

Strategies for the Synthesis of Thiazolidinone Heterocycles

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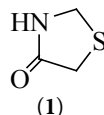
Abstract

Thiazolidinones is the special class of heterocyclic compounds with a broad spectrum of biological activities such as anti-inflammatory, antiproliferative, antihistaminic, anti-HIV, hypnotic, anaesthetic, antifungal, anthelmintic and antiviral agents as well as CNS stimulants. Therefore researchers have synthesized these condensed heterocycles through different complex pathways as target structures for biological studies. This review focuses on the various strategies followed for the convenient synthesis of thiazolidinone based heterocyclic derivatives. The steps included condensation followed by cyclization of Schiff's bases, either in a step-wise manner or in one pot under different conditions. Mercaptoacetic acid, thiolactic acid, chloroacetyl chloride, potassium thiocyanate, ethylchloro acetate and ammonium thiocyanate are the most common reagents used for the synthesis thiazolidinone appended on different heterocyclic skeletons.

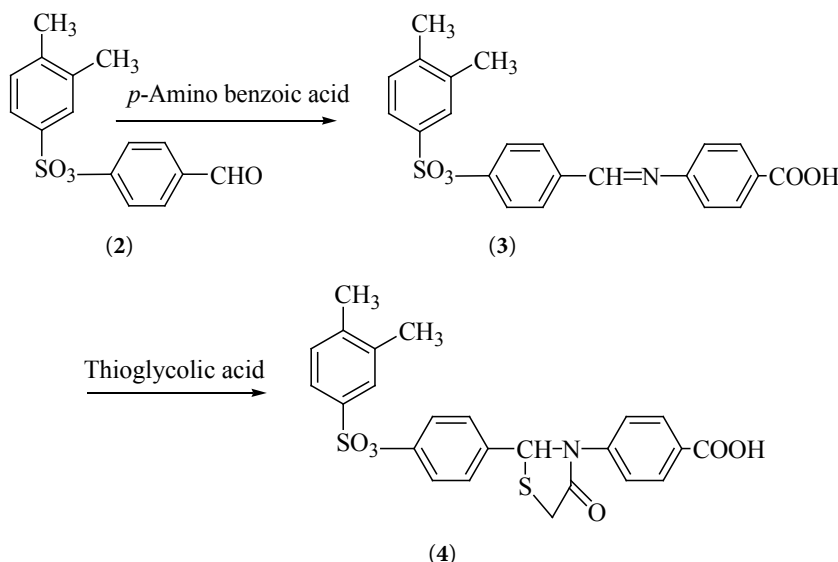
Keywords: Schiff's bases; Mercaptoacetic acid; Condensation; Heterocycles

Thiazolidinones

Thiazolidinones and their derivatives display a large variety of activities such as antibiotic, diuretic, organoleptic, tuberculostatic, antileukaemic and antiparasitical [1,2]. As far as literature is concerned, little is known about thiazolidinones and their bioactivities. Thiazolidinones (1) are classified as doubly unsaturated five membered heterocyclic compounds contain one nitrogen, one sulphur and three carbon atoms including a carbonyl group.



Moawad et al. reported that reaction of Schiff base (3) with mercaptoacetic acid in dry benzene afforded thiazolidinone (4). Schiff base (3) was obtained by the condensation of, 3,4-xylene sulphonate ester of *p*-hydroxy benzaldehyde (2) with *p*-aminobenzoic acid [3].



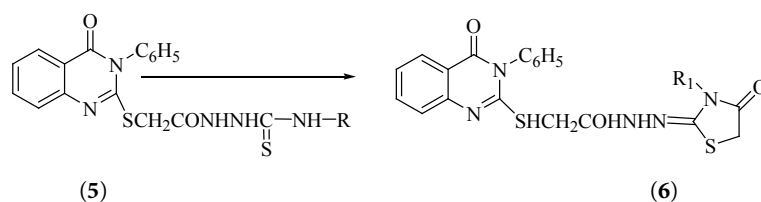
Gursoy et al. prepared new compounds (5) and (6) incorporating the quinazoline moiety [4].

