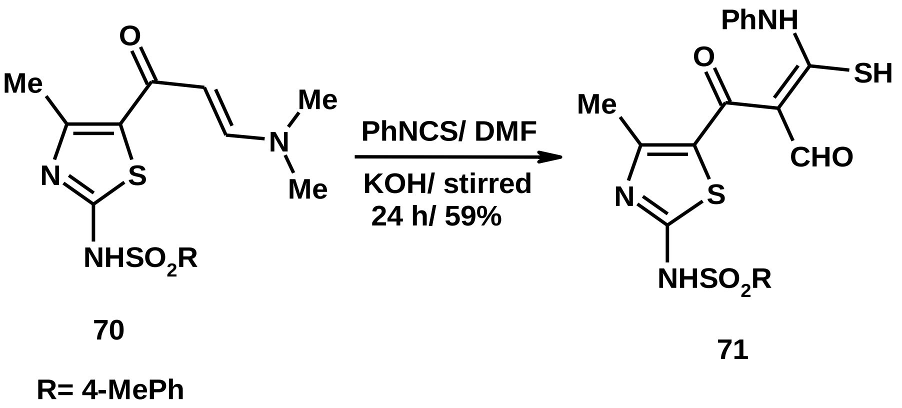
	H	 X= $2-NO_2$, $4-NO_2$	[102]
	H		[38]
	CN		[103]
 X= H, Cl, NO_2, Me, OMe	H		[104]

Also, enaminone **8** reacted with some secondary amines namely piperidine and morpholine, in refluxing ethanol, to yield 1-substituted-3-(piperidinyl) and/or (morpholinyl)prop-2-en-1-ones **69**, in 55-75% yields (Scheme 37).[39,100,102]



Scheme 37

When 3-(dimethylamino)-1-(4-methyl-2-(tosylamino)thiazol-5-yl)prop-2-en-1-one (**70**) was treated with phenyl isothiocyanate, in the presence of potassium hydroxide, 3-mercapto-2-[4-methyl-2-(tosylamino)thiazole-5-carbonyl]-3-phenylaminopropenal was obtained (**71**) (Scheme 38).[22]



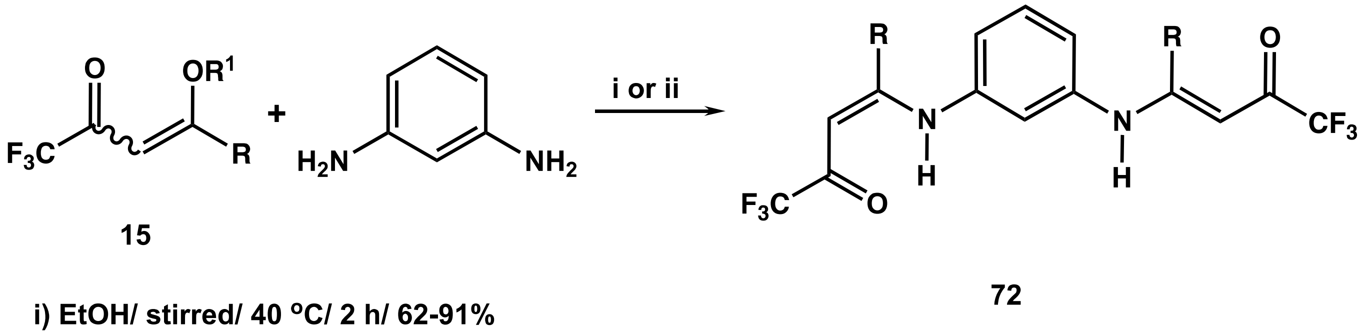
Scheme 38

3. SYNTHESIS OF *BIS*-ENAMINONES

In addition to the reported methods for the synthesis of enaminones, some *bis*-enaminones were also prepared as collected below.

