

Research Article

Ultrasound Synthesis of Five-Membered Heterocycles

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Abstract

Five-membered ring heterocyclic compounds are a cyclic structure with at least one different kind of hetero atoms in the ring. Nitrogen, oxygen, and sulphur are the most common heteroatoms. In recent years, Five-membered rings heterocyclic compounds analogues and derivatives have a great interested due to their biological and pharmacological properties. Ultrasonic irradiation has been found a unique method for activation and acceleration of organic synthesis as well as a flexible platform for synthesis five- membered rings to get better yields, economic and eco- friendly reactions. Ultrasound irradiation enables many chemical reactions to proceed, even with some reactions which could not be achieved under conventional (**Figure 1**).In this short review; we summarized the most important methods for the ultrasound synthesis of five-membered heterocycles from 2010 up to 2015.

Keywords: Ultrasound Irradiation, green synthesis, five-membered rings, heterocyclic compounds



Figure 1 Ultrasonic cleaning bath

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Introduction

Ultrasound-assisted organic synthesis is used as a modern and eco-friendly technique that is being used to accelerate organic synthesis [1-4]. The chemical effects of ultrasound were first reported by Richards and Loomis in 1917 [5].

The effect of ultrasound during organic reaction is due to cavitation. The rarefaction-compression cycle in activation process, which involves the separation of molecules of liquids and then the collapse of the bubbles, provides strong impulsions that generate short-lived regions with high temperature and pressure. Such localized hot spots can be thought of as micro reactors in which the sound energy is converted into beneficial chemical form [6-9](**Figure 2**).

The use of ultrasound to accelerate reactions has proven to be a useful tool for meeting the green chemistry goals of minimization the production of waste and reduction of energy requirements. Ultrasound may display cleaner reactions by improving yields and selectivities particularly those involving free radical intermediates. Sonochemistry is widely used for improving the reactions that use expensive reagents, high temperatures and prolonged reaction conditions [10-14].

Heterocyclic compounds are present in a wide variety of many natural occurring compounds, vitamins, drugs, biomolecules, pigments and biologically active compounds. Most of the heterocycles have a wide range of applications in synthetic pharmaceuticals, agrochemicals and industrial chemistry [15-20].

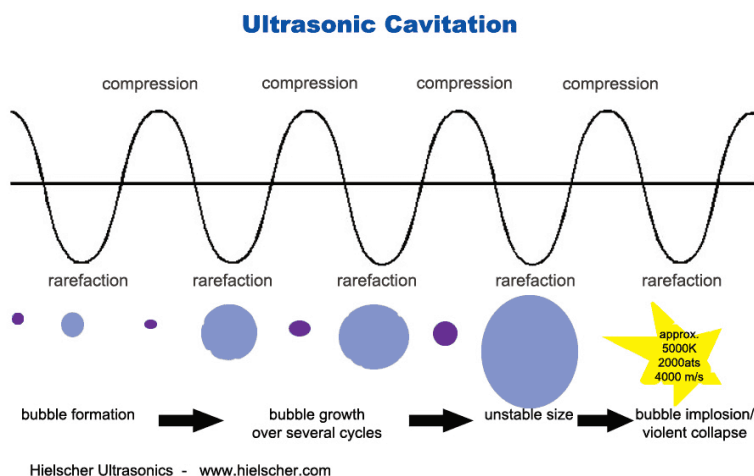
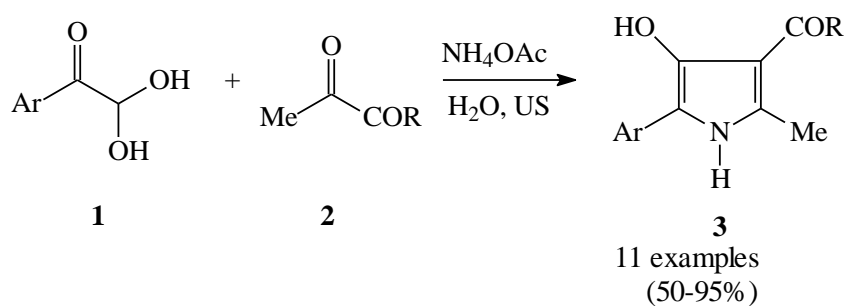


Figure 2 Ultrasound Cavitation

Applications of ultrasound in five-membered heterocyclic rings formation

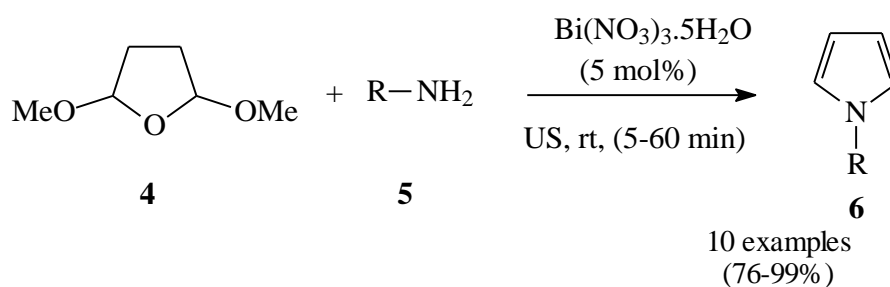
Synthesis of five-membered rings with one hetero atom

5-Aryl -4-hydroxy -1H- Pyrrole-3-carboxylic acid esters (**3**) were synthesized via three component reaction of substituted arylglyoxal hydrate (**1**) and β -dicarbonyl compounds (**2**) in the presence of ammonium acetate in aqueous media [21] (**Scheme 1**). The products were obtained in good yields without need for purification.



Scheme 1

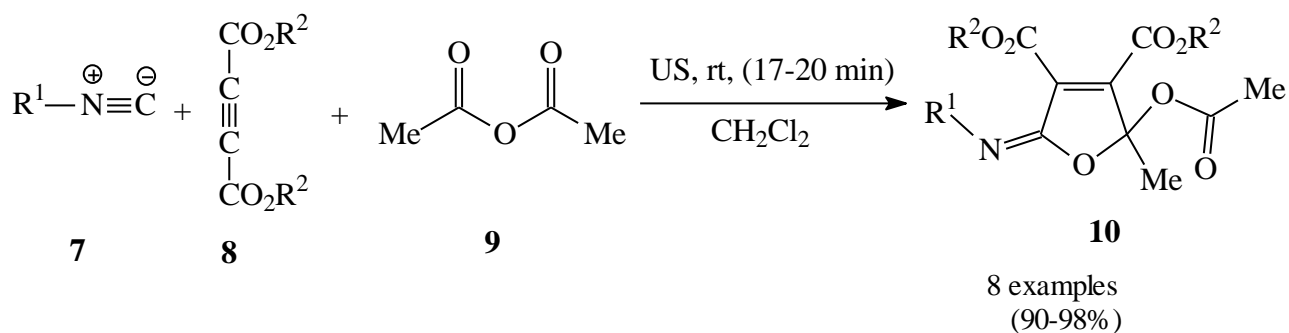
A series of N-substituted pyrrole derivatives (**6**) were synthesized by reaction of 2,5-dimethoxy tetrahydrofuran (**4**) and various amines (**5**) in the presence of catalytic amount (5 mol%) of bismuth nitrate pentahydrate under ultrasonic irradiation at room temperature [22] (**Scheme 2**).



Scheme 2

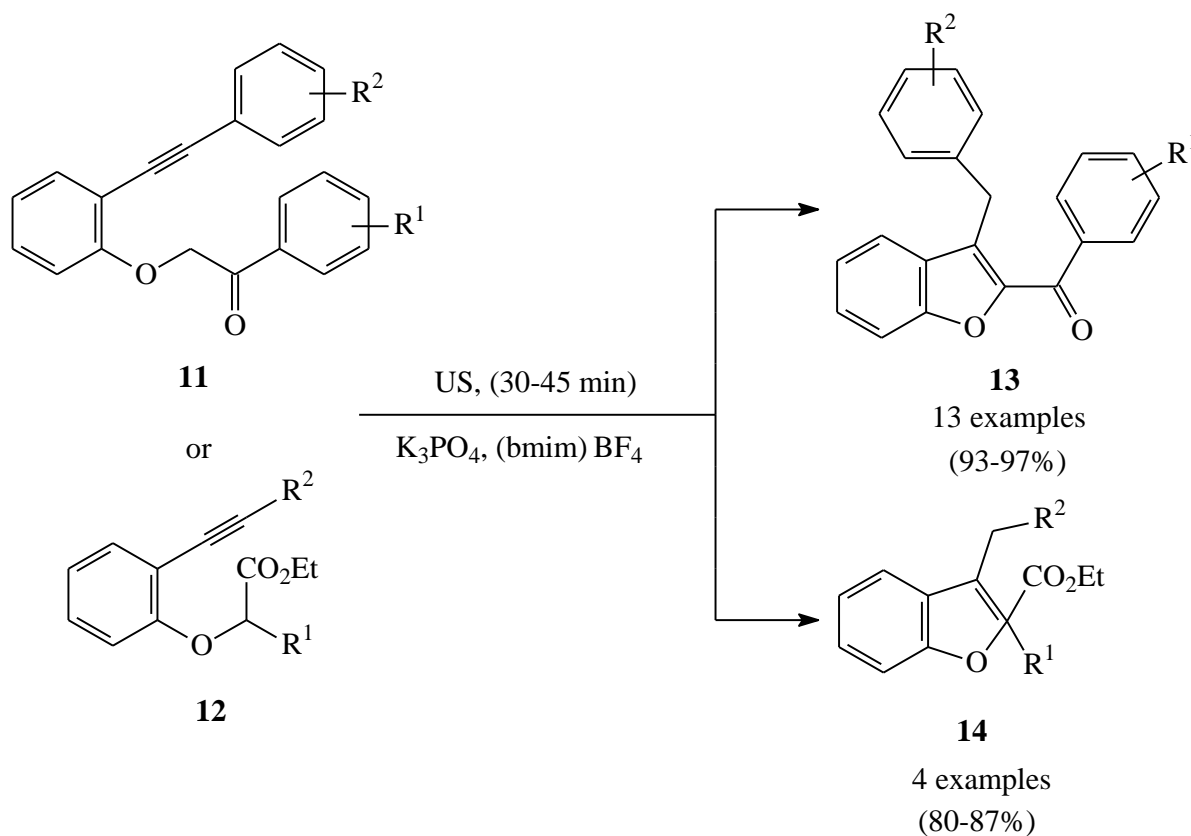
A comparative study in the absence of ultrasound showed that the products were isolated in lower yield (47-60%) and required longer reaction times (12 h.).