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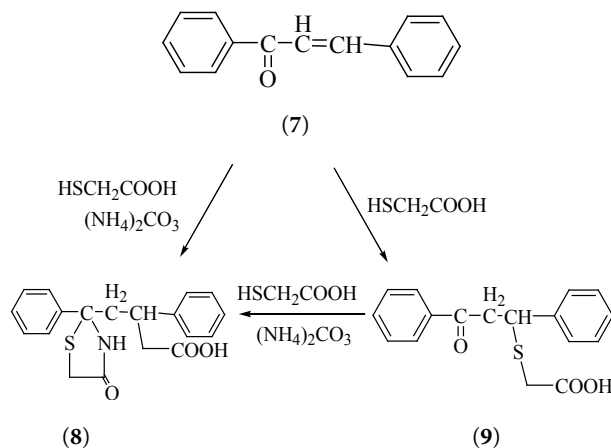
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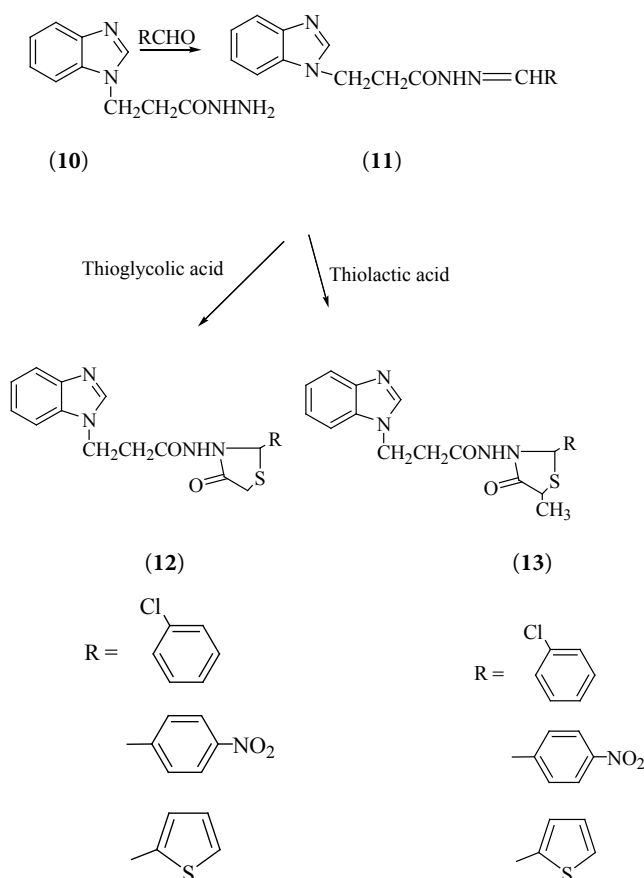
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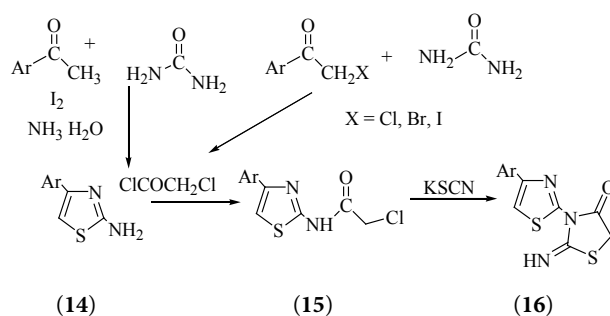
Ansari et al. synthesized 4-thiazolidinones (8, 9) starting from chalcone (7), by two different routes as shown in the following scheme [5].



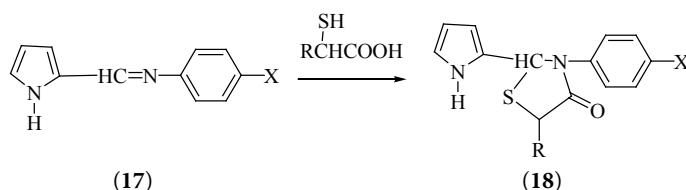
El-masry et al. reported that condensation of compound (10) with aromatic and heterocyclic aldehydes in absolute ethanol afforded the corresponding Schiff's bases (11). The cyclocondensation of some substituted Schiff's bases with thioglycolic acid and thiolactic acid afforded the corresponding thiazolidinone (12) and (13) [6].



Anthonsen et al. synthesized 2-imino-3-(4-arylthiazol-2-yl)-thiazolidin-4-ones (16) via the key intermediates, 2-amino-4-arylthiazoles (14) which were reacted with chloroacetyl chloride to produce the corresponding 2-chloro-acetamido-4-arylthiazoles (15). The latter was treated with potassium thiocyanate in refluxing acetone to afford the product (16) [7].

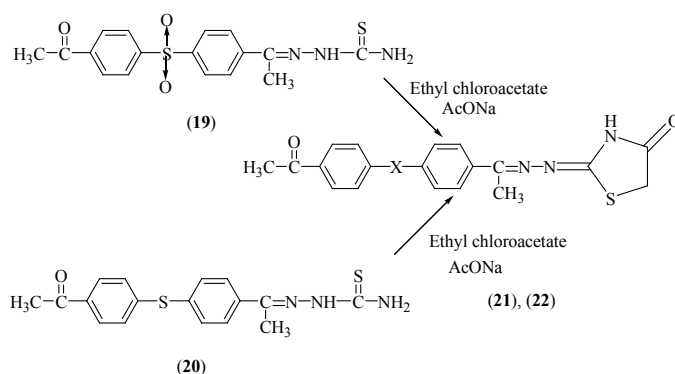


Ocal et al. synthesized substituted 4-thiazolidinones (18) by the reaction of mercaptoacids aldimines (19) which were obtained by the condensation of pyrrole-2-carbaldehyde with different aromatic amines [8].