3.1. From *β*-alkoxy ketones

N,N′-*Bis*{1-[aryl(heteroaryl)]-4,4,4-trifluoro-3-oxo-1-buten-1-yl}-1,3-phenylenediamines **72** was isolated, in 47-91% yields, by reaction of 4-alkoxy-4-(aryl/heteroaryl)-1,1,1-trifluoroalk-3-en-2-ones **15** with 1,3-phenylenediamine**,** at a molar ratio 2:1, under different reaction conditions (Scheme 39).[105]



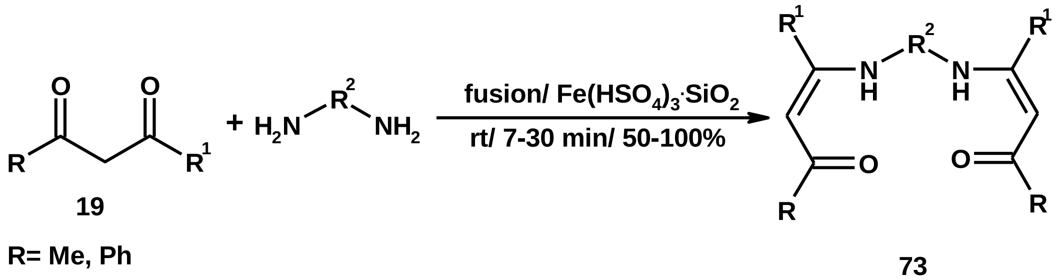
i) EtOH/ stirred/ 40 °C/ 2 h/ 62-91%
ii) H_2O/ $CHCl_3$/ stirred/ 60 °C/ 4 h

R= H, 2-furyl, Ph, 4-FPh, 4-ClPh, 4-BrPh,
4-NO_2Ph, 4-MePh, 4-MeOPh
R^1= Me, Et

Scheme 39

3.2. From *β*-diketones

Condensation of diamines with dicarbonyl compounds **19** under solvent-free conditions, in the presence of silica ferric hydrogen sulfate [$Fe(HSO_4)_3 \cdot SiO_2$] as a catalyst, afforded the corresponding *bis*-enaminones **73**, in good to excellent yields (Scheme 40).[106]