Very efficient and rapid catalyst free one-pot three component synthesis of 3,5-dihydro-5-imino-2-methyl furan 3,4-dicarboxylate derivatives from the reaction of isocyanides (**7**) and dialkyl acetylene dicarboxylates (**8**) in the presence of acetic anhydride (**9**) in CH_2Cl_2 under ultrasound irradiation [23] (**Scheme 3**).



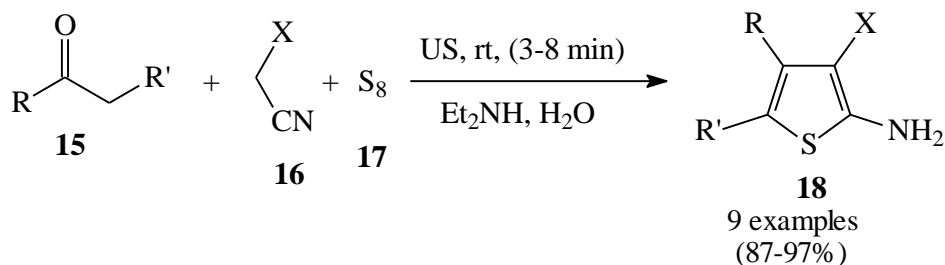
Scheme 3

The synthesis of 2,3-disubstituted benzo[b]furan derivatives (**13**, **14**) was achieved by 5 exo-dig carbanion-yne intramolecular cyclization for compounds (**11**) or (**12**) respectively, using anhydrous potassium phosphate in ionic liquid (1-butyl-3-methyl imidazolium tetrafluoroborate) [(bmim) BF_4] under ultrasound irradiation at room temperature [24] (**Scheme 4**). The ionic liquid was used as catalyst as well as reaction medium for the formation of C-C bond.



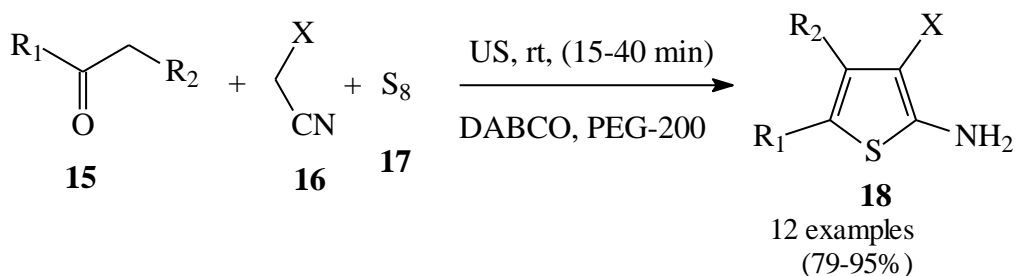
Scheme 4

2-Aminothiophene (**18**) were prepared by the reaction of ethyl cyanoacetate or malonitrile (**16**) with α -methylene carbonyl compounds (**15**) and elemental sulfur (**17**) in the presence of $\text{Et}_2\text{NH}/\text{H}_2\text{O}$ under US irradiation. All the products obtained in good yields and the reactions were completed within 5 min [25] (**Scheme 5**).



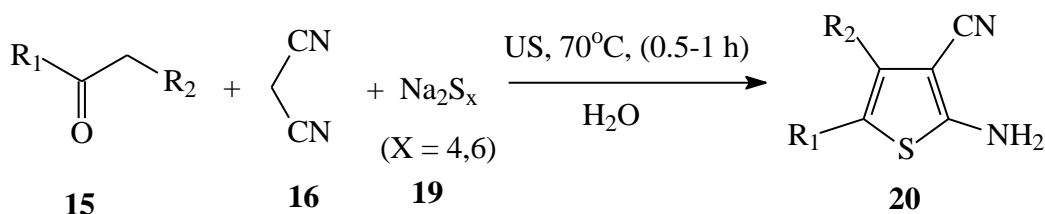
Scheme 5

Linag and co-workers [26] reported the synthesis of multisubstituted 2-amino thiophene derivatives (**20**) via intramolecular cyclization of α -methylene carbonyl compounds (**15**) and cyano derivatives (**16**) and sulfur (**17**) in the presence of catalytic amount DABCO (1,4-diaza bicyclooctane) as an Gewald reaction base in PEG-200 (polyethylene glycol -200) under ultrasound irradiation for (15-40 min) in moderate to high yields (79-94%). (**Scheme 6**)



Scheme 6

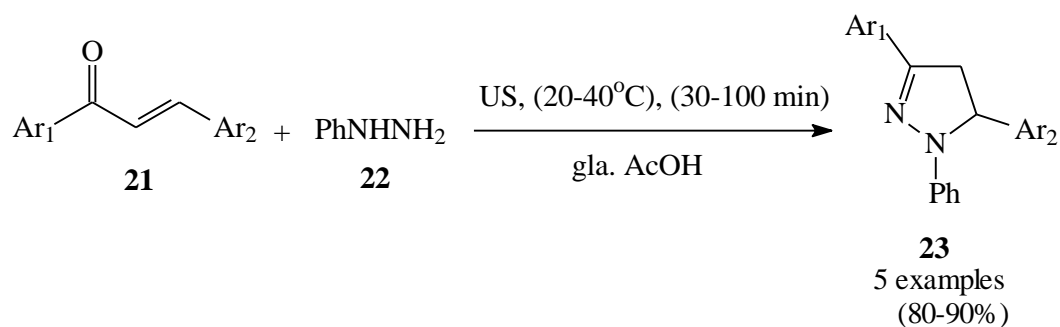
One-pot ultrasound-irradiated aqueous reaction between ketones or aldehydes (**15**), malononitrile (**16**) and sodium polysulfide (**19**). The reactions are converted into the corresponding 2-aminothiophene derivatives in moderate to high yields [27] (**Scheme 7**). In comparison with conventional methods, the prominent features of this sonocatalyzed procedure are experimental simplicity, good functional group tolerance, atom efficiency, and the use of water as a green solvent.



Scheme 7

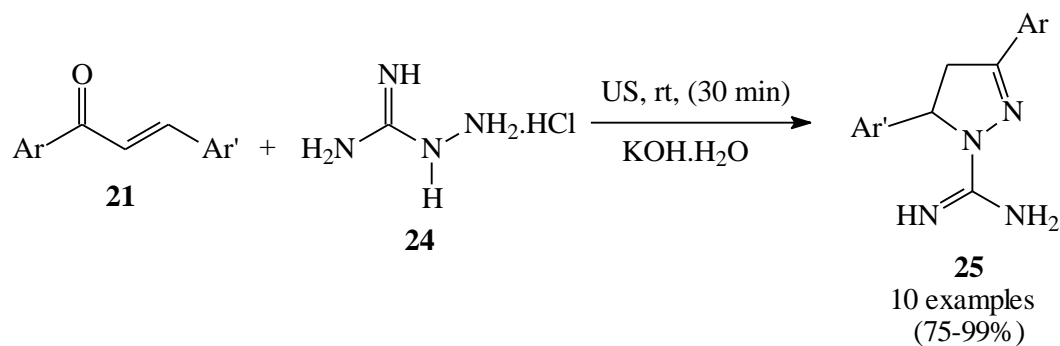
Synthesis of five-membered rings with two hetero atoms

US-promoted synthesis of N-aryl pyrazolines (**23**) was reported by Gupta and coworkers 2010 [28]. The pyrazoline ring was obtained from the cyclization reaction of chalcones (**21**) with phenyl hydrazine (**22**) in the presence of acid catalyst (glacial acetic acid) in good yields (**Scheme 8**).