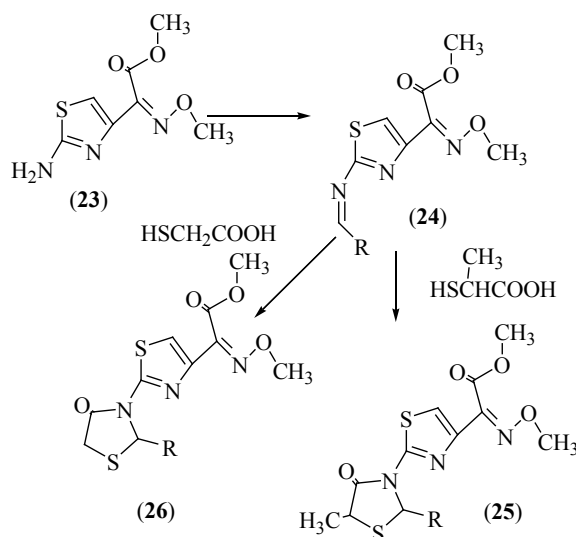


$R = H, CH_3$

Abbadly et al. obtained 4-acetylthiosemicarbazone-4-acetyl diphenyl sulphone (19) and 4-acetylthiosemicarbazone-4-acetyldiphenyl sulphide (20) which on further reaction with ethylchloroacetate in the presence of fused $AcONa$ gave 4-(4'-thiazolidinone-2''-acetylazino)-4'-acetyldiphenyl sulphone (21) and 4-(4'-thiazolidinone-2''-acetylazino)-4'-acetyldiphenyl sulphide (22), respectively [9].

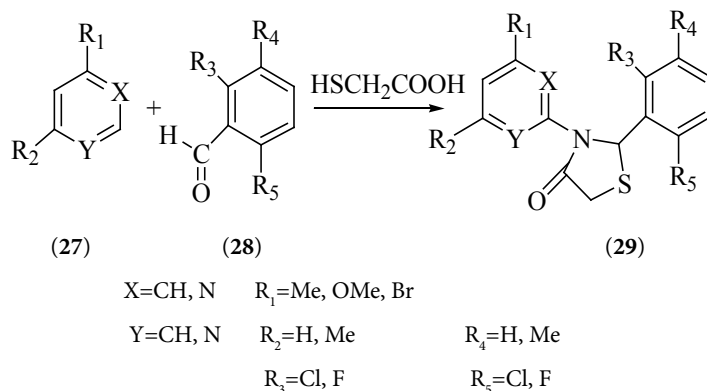


Parekh et al. prepared that substituted Schiff's bases (24) by the treatment of 2-amino-4-(α -methoxyiminocarbomethoxymethyl)-thiazole (23) with different aromatic mercaptopropionic acid in dry benzene furnished desired thiazolidinones of type (25) and (26) respectively. The products were evaluated for their *in vitro* growth inhibiting activity against several microbes. Some of them showed significant anti-tubercular and antifungal activity [10].

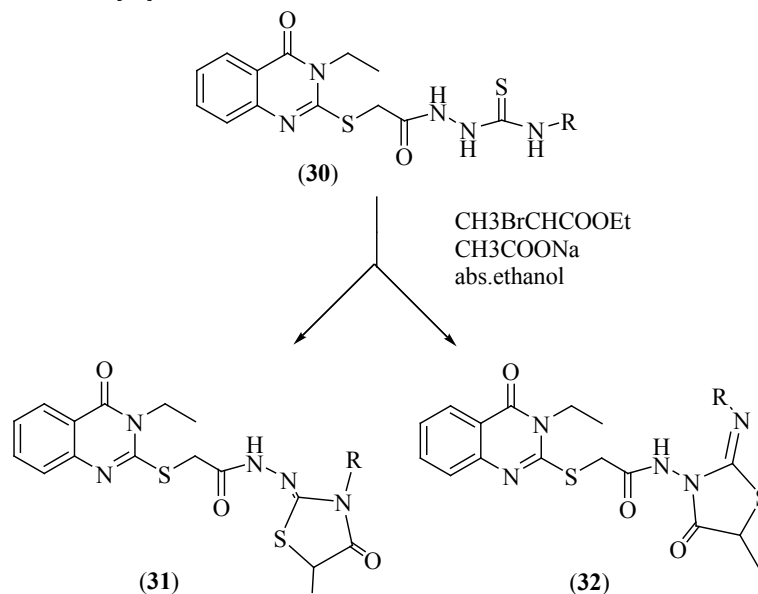


$R = Ar$

Monforte et al. reported the synthesis of 2,3-diaryl-1,3-thiazolidin-4-ones (29) by reacting an aromatic aldehyde (28) with an equimolar amount of (hetero) aromatic amine (27) in the presence of an excess of mercaptoacetic acid. The microwave-irradiation dramatically shortened the reaction times, affording the desired products in high yields [11].

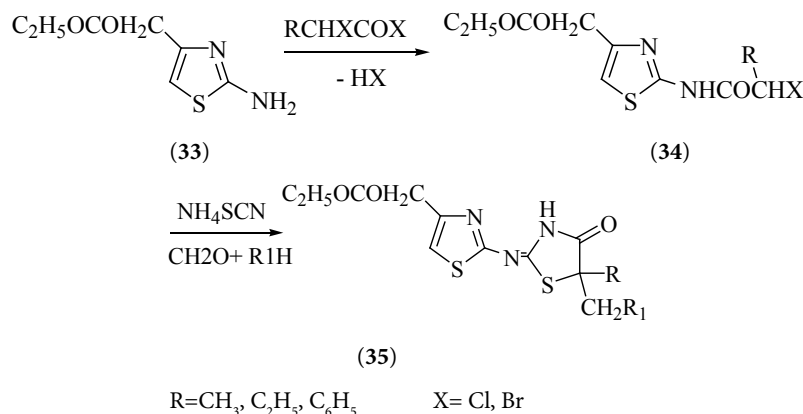


Terzioglu et al. synthesized the target compound, 2-(3-ethyl-4(3*H*)-quinazolinone-2-ylmercaptoacetylhydrazono)-3-alkyl/aryl-5-methyl-4-thiazolidinones (31) and 2-arylimino-3-(3-ethyl-4(3*H*)-quinazolinone-2-ylmercaptoacetylamino)-5-methyl-4-thiazolidinones (32) by the cyclization of 3-ethyl-4(3*H*)-quinazolinone-2-ylmercaptoacetyl-4-alkyl/arylthiosemicarbazides (30) with ethyl 2-bromopropionate in presence of anhydrous sodium acetate in absolute ethanol [12].

