INTRODUCTION

SYNTHESIS OF ISOTHIOCYANATES

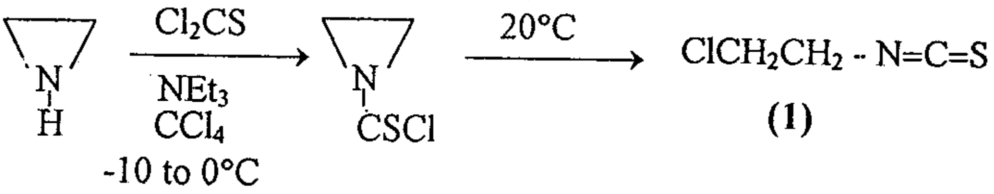
Isothiocyanates can be prepared by various procedures, the choice of which would depend on the target molecule. Heterocyclic isothiocyanates reported so far were obtained by methods in vogue for the synthesis of alkyl, aryl, and acyl isothiocyanates.(1,2)

A) From heterocyclic reagents with ring cleavage:-

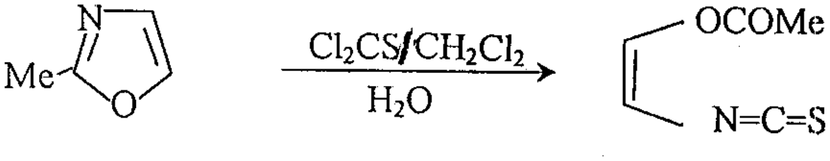
Some reagents brought about rupture of heterocycles leading to the formation of different isothiocyanates.

1) By the action of thiophosgene on heterocycles:-

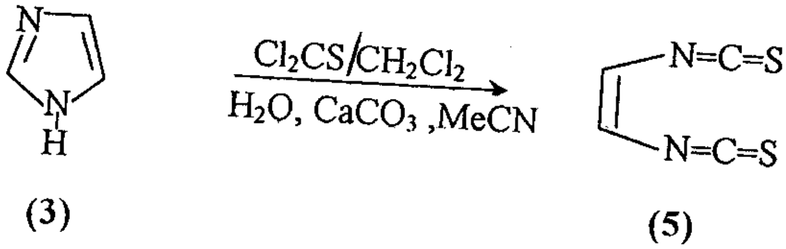
The condensation of aziridine with thiophosgene gave 2-chloroethyl isothiocyanate **(1)** in (45 - 50%) yields.(3)



The reaction of 2-methyloxazole **(2)** and imidazole **(3)** with thiophosgene underwent hydrolytic cleavage to the corresponding isothiocyanates **(4)**(4,5) and **(5)**(6) respectively.

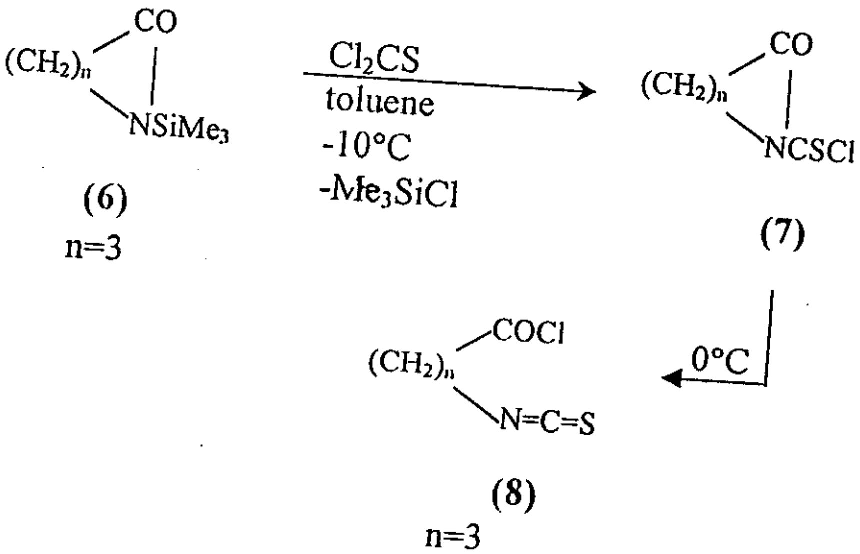


(2) (4)