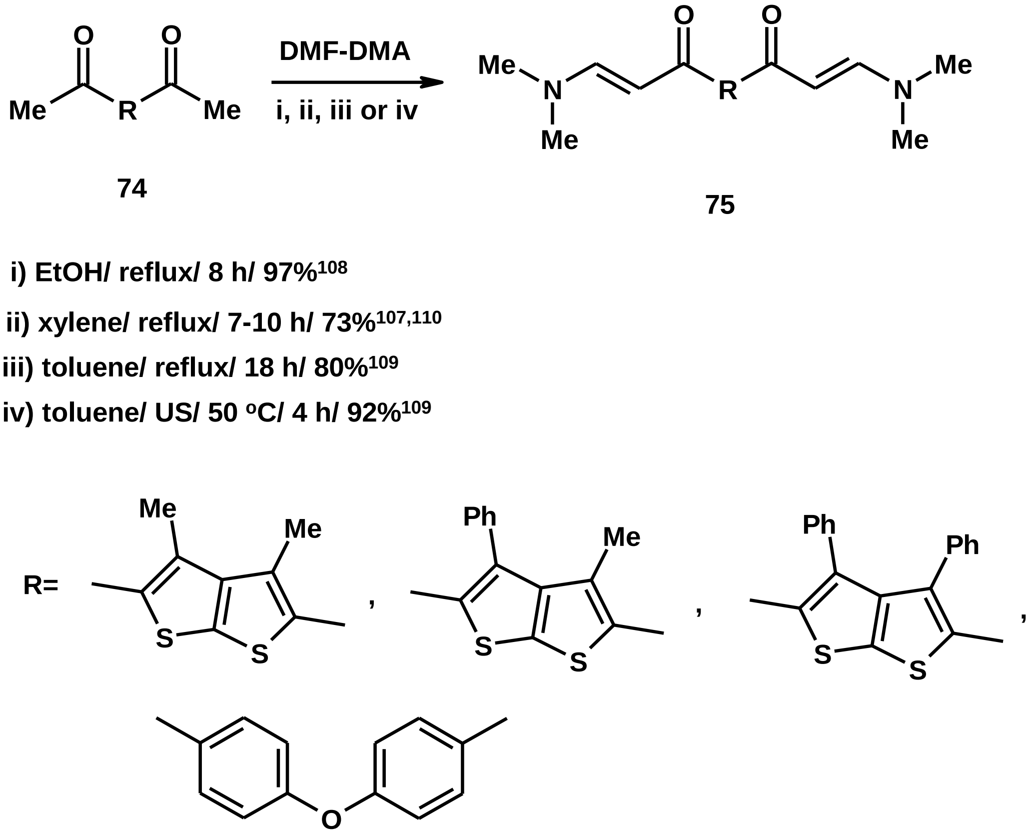
19

73

R= Me, Ph
R^1= Me
R^2= $(CH_2)_2$, $(CH_2)_3$, 1,2-Ph, 1,4-Ph

Scheme 40

Reaction of diethanone derivatives **74** with DMF-DMA, under different reaction conditions (in refluxing solvent or under ultrasonic irradiations), gave the corresponding *bis*-enaminone derivatives **75** (Scheme 41).[107-110]



74

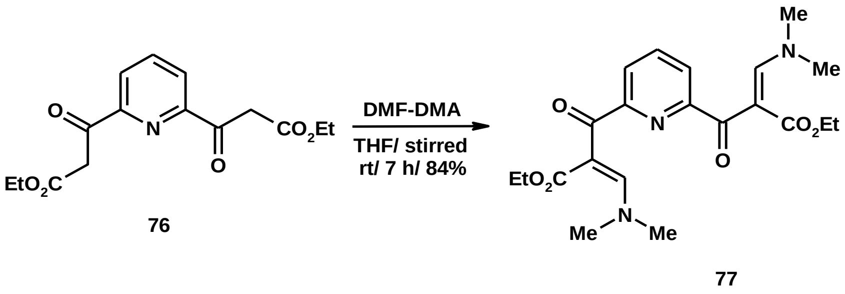
75

i) EtOH/ reflux/ 8 h/ 97%[108]
ii) xylene/ reflux/ 7-10 h/ 73%[107,110]
iii) toluene/ reflux/ 18 h/ 80%[109]
iv) toluene/ US/ 50 °C/ 4 h/ 92%[109]