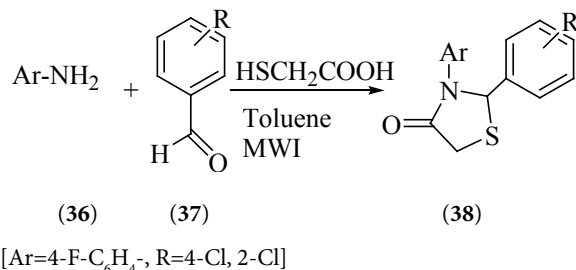


R = CH₃, C₆H₁₁

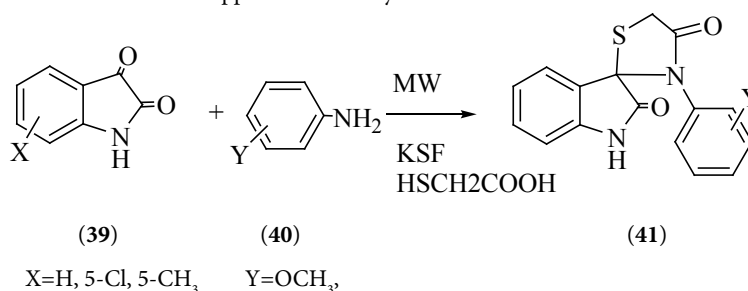
Altintas et al. synthesized by refluxing of 4-carboxymethyl-2-[(α-chloropropionyl/ α-bromobutyryl/ α-chloro-(phenyl)acetyl)amino]thiazoles (33) with ammonium thiocyanate to obtain 4-thiazolidinone (34). 5-(*N,N*-disubstitutedaminomethyl-2-[(4-carboxymethylthiazol-2-yl)imino] 4-thiazolidinones (34) was evaluated as antibacterial and antifungal activities [13].



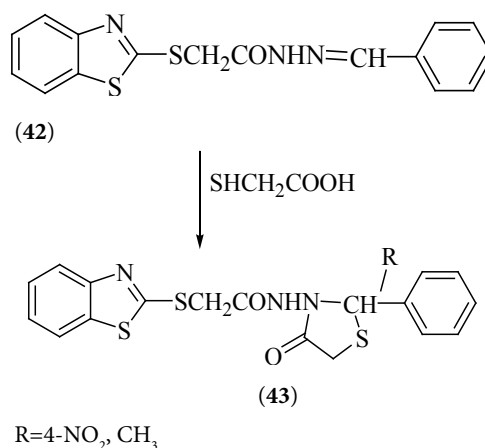
Sriram et al. reported that the synthesis of the 2,3-diaryl-1, 3-thiazolidin-4-ones (38) was accomplished by reacting substituted benzaldehyde (37) with equimolar amount of an appropriate substituted aromatic amine (36) in the presence of an excess of mercaptoacetic acid in toluene under microwave irradiation. Unlike the conventional methods (reaction time 48 h and yields of 30-70%), microwave-assisted reactions were very facile (6-8 min.) and provided very good yields (64-82%) [14].



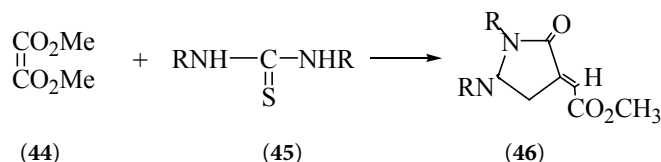
Arya et al. prepared *spiro*[indole-thiazolidines] (41) by the multicomponent condensation between indole-2,3-dione (39), amines (40) and mercaptoacetic acid using montmorillonite KSF as solid support in 85-90% yield in 4-5 min under MW reaction conditions [15].



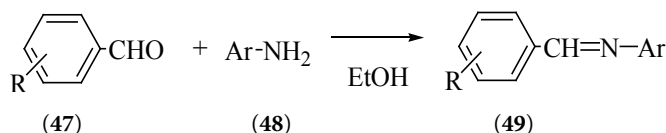
Desai et al. synthesized 4-thiazolidinones (43) in a good yields from the heterocyclization reaction of 2-(benzothiazol-2-ylthio)-N'-benzylideneacetohydrazide (42) with mercaptoacetic acid in DMF in the presence of catalytic amount of anhydrous ZnCl₂ under microwave irradiation and compared with conventional methods [16].

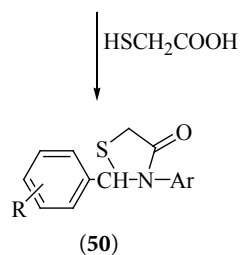


Nagarajan et al. synthesized *E*-thiazolidinone (46) by the cyclization of acetylene dicarboxylic ester (44) with thiocarbamoyl derivatives (45) [17].