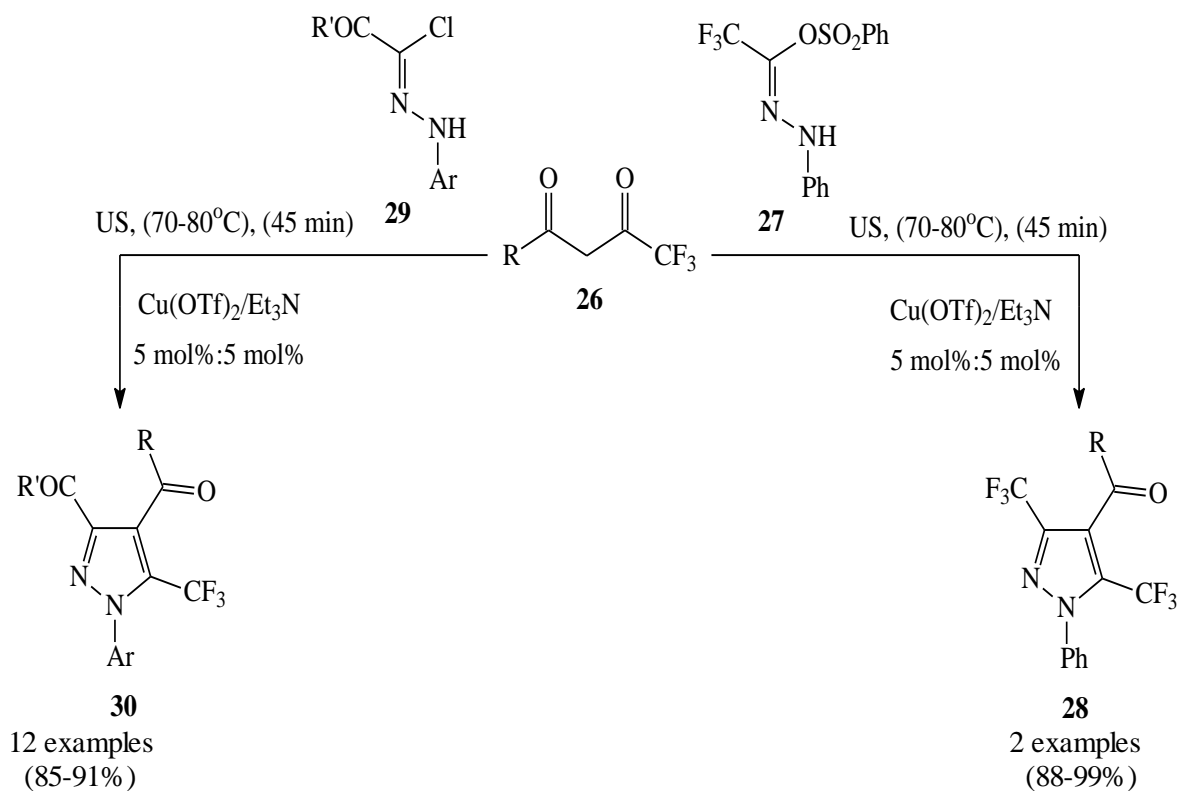


Scheme 8

3,5-Diaryl-4,5-dihydro-1H-pyrazole-1-carboximidamides (**25**) were prepared via the efficient reaction of chalcones (**21**) and aminoguanidine hydrochloride (**24**) in aqueous KOH under US irradiation [29] (**Scheme 9**).



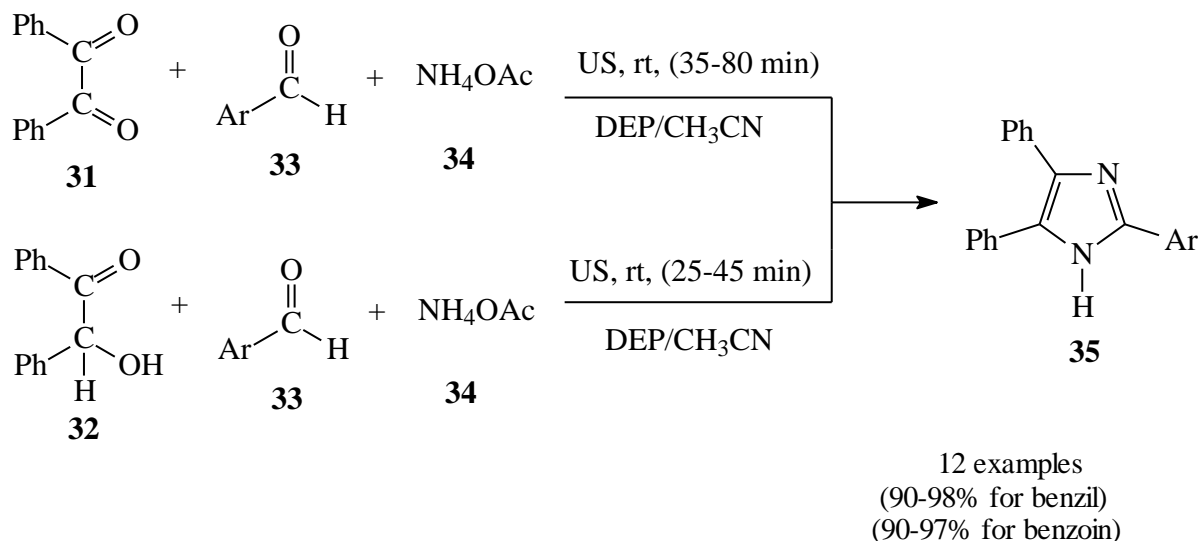
Scheme 9



Scheme 10

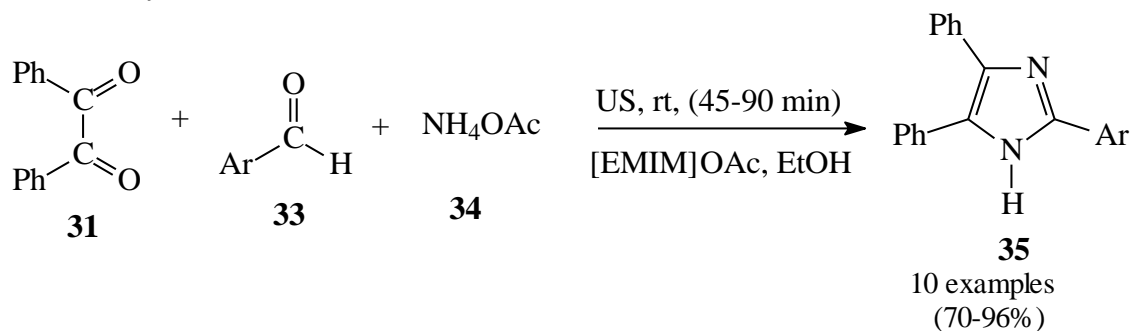
Green and regioselective synthesis of pyrazole containing the trifluoromethyl moiety (**28**, **30**) was achieved by the reaction of β -diketone derivatives (**26**) with trifluoromethyl carbohydrazonoyl benzene sulfonate (**27**) or α -keto hydrazonoyl halides (**29**) respectively [30]. The reactions were catalyzed by copper triflate/triethyl amine in ethanol under us irradiation and thermal condition. The sonochemical technique gave excellent yields in shorter time than the corresponding classical reaction (**Scheme 10**).

Nagargoie and co-workers [31] reported a new user friendly one-pot procedure for the synthesis of 2-aryl-4,5-diphenyl-1H-imidazoles (**35**) by the condensation reactions of benzil (**31**) or benzoin (**32**), with aromatic aldehyde (**33**) and ammonium acetate (**34**) using diethyl bromophosphate (DEP) as a mild oxidant under ultrasound irradiation at r.t. (**Scheme 11**)



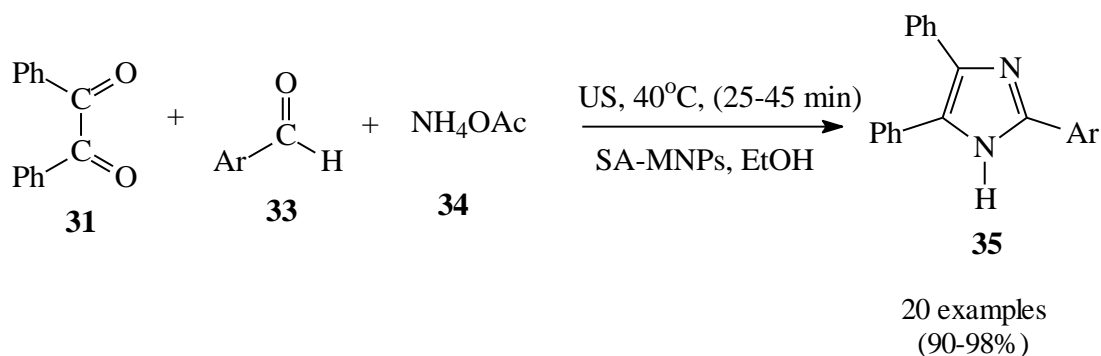
Scheme 11

A one pot three component efficient and mild method for synthesis of 2-aryl-4,5-diphenyl imidazole (**35**) from benzil (**31**), aromatic aldehyde (**33**) and ammonium acetate (**34**) using 1-ethyl-3-methyl imidazole acetate ([EMIM]OAc) as a catalyst under ultrasound irradiation at room temperature was reported by Zang and co-workers [32]. Their study revealed that the use of sonication combining ionic liquid offer several advantages including milder conditions, easy work-up and excellent yields (**Scheme 12**).