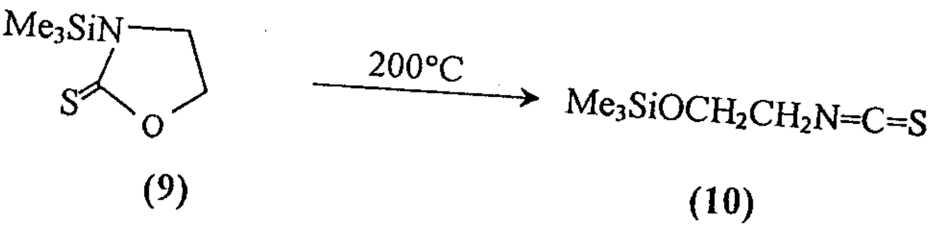


Benzoxazole and benzimidazole afforded similar products.(7)

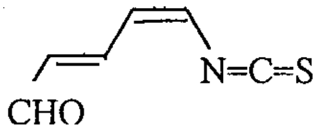
N-Trimethylsilyl lactams **(6)** reacted with thiophosgene to give the N-chlorothiocarbonyl derivatives **(7)** which underwent ring opening , affording ω-isothiocyanatocarboxylic acid chlorides **(8)** in good yield.(8)



It is noteworthy that on heating N-(trimethylsilyl) oxazolidine-2-thione **(9)** [(trimethylsilyl) oxy] ethyl isothiocyanate **(10)** was obtained in (80%) yield.(9)



On the other hand, the reaction of pyridine, quinoline and isoquinoline with thiophosgene led to the formation of isothiocyanates **(11)**(10) , **(12)**(11) and **(13)** (11) resbectively.



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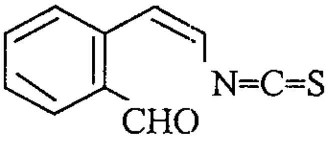

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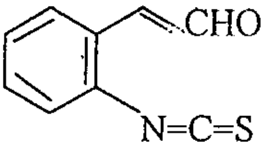
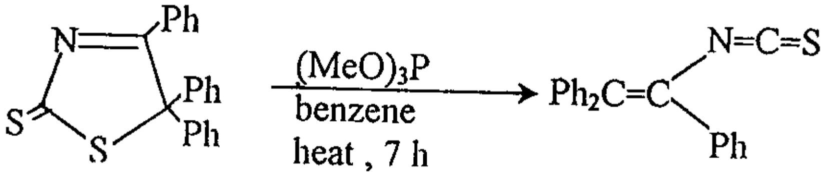


(13)

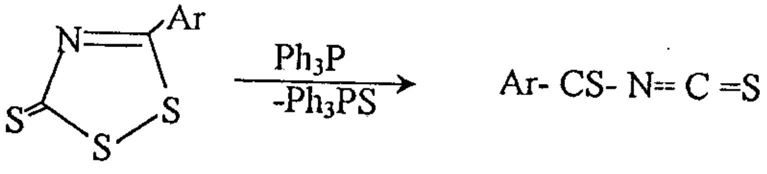
2) Phosphorus reagent aided conversion of sulfur compounds to isothiocyanates:

Photolysis or treatment of 3-thiazolin-2-thione **(14)** with trimethoxyphosphine gave isothiocyanate **(15)** in excellent yield.(12)