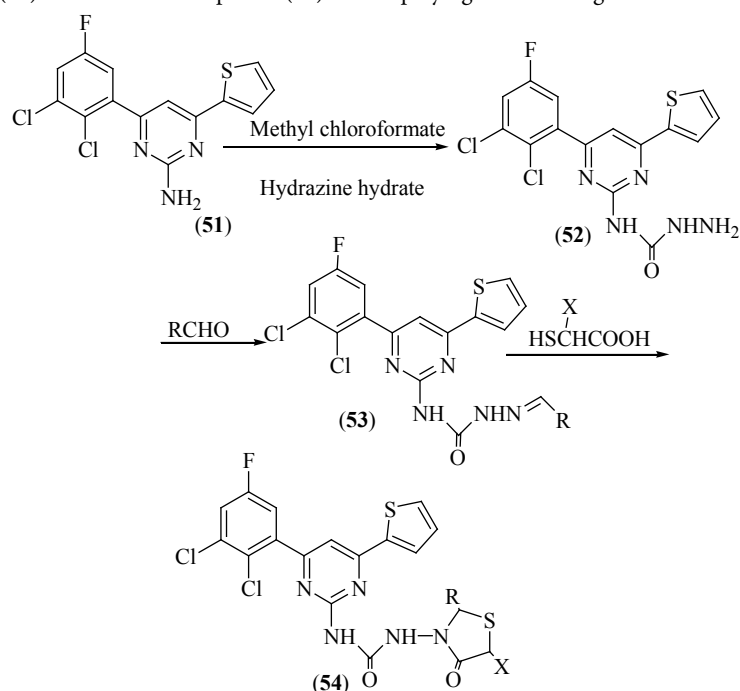
Sahu et al. reported that condensation of substituted benzaldehydes (47) with primary aryl amines (48) gave a series of Schiff bases (49) which, on reaction with mercaptoacetic acid, resulted in the formation of the corresponding 4-thiazolidinones (50) [18].





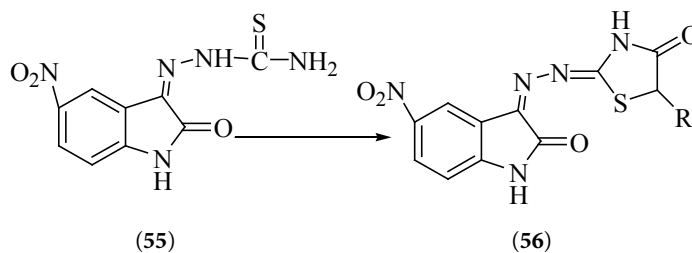
Ar=4-NO₂, Phenyl, 4-Cl and Naphthyl R=2-OH, 4-N(CH₃)₂, 4-NO₂, 4-Cl and 4-OCH₃

Desai and Shah synthesized of 2-(substituted phenyl)-3-[4-(2,4-dichloro-5-fluorophenyl)-6-(2-thienyl)pyrimidine-2-yl-ureido] 5H/ methyl/ carboxymethyl-4-thiazolidinones (54). It starts with compound (51) and employing the following reaction scheme [19].

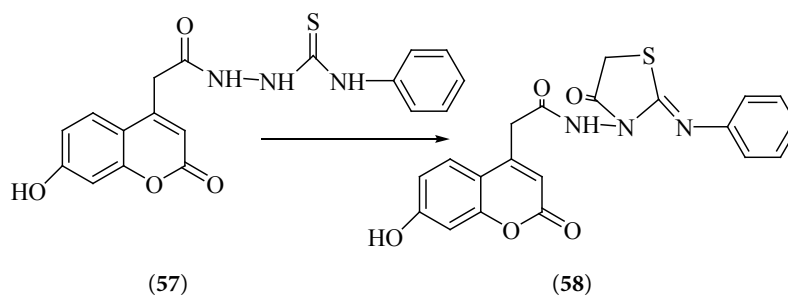


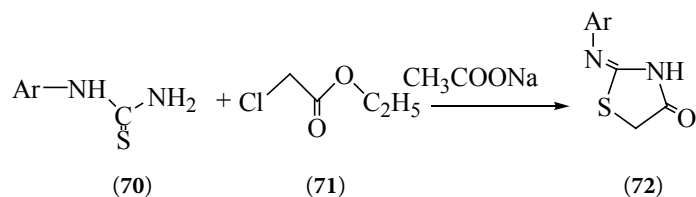
R=aryl; X=H, CH₃, CH₂COOH

Terioglul et al. synthesized series of 5-nitro-3-[(5-nonsubstituted/methyl-4-thiazolidinone-2-ylidene) hydrazono]-1H-2-indolinones (56) by cyclization of 5-nitro-1H-indole-2,3-dione-3-thiosemicarbazone (55) with ethyl bromoacetate or ethyl 2-bromopropionate [20].