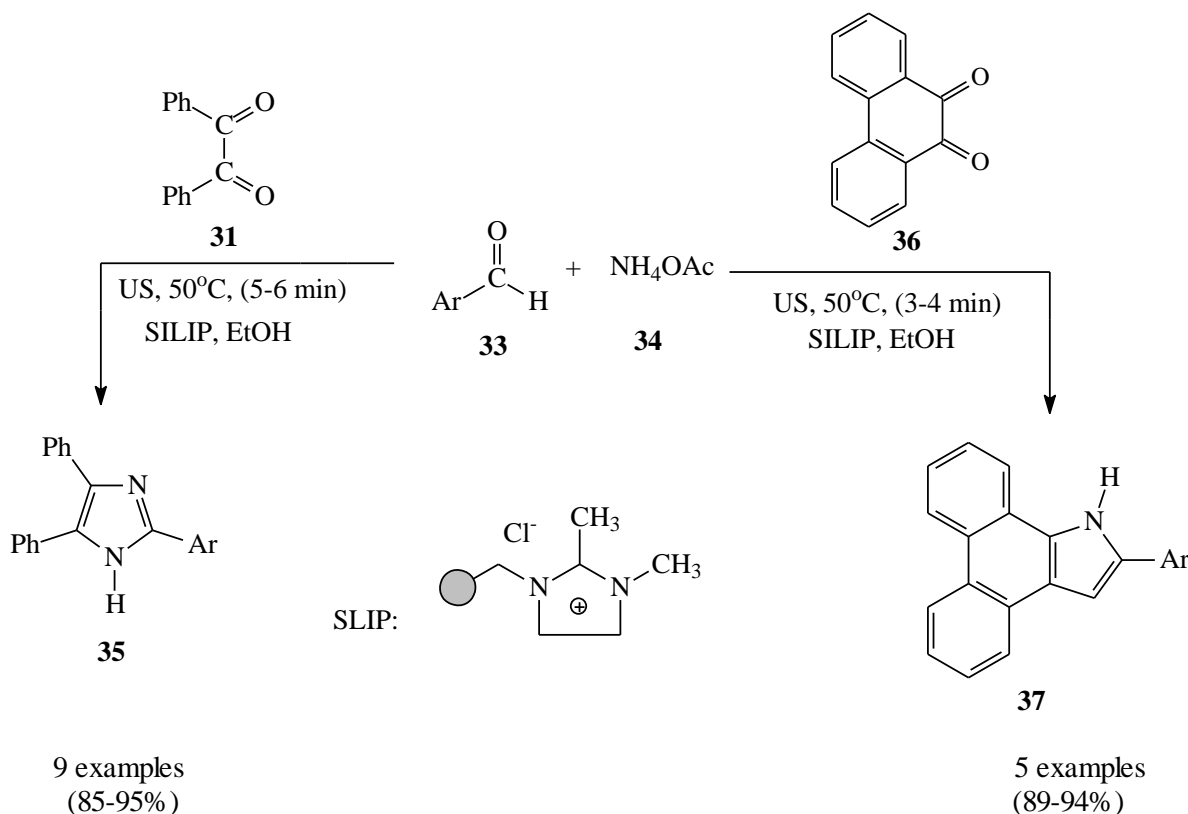
Scheme 12

Also, the reaction between benzil (**31**), aromatic aldehyde (**33**) and ammonium acetate (**34**) was evaluated using sulfamic acid-functionalized iron oxide magnetic nanoparticles (SA-MNPs) as a solid acid catalyst under ultrasound irradiation [33] (**Scheme 13**).



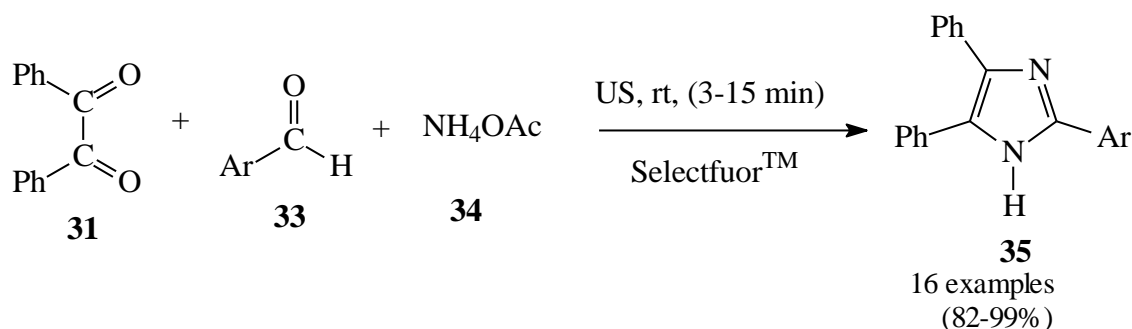
Scheme 13

In 2013, Jourshari and co-workers [34] reported the synthesis of imidazole derivatives by the reactions of 9,10-phenanthrene quinone (**36**) or benzil (**33**) using supported ionic liquid like phase (SILIP) as a green catalyst (prepared by using Merrified resin 1% cross linked, 200-400 Mesh, 1-13 mmol/g) under ultrasonic irradiation. Ultrasonic irradiation reduced the reaction time (3-6 min) compared to conventional heating (20 min). The catalyst is easily separated from reaction mixture by filtration and also displays recyclable activity (**Scheme 14**).

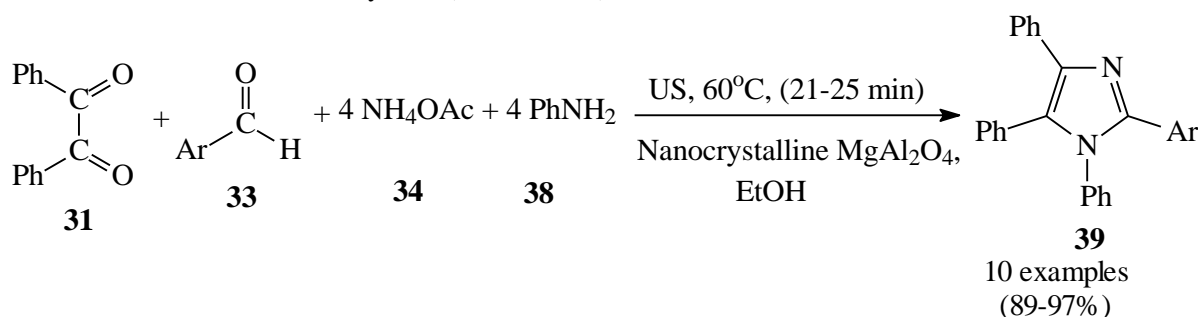


Scheme 14

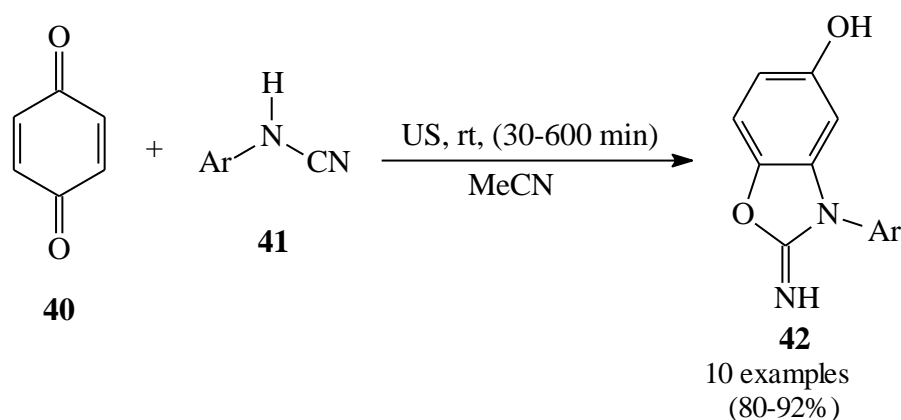
Heravi and co-workers [35] showed that selectfluor™ (15% mol) can also be used as a catalyst with ultrasound irradiation for the synthesis of 2,4,5-trisubstituted imidazoles (**35**) by a one pot reaction of benzil (**31**), aromatic aldehydes (**33**) and ammonium acetate (**34**). The products were obtained in excellent yield in short reaction times (**Scheme 15**).



1,2,4,5-Tetrasubstituted imidazoles (**39**) have been prepared by utilization of nano-crystalline magnesium aluminated MgAl_2O_4 in ethanol under ultrasound irradiation [36]. The condensation of benzil (**31**), aromatic aldehydes (**33**), ammonium acetate (**34**) and primary aromatic amine (**38**) in the presence of nanocrystalline magnesium aluminate in ethanol under ultrasound irradiation at 60°C , the reaction were completed within (12-25 min), and the target compounds were obtained in excellent yields (**Scheme 16**).

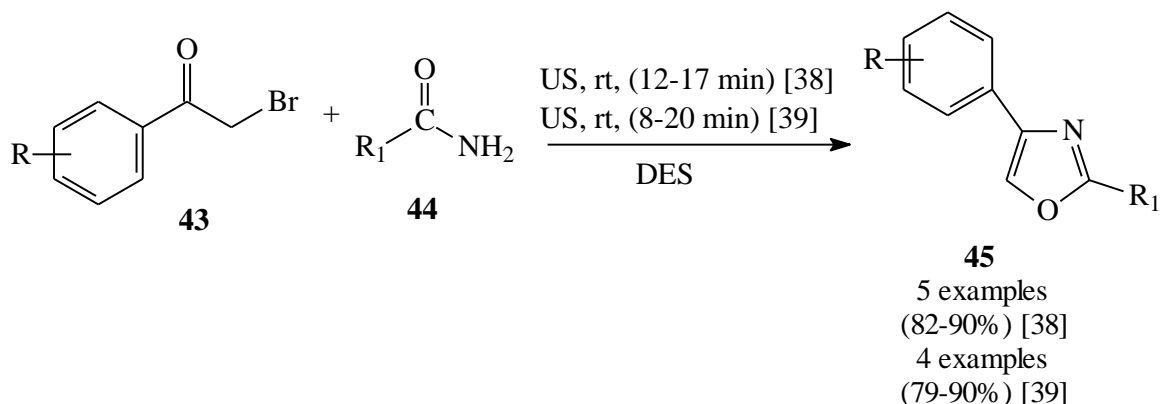


p-Benzoquinone (**40**) reacted with several aromatic cyanamides (**41**) in acetonitrile under US irradiation at room temperature to give 2-imino-3-aryl-2,3-dihydrobenzo[d]oxazole-5-ol derivatives (**42**) in excellent yields [37]. (**Scheme 17**).