Scheme 41

3.3. From *β*-ketoesters

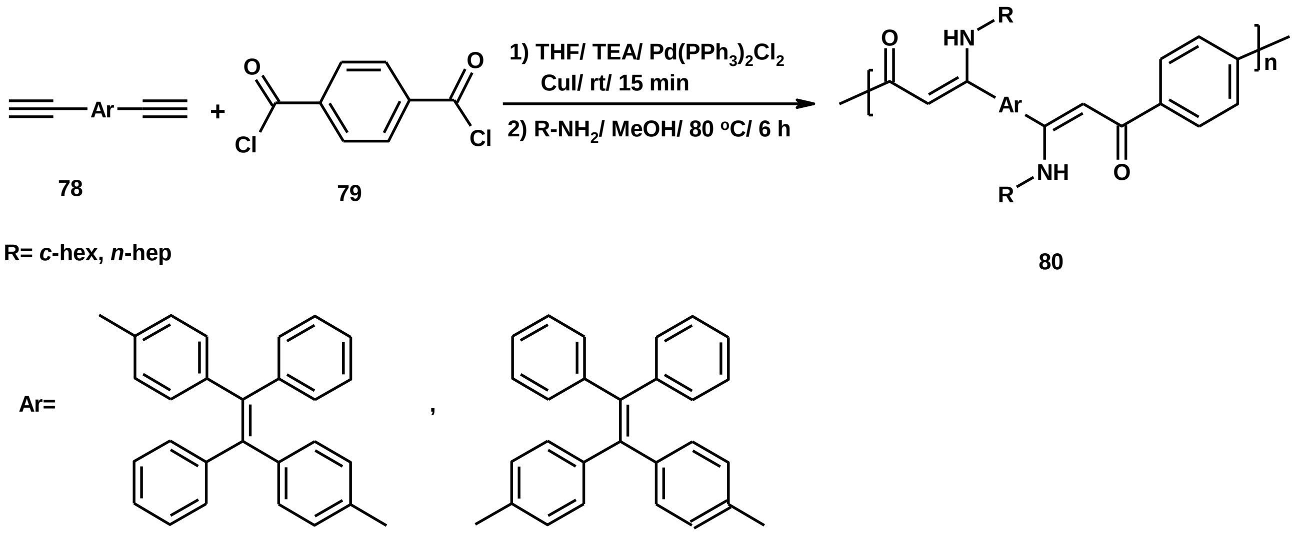
Pyridine-2,6-*bis*[ethyl 2-(*N*,*N*-dimethylamino)methylene-3-oxopropanoate] (**77**) was prepared by the reaction of pyridine-2,6-*bis*(ethyl 3-oxopropanoate) (**76**) with 1,1-dimethoxytrimethylamine, in dry THF (Scheme 42).[111]



Scheme 42

3.4. From acyl chloride

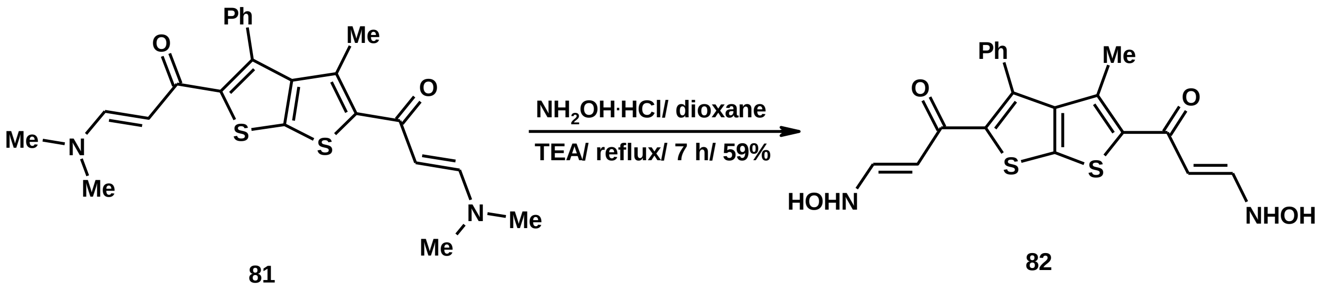
The sequential polymerizations of tetraphenylethene (TPE)-containing diynes **78**, terephthaloyl dichloride (**79**) and primary amines furnished conjugated polymers **80**. This polymerization was carried out in THF under nitrogen in the presence of Pd(PPh$_3$)$_2$Cl$_2$, CuI and TEA as depicted in Scheme 43.[112]



Scheme 43

3.5. From other enaminones

1,1'-(3-Methyl-4-phenylthieno[2,3-*b*]thiophene-2,5-diyl)*bis*(3-(hydroxyamino)-prop-2-en-1-one) (**82**) were prepared by reaction of enaminone **81** with hydroxylamine hydrocholoride, in refluxing dioxane (Scheme 44).[107]



Scheme 44

4. CONCLUSION

In conclusion, a variety of enaminones and *bis*-enaminones linked a numerous substituents were efficiently obtained starting from a diversity of substrates and reagents. The methods developed to synthesize these enaminones were collected according to the synthons employed.

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