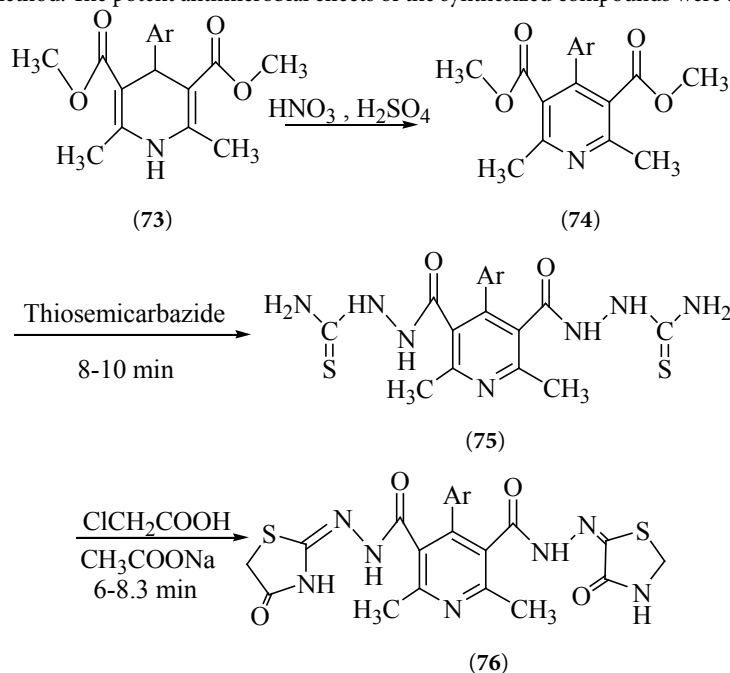


Cacic et al. reported the cyclization of thiosemicarbazide (57) with chloroacetylchloride in chloroform which afforded thiazolidinone derivative (58) [21].



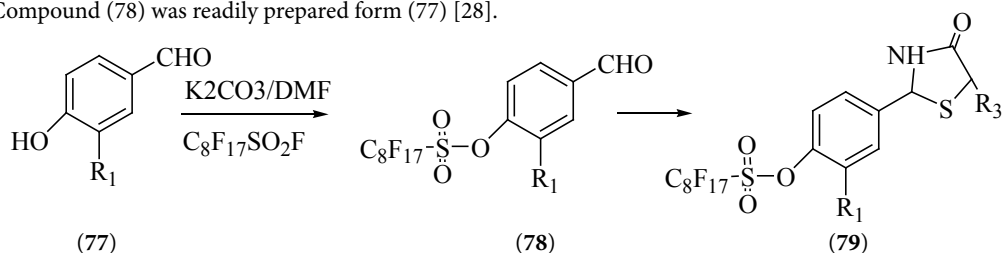


Ameta et al. reported a fast and facile procedure for the synthesis of pyridothiazolidinone (76) starting from dihydropyridine (73). Oxidation of (73) with nitrating mixture ($\text{HNO}_3/\text{H}_2\text{SO}_4$) produced the anticipated 2,6-dimethylpyridine derivatives (74), which were subsequently condensed thiosemicarbazide in ethanol to produce the key intermediate 2,2-[4-(4-substitutedphenyl)-2,6-dimethylpyridine-3,5-diyl] dicarbonyldihydrazine carbothiomides (75). Compound (75) finally provided (76) on reacting with ClCH_2COOH and CH_3COONa . All the reaction were carried by conventional as well as microwave method. The potent antimicrobial effects of the synthesized compounds were also investigated [27].



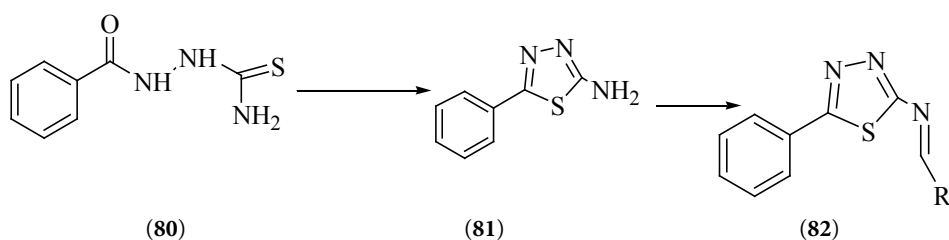
Ar=4-F-C₆H₄, 4-Cl-C₆H₄, 4-OCH₃-C₆H₄, 4-NO₂-C₆H₄, C₆H₅

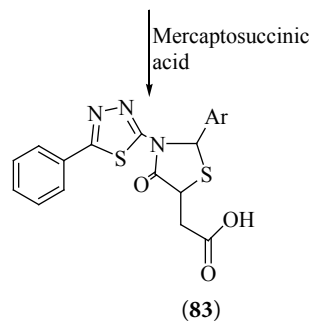
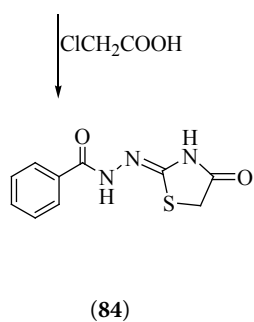
Yan et al. reported the construction of 4-thiazolidinone ring (79) by a three component reaction of fluorosulfonyl benzaldehydes (78), an amine and mercaptoacetic acid. Compound (78) was readily prepared from (77) [28].