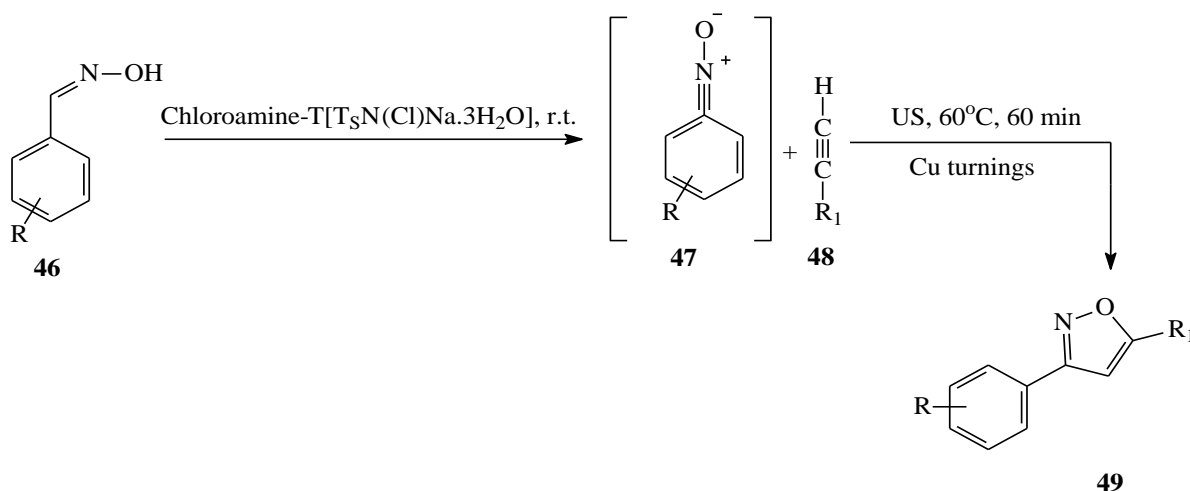
In 2013, Singh and co-workers [38] described the formation of 2,4-disubstituted oxazole derivatives (**45**) via thermal and ultrasonic technique. The reaction between phenacyl bromide derivatives (**43**) and amide (**44**) in the presence of deep eutectic solvent (DES) [choline chloride (1 eq.)-urea (2 eq.)] as a green solvent (**Scheme 18**). The sonochemical method improved yields, reduced reaction time, and also saved 85% energy. DES solvent proved to be recyclable. It was able to re-used four times with little loss in yield. The scope of the ultrasound assisted methodology in deep

eutectic solvent towards the synthesis of oxazole derivatives under ultrasound or conventional technique was extended by the same groups [39]. The sonochemical method increased the yields and rate of reaction at target compounds (improvement by 60%) as observed by XRD studies. This fact was proven by SEM images which showed rod-shaped crystals in contrast to clustered aggregates obtained by thermal method.



Scheme 18

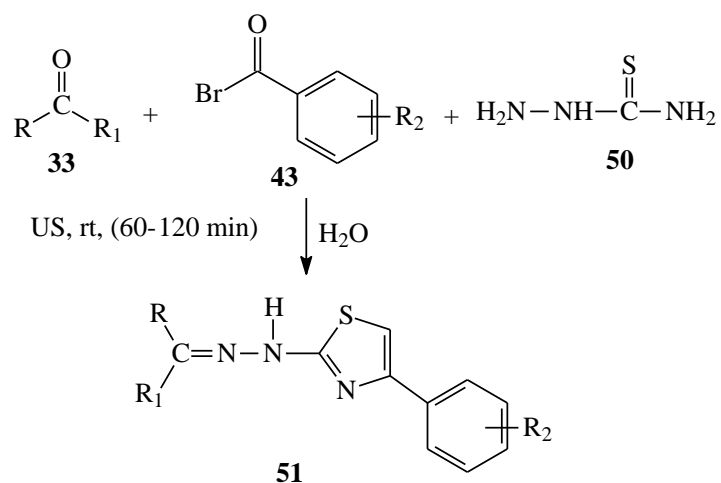
Koufaki and co-workers [40] reported a one-pot regioselective synthesis of 3,5-disubstituted isoxazoles (**49**) based on copper(I) catalyzed 1,3-dipolar cycloaddition reaction between *in situ* generated nitrile oxides (**46**) and alkynes (**48**) under ultrasonic irradiation (Scheme 18). The combined use of 40 kHz ultrasonic bath and 20 kHz probe decreased the reaction time from 72 h to 1 h with increased yields. However, when reactions carried out under metal free conditions, the 1,3-dipolar cycloadditions were regioselective giving low to good yields. (Scheme 19)



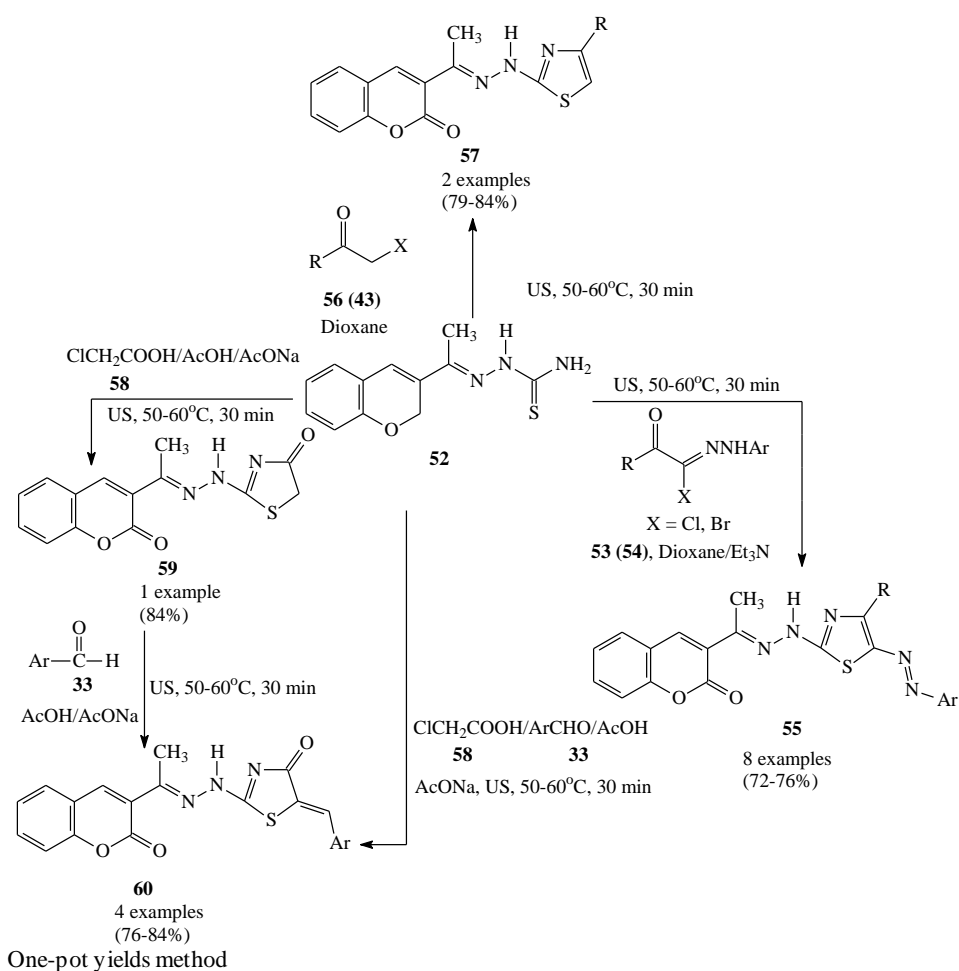
[T_sN(Cl)Na•3H₂O]: [N-chloro-4-methylbenzenesulfonamide sodium salt.

Scheme 19

N-(4-Arylthiazoly-2-yl)hydrazones (**51**) were prepared by US-promoted multicomponent reaction between aromatic aldehydes or ketones (**33**), substituted phenacyl bromide (**43**) and thiosemicarbazide (**50**) [41]. The reactions were carried out in water at room temperature within (60-120 min). The sonochemical method afforded better yields in shorter times than the corresponding conventional stirring. (Scheme 20)



12 examples
(86-96%)
Scheme 20



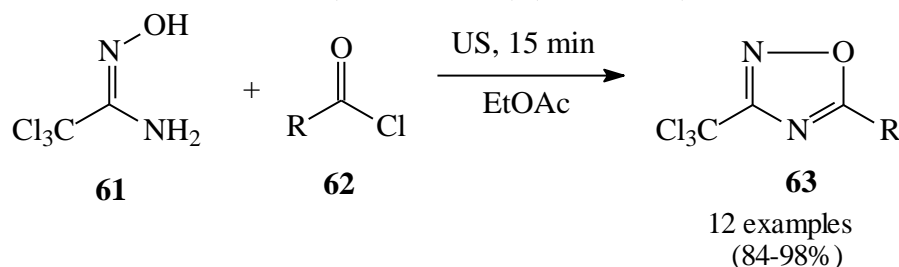
Scheme 21

Gomha and co-workers [42] reported the synthesis of some thiazoles bearing a coumarin nucleus, under ultrasound irradiation. The cyclization of thiosemicarbazide derivatives (**52**) and hydrozonyl halides derivatives (**53**) or (**54**) in the presence of TEA under ultrasound irradiation at 50-60°C for 30 min afforded thiazolyl-chromenone derivatives (**55**) in good yields. The thiazole derivatives (**57**) were synthesized by the reaction of (**52**) with chloroacetone (**56**) or phenacyl bromide (**43**) in dioxide under ultrasound irradiation.