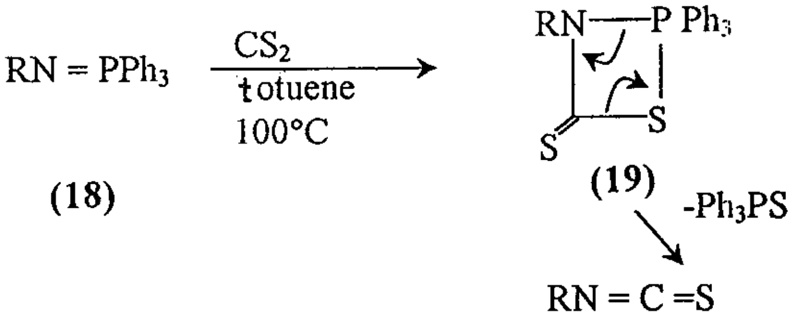
(14) **(15)**

Similarly, thioaroyl isothiocyanates(13) (**17**) have been prepared by the reaction of dithiazolinethione (**16**) with triphenylphosphine.



(16) **(17)**

On the other hand , the synthesis of isothiocyanate (**20**) by the cycloreversion of four-membered rings (**19**), obtained by the reaction of (N-alkyl-and N-arylimino) phosphoranes (**18**) with carbon disulfide.(14,15)



(20)

This reaction was successfully applied to the synthesis of 5-nitro-2-furylvinylene isothiocyanate, potentially useful as an antibacterial agent.(15)

B) From heterocyclic reagents without ring cleavage:-

Synthesis of some heterocyclic isothiocyanates involved the usual nucleophilic substitution with thiocyanic acid or its salts and manipulation of a primary amino group in the ring .

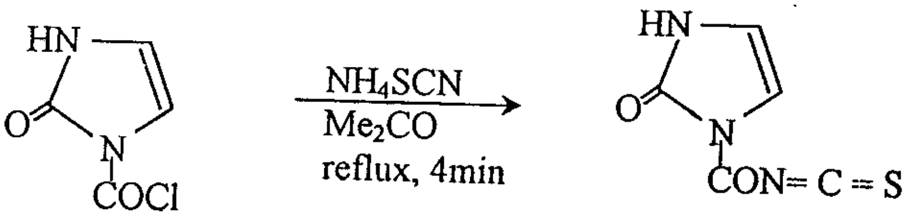
1) Substitution with thiocyanic acid or its salts :-

The condensation of carbonimidoyl dichlorides with potassium thiocyanate or potassium thioacetate in THF afforded the corresponding isothiocyanates(16) **(21)**.

$$RN = CCl_2 \xrightarrow[THF]{KSCN} RN = C = S$$

(21)

On the other hand , (2-oxoimidazolinyl) carbonyl isothiocyanate **(23)** have been prepared by reaction of 2-oxo-4-imidazoline-1-carbonyl chloride **(22)** with ammonium thiocyanate in boiling acetone.(17)



(22) **(23)**

2) *Use of dithiocarbamic acids and their salts or esters:-*

Primary amines, N-unsubstituted sulfonamides were also converted into the corresponding sulfonyl isothiocyanates(18) **(24)**.

A modified version by heating alkyl dithiocarbamates gave sulfonyl isothiocyanates in good yield(19) **(24)**.

$$RSO_2NH_2 \xrightarrow[2)\ COCl_2]{1)CS_2,\ KOH} RSO_2N=C=S$$

R= alkyl , aryl,
2-thienyl

(24)

A similar approach was used for the preparation of aryl isothiocyanates by heating the corresponding methyl dithiocarbamates in boiling toluene in the presence of sodium hydroxide.(20) This procedure seems to be simple and may have a wider application. Recently, desulfurization of methyl dithiocarbamates with mercury (**II**) oxide in DMF in the presence of ammonium hydroxide was enployed to prepare several aromatic diisothiocyanates (21) in good yields.