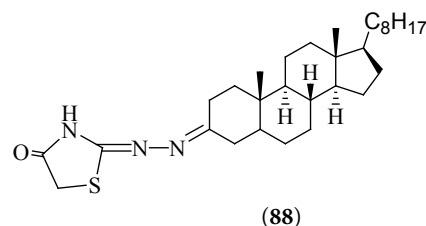
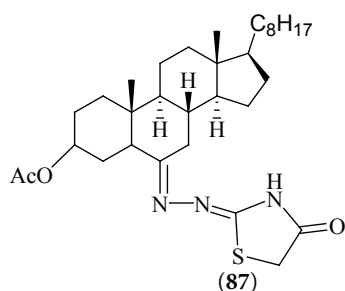
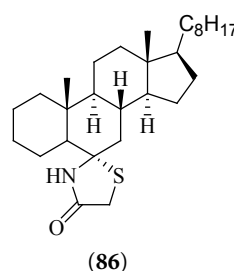
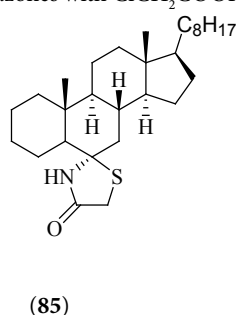
Talesara et al. also synthesized 2-amino-5-(4'-pyridyl)-1,3,4-thiaziazole (81) by cyclization of *iso* nicotinoyl thiosemicarbazide (80) with conc. sulphuric acid. When (81) was refluxed with various aldehydes. It gave the corresponding arylidene derivatives (82) which on further treatment with mercaptosuccinic acid furnished thiazolidinone derivative (83). Another thiazolidinone derivative 2-isonicotinoylhydrazido-1,3-thiazolidinone (84) was obtained by the successive treatment of isonicotinoylthiosemicarbazide (80) with chloroacetic acid in the presence of sodium acetate [29].

[Ar=4-OCH₃-C₆H₄, 3,4,5-OCH₃-C₆H₂, 3-NO₂-C₆H₄, 4-NO₂-C₆H₄, 4-(CH₃)₂NHC₆H₄, C₆H₅, C₄H₃O(2-furyl)].

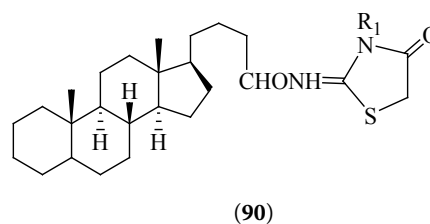
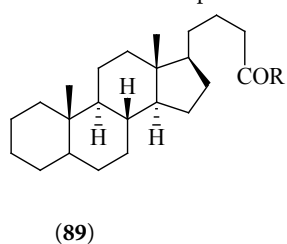




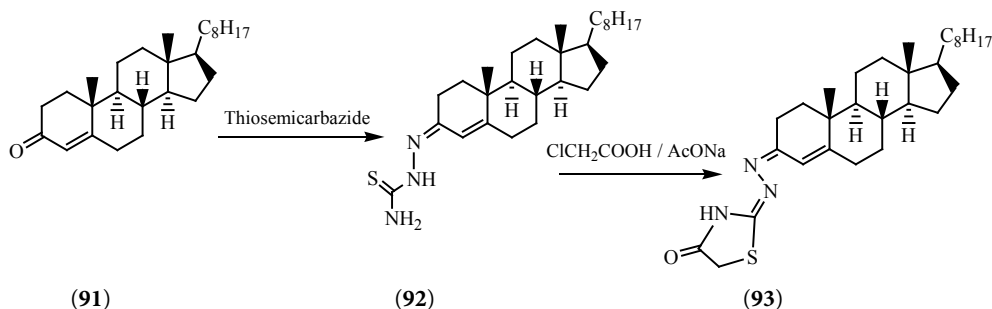
Literature has revealed that very few steroidal thiazolidinones have been prepared and studied so far. Shafullah and Ali reported the synthesis and mass spectral studies of the *spiro* thiazolidinones (85) and (86). Steroidal thiazolidinones of cholestane series (87) and (88) were also prepared by cyclizing the respective thiosemicarbazones with ClCH₂COOH [30].



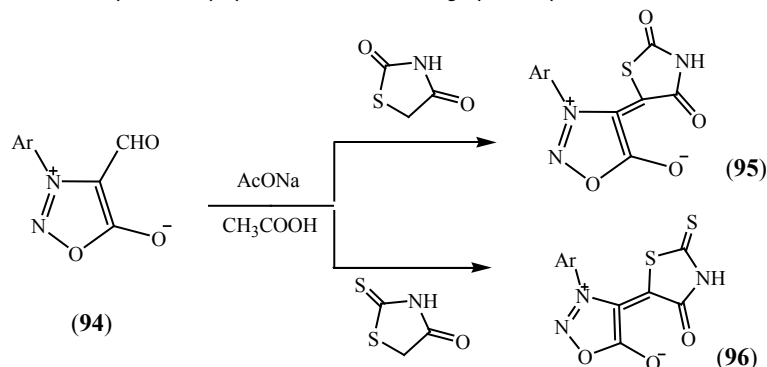
Siddiqui et al. prepared C-24 thiazolidinone derivatives (90) [R=H, OH, R¹=H] of bile acids from esters (89) [R²=OMe] in three steps involving hydrazinolysis with NH₂NH₂ to give (89) [R²=NHNH₂], reaction with potassium or ammonium thiocyanate to give (89) [R²=NHNHC(S)NH₂] and finally cyclocondensation with chloroacetic acid in presence of anhydrous sodium acetate and AcOH [31].



Shamsuzzaman and Siddiqui reported the synthesis of 3-Diazo(4'-thiazolidinone) cholest-4-ene (93) from cholest-4-en-3-one thiosemicarbazone (92) and chloroacetic acid in presence of sodium acetate. The compound (92) was obtained by reacting cholest-4-en-3-one (91) with thiosemicarbazide in presence of conc. HCl [32].