The 4-thiazolidinone derivatives (**59**) were prepared from the reactions of thiosemicarbazide (**52**) with chloroacetic acid (**58**) in glacial acetic acid in the presence of anhydrous sodium acetate. In addition, the arylidines (**60**) were prepared by ultrasonic irradiation of 4-thiazolidinone derivatives (**59**) and aromatic aldehydes (**33**) in glacial acetic acid in presence of anhydrous sodium acetate. One pot synthesis has been also used to synthesis compounds (**60**) under US irradiation in good yields (**Scheme 21**).

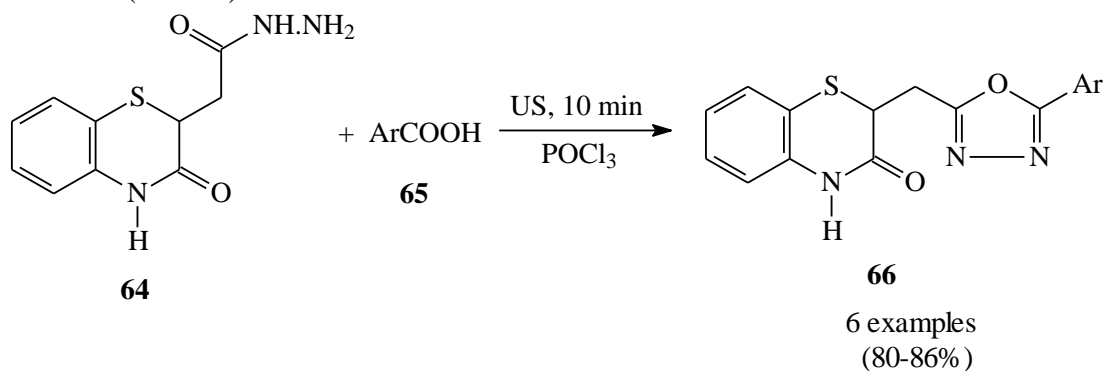
Synthesis of five-membered rings with three hetero atoms

US-promoted synthesis of 3-trichloromethyl-5-alkyl(aryl)-1,2,4-oxadiazoles (**63**) was reported by Bretanha and co-workers [43]. The heterocycles rings was gained from the reaction of trichloroacetoamidoxime (**61**) with acyl chloride (**62**) in ethyl acetate. The products were obtained in short times (15 min.) and excellent yields (84-98%), better than through the utilization of conventional methods (61-90% in 20h) (**Scheme 22**).



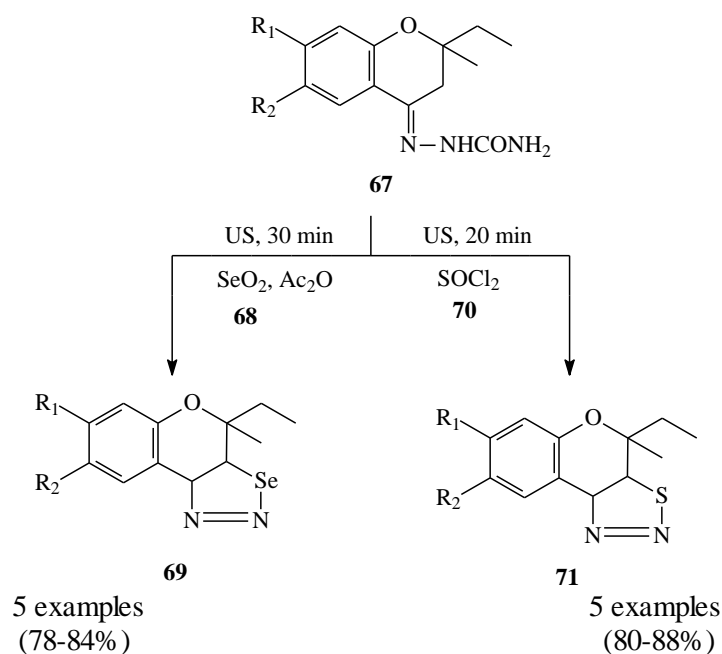
Scheme 22

The cyclization reaction between 2-hydrazinocarbonyl methyl-3-oxo-1,4-benzothiazine (**64**) and aromatic acids (**65**) afforded 1,3,4-oxadiazoles (**66**) [44] (**Scheme 23**). The reactions were catalyzed by phosphorous oxychloride under US irradiation. A comparison study shows that US irradiation proved to be faster with higher yields (80-86%) than conventional methods (52-59%).



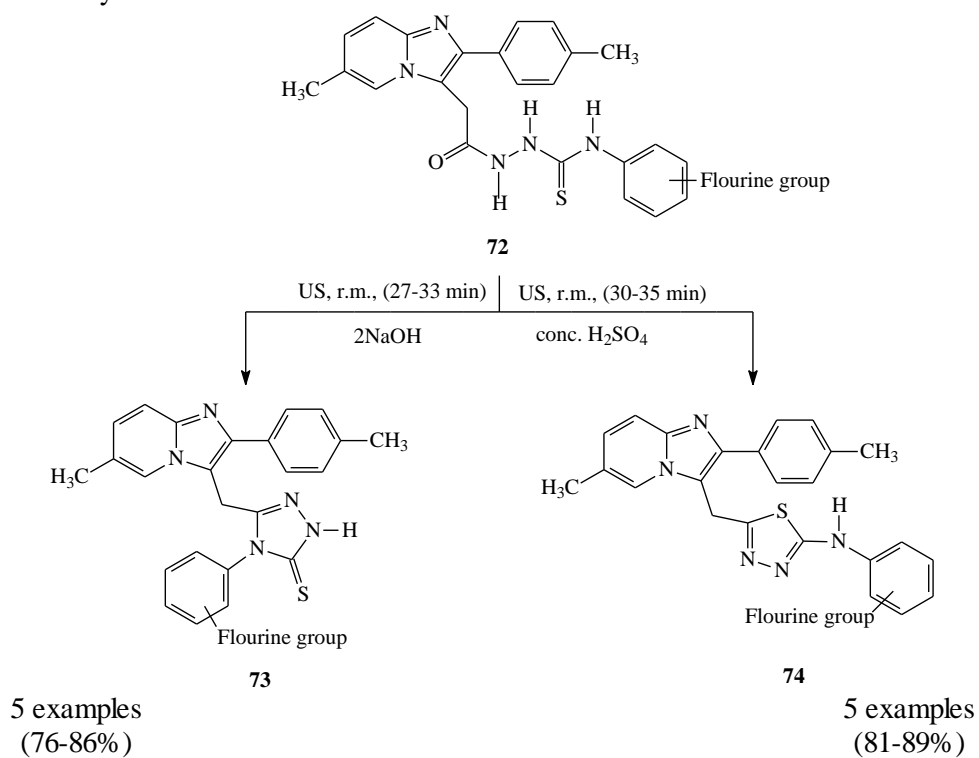
Scheme 23

Reaction of semicarbazones (**67**) with SeO_2 (**68**) in acetic anhydride under US irradiation for 30 min formed selenadiazole (**69**) in (78-84%) yields. The target compounds (**69**) also obtained under conventional thermal heating and microwave irradiation with (68-73%) in 2-3 h) and (86-95% in 10 min) respectively. Similarly, substrate (**67**), upon reaction with SOCl_2 (**70**) under ultrasonic, microwave irradiation and classical heating afforded 1,2,3-thiadiazoles (**71**) in (80-88%) in 20 min, (89-97%) in 5 min, and (69-74%) in 2 h respectively [45] (**Scheme 24**).



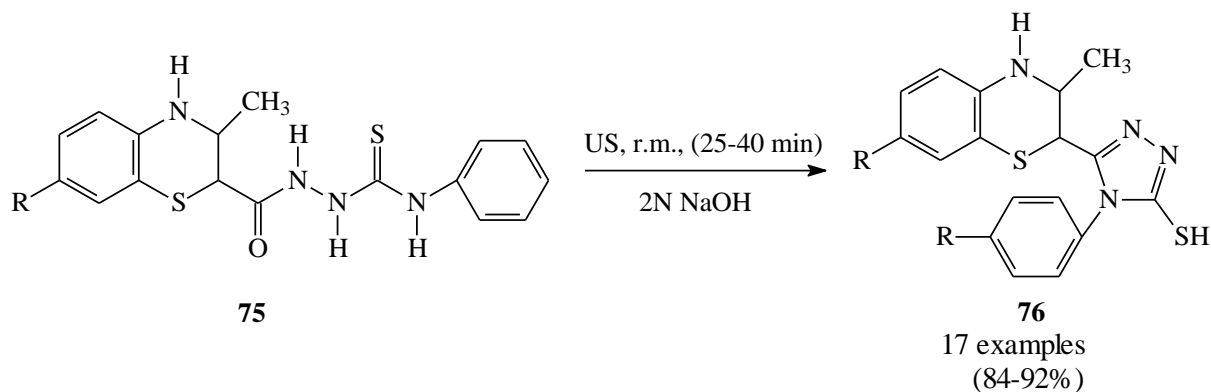
Scheme 24

An ultrasound-mediated synthesis of fluorinated triazole (**73**) and thiazoles (**74**) derivatives were reported by Salunkhe [46]. The products were achieved in good yields via the intramolecular cyclocondensation of thiosemicarbazide derivatives (**72**) in basic or acidic media, respectively (Scheme 25). The reactions were carried out by green synthesis technique such as MW irradiation and ultrasound method as well as conventional methods. Ultrasound gave the best yields.