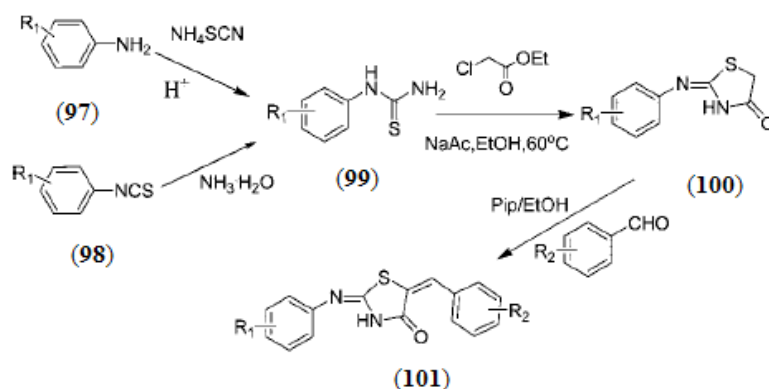
Shih et al. reported the synthesis of 5-(3-Arylsydnon-4-ylmethylene)thiazolidine-2,4-diones (95 a-d) and 5-(3-Arylsydnon-4-ylmethylene)-2-thioxothiazolidin-4-ones (96 a-d) from 3-aryl-4-formylsydnones (94 a-d) in high yields by the modified Knoevenagel condensation [33].



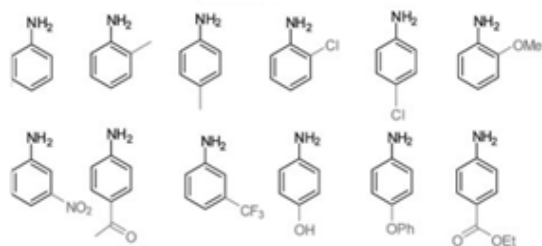
Ar=C₆H₅; b: Ar=p-CH₃C₆H₄; c: Ar=p-CH₃OC₆H₄; d: Ar=p-C₂H₅OC

All the synthesized thiazolidine derivatives (95a-d, 96a-d) were screened by paper-disc method to identify their antimicrobial activities against three bacteria viz. *Staphylococcus aureus*, *Proteus vulgaris* and *Escherichia coli*, and two fungal cultures viz. *Aspergillus niger* and *Penicillium citrinum*. The reference drugs were Norfloxacin and Griseofulvin, respectively. The screening data indicated that the tested sydnonyl-substituted thiazolidine derivatives exhibited no obvious antibacterial activity compared with the standard drug Norfloxacin. However, thiazolidine derivatives displayed significant antifungal activities against *Penicillium citrinum* and *Aspergillus niger*.

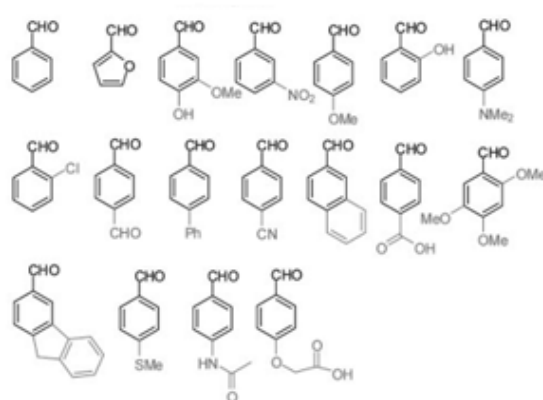
Zhou et al. reported the synthesis of library of 2-Arylimino-5-arylidene-thiazolidin-4-ones (101) by reacting 2-Arylimino-thiazolidin-4-ones (100) with different benzaldehydes. The compound (100) was obtained after reacting chloroethyl acetate with primary thiourea (99) in presence of sodium acetate. The compound (99) was obtained by the reaction of primary anilines (97) and NH₄SCN in 10 mL of 6 N HCl or from the reaction of corresponding thiourea (98) with NH₃·H₂O. These compounds selectively killed both non-small cell lung cancer cell line H460 and its paclitaxel-resistant variant H460taxR at an IC₅₀ between 0.21 and 2.93 μM while showing much less toxicity to normal human fibroblasts at concentrations up to 195 μM [34].



Anilines used



Aldehydes Used



Conclusion

In this article we have mentioned the different routes for the synthesis of thiazolidinone derivatives. The steps included condensation followed by cyclization or multi component reaction (MCR), either in a step-wise manner or in one pot has been achieved successfully to obtain the aforementioned class of heterocycles under different conditions. Most of the preparative methods included mercaptoacetic acid, thiolactic acid, chloroacetyl chloride, potassium thiocyanate, ethylchloro acetate and ammonium thiocyanate are the most common reagents for the synthesis of thiazolidinone appended on different heterocyclic skeletons. Also many series of substituted thiazolidinone -fused five membered heterocycles possessing N-, S- and O- have been constructed in potential yields and with conventional methods. Hence these protocols provide convenient strategies to annelate different heterocyclic nuclei with widespread bioactive thiazolidinone thereby extending the categories of heterocyclic systems. These strategies may also provide valuable information for the further design and development of more active biological agents through various modifications and derivatizations.

Acknowledgements

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