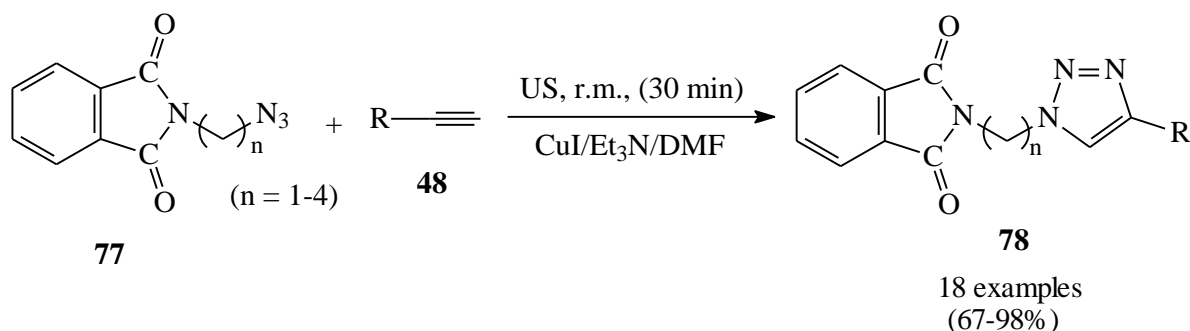
Scheme 25

The benzothiazonyl 1,2,4-triazole derivatives (**76**) have been synthesized from thiosemicarbazide derivatives (**75**) via intramolecular cyclization in 2N NaOH. The same reactions accomplished in the absence of sonication afforded lower yields (56-74% in 3 h) [47] (**Scheme 26**).



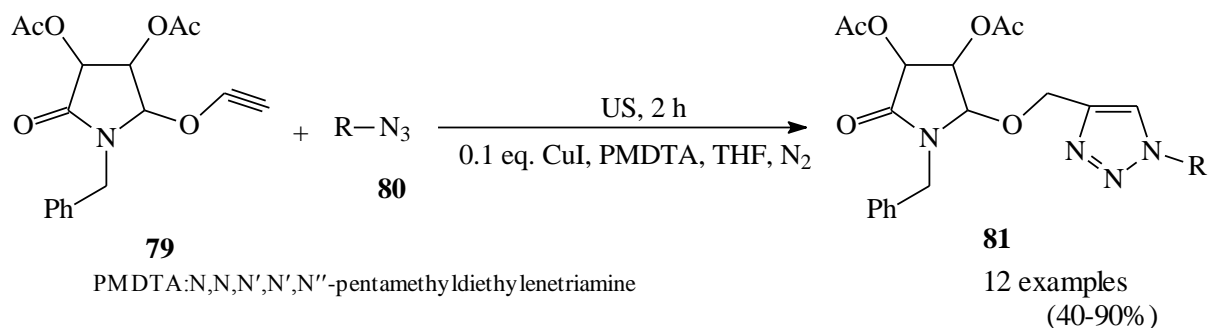
Scheme 26

In 2012, da Silva and co-workers[48] described a click chemistry method under ultrasound irradiation for synthesis a series of 1,2,3-triazole derivatives (**78**) from N-phthalimidoalkyl-azides (**77**), and alkyne derivatives (**48**) in the presence of CuI, in Et₃N and DMF as a solvent under US irradiation at room temperature. The products were obtained in good to excellent yields (67-98%). (**Scheme 27**)



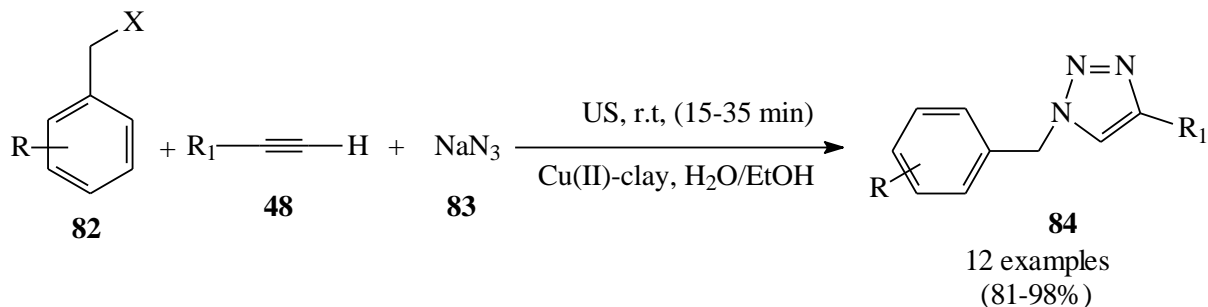
Scheme 27

In 2014, Stetani and co-workers [49] reported the synthesis of triazolyl pyrrolidone derivatives (**81**) through click chemistry process under US irradiation. Ether pyrrolidinones(**79**) reacted with alkyl(aryl) azides (**79**) to afforded the desired cyclization products 1,2,3-triazolyl pyrrolidones (**81**) in good to high yields (**Scheme 28**). The reaction rates showed a dependence on the substituent present on aromatic azides.



Scheme 28

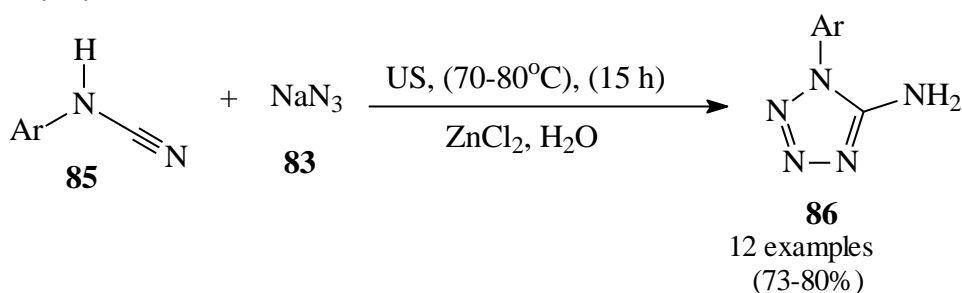
Several 1,4-disubstituted-1,2,3-triazolo derivatives have been prepared via one-pot assisted Huisgen [3+2] cycloaddition between benzyl bromide (**82**), aryl acetylene (**48**) and sodium azide (**83**) under US irradiation [50] (**Scheme 29**). The reaction was carried out in the presence of Cu(II) doped-clay, and the product was obtained in excellent yields. The catalyst could be recycled up to 6 consecutive cycles without significant loss in catalyst activity.



Scheme 29

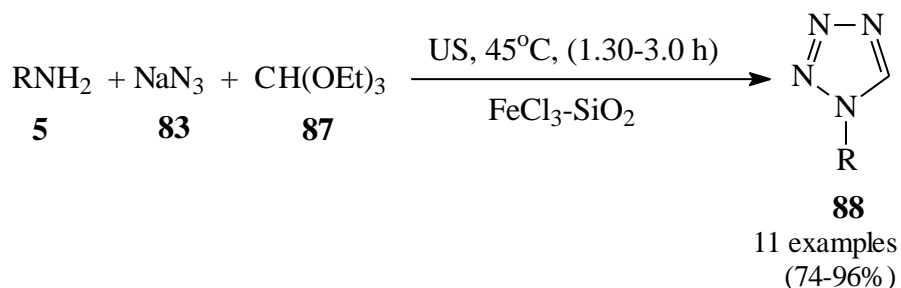
Synthesis of five-membered rings with four hetero atoms

The regioselective synthesis of the 1-aryl-5-amino-1H-tetrazoles (**86**) has been described by Habibi and co-workers [51]. Reaction of aryl cyanamides (**85**) with sodium azide (**83**) in the presence of ZnCl₂ under ultrasound irradiation at 70-80°C. The reaction afforded the products in good yields either with electron withdrawing or electron donating groups attached to aryl cyanamides (**85**). (**Scheme 30**)



Scheme 30

1-Aryl-1H-1,2,3,4-tetrazoles (**88**) were prepared by one-pot three components under US irradiation in solvent-free conditions [52]. The reactions of arylamines (**5**), sodium azide (**83**) and triethyl orthoformate (**87**) catalyzed by FeCl₃-SiO₂ as an effective heterogeneous catalyst were completed within (1.30-3.0 h) (**Scheme 31**). The products were isolated in moderate to excellent yields. In comparison to conventional methods, the US irradiation enhanced the yields and rates of reactions.

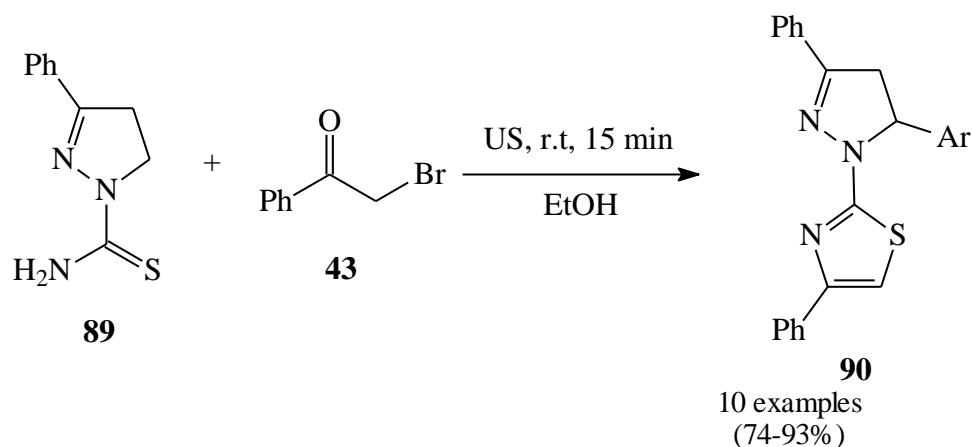


Scheme 31

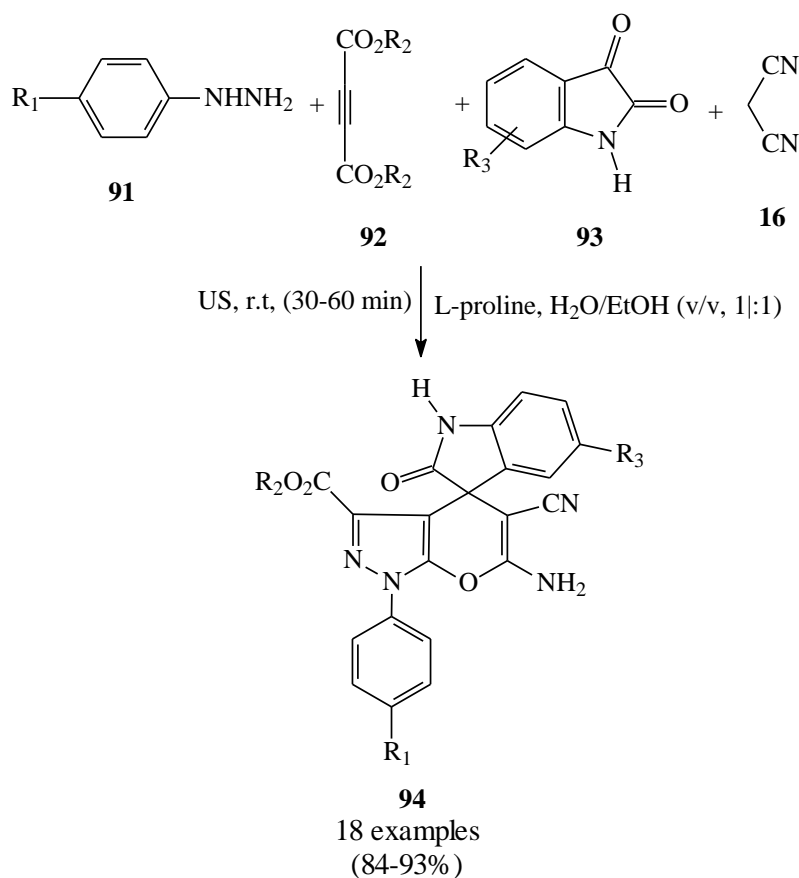
Miscellaneous

The cyclization reaction between 1-thiocarbamoyl-4,5-dihydro-1H-pyrazole (**89**) and phenacyl bromide (**43**) in ethanol under US irradiation at room temperature for 15 min. gave 2-(3,5-diaryl-1H-pyrazol-1-yl)-4-phenyl thiazole

derivatives (**90**) [53]. The sonochemical method gave better yields in shorter times (74-93% in 15 min) without the need for additional purifications than with conventional methodology (60-72% in 1 h). (**Scheme 32**)



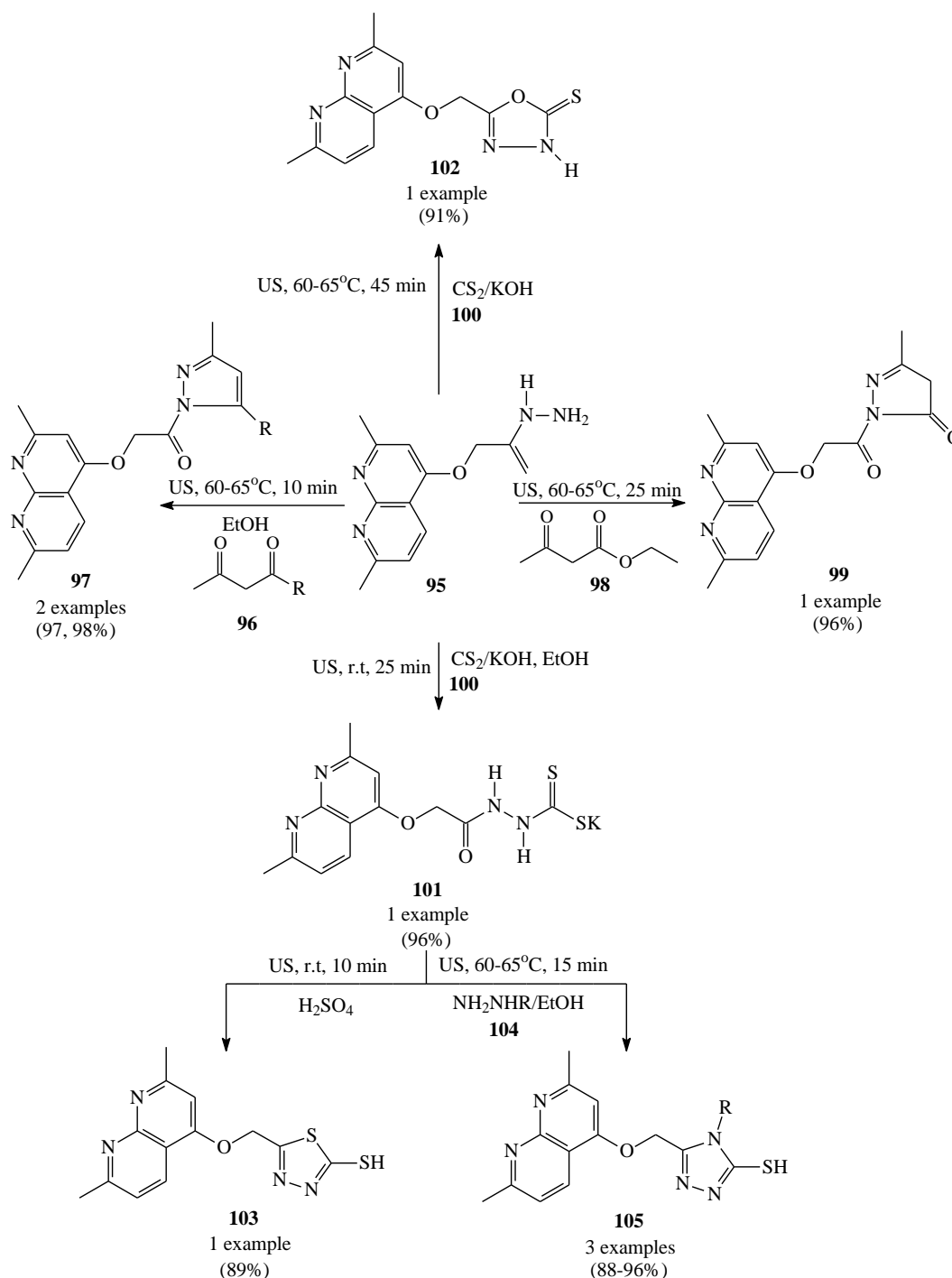
Scheme 32



Scheme 33

Recently, Liju and co-workers[54] reported the four components one-pot synthesis of spiro [indole-3,4'-pyrano[2,3-c] pyrazole derivatives (**94**) by the reaction between phenyl hydrazine derivatives(**91**), dialkyl acetylene dicarboxylate (**92**) with isatin (**93**) and malonitrile (**16**) catalyzed by L-proline (10 mol%) in water/ethanol (v/v, 1:1) under

ultrasonic irradiation at room temperature (**Scheme 33**). All products were obtained after easy workup in excellent yields.



Scheme 34

In 2014, Ahmed and co-workers[55] reported the synthesis of pyrazoles (**97** and **99**), 1,3,4-oxadiazole (**102**), 1,3,4-thiadiazole (**103**) and 1,3,4-triazoles (**105**) incorporated into naphthyridine nucleus under sonication conditions (**Scheme 34**). The reactions between 1,8-naphthyridine acetohydrazide derivatives (**95**) and the diketone namely acetyl acetone, benzoyl acetone (**96**) and ethyl acetoacetate (**98**) afforded compounds (**97**) and (**99**) respectively.

When compound (**95**) reacted with excess carbon disulphide (**100**) in ethanolic KOH under ultrasound irradiation at room temperature, the dithiocarbamate potassium salt (**101**) was isolated in yield 96%. However, when the temperature increases to 65°C, the oxadiazole derivative (**102**) was formed in good yield. When potassium salt was treated with concentrated H₂SO₄ at 0°C and subject to ultrasound irradiation, 1,3,4-thiadiazol-2-thiol derivatives (**103**) was isolated. Furthermore, reaction of (**101**) with hydrazine derivatives (**104**), under ultrasonic irradiation aminotriazole derivatives (**105**) was obtained. Also, the reaction, carried out under classical stirring without ultrasound, demand a longer reaction time than those require when ultrasound was used.

Conclusion

In conclusion, we have summarized the recent applications of ultrasound-promoted synthetic methodologies for preparation five-membered heterocyclic rings. In comparison to conventional methods, ultrasound irradiation provides several advantages such as enhanced reaction rates, selectivity, shorter reaction times, higher yields and environment friendly technique.

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Publication History

Received 24th Nov 2014
Revised 08th Dec 2014
Accepted 19th Dec 2014
Online 30th Dec 2014