3) *Conversion of primary amines with sulfur reagents:-*

Primary amines reacted with thiophosgen(1,2,22) to give unstable thiocabamoyl chlorides which in turn furnished isothiocyanates **(25)**. Yields were moderate or good. This reaction should be carried out under controlled conditions using a protic solvents like chloroform , toluene etc .

$$RNH_2 + Cl_2CS \longrightarrow RNH-CS-Cl \longrightarrow RN=C=S$$

(25)

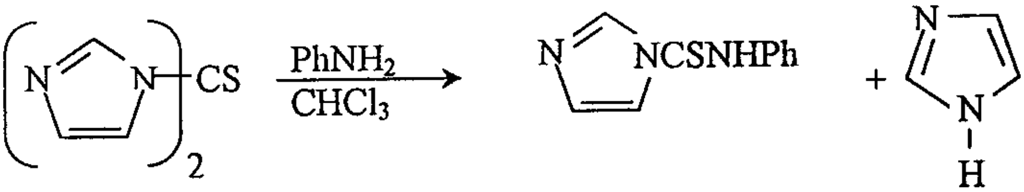
Thiophosgene should not be employed for amines with a vicinal reactive group because of cyclocondensation reactions .

This method was modified over the years and diethylthiocarbamoyl chloride,(23) bis (diethylthiocarbamoyl) sulfide or disulfide,(24) di-2-pyridyl thiocarbonate,(25,26) 1,1′-(thiocarbonyldioxy) dibenzotriazole,(27) and 1,1′-thiocarbonyl-2,2′- dipyridone(28) were introduced as the substitute of highly toxic thiophosgene.

4) Decomposition of thioureas:-

The pyrolysis of acylthioureas in o-dichlorbenzene afforded isothiocyanates as the major product.(27)

A convenient method using 1,1′-diimidazolylthiourea (**26**) in the preparation of isothiocyanates(28) (**29**).

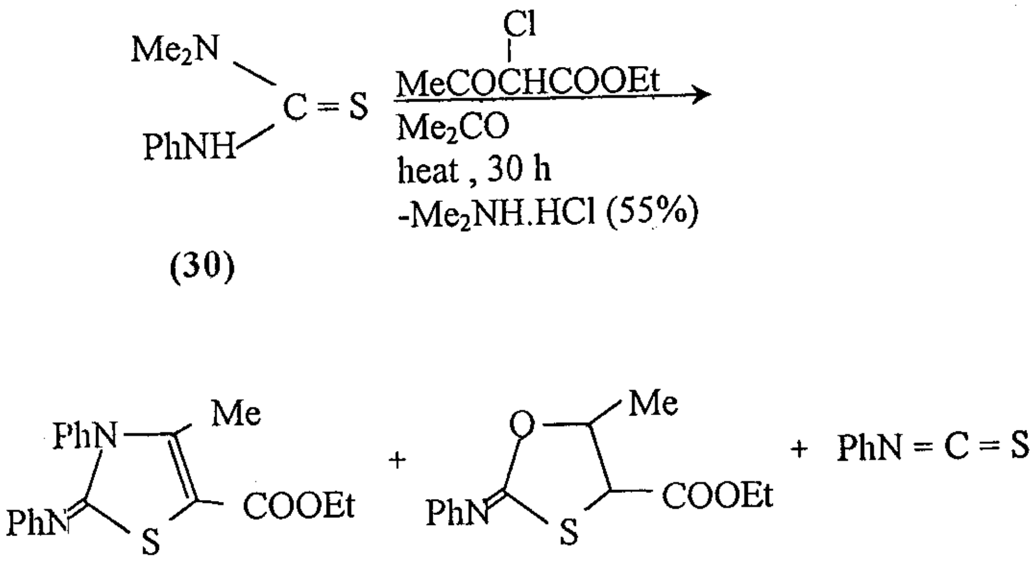


(26) **(27)** **(28)**

$$(27) \xrightleftharpoons{\text{Heat}} \text{PhN} = \text{C} = \text{S} \quad + (28)$$

(29)

On the other hand, ethyl α-chloroacetoacetate was used in the conversion of N,N-dialkyl-N`-arylthioureas (**30**) to the corresponding 3-aryl-2-(arylimino)-4-thiazolines (**31**) and 1,3-oxathioles (**32**) along with a small amount of aryl isothiocyanates(29) (**33**).



(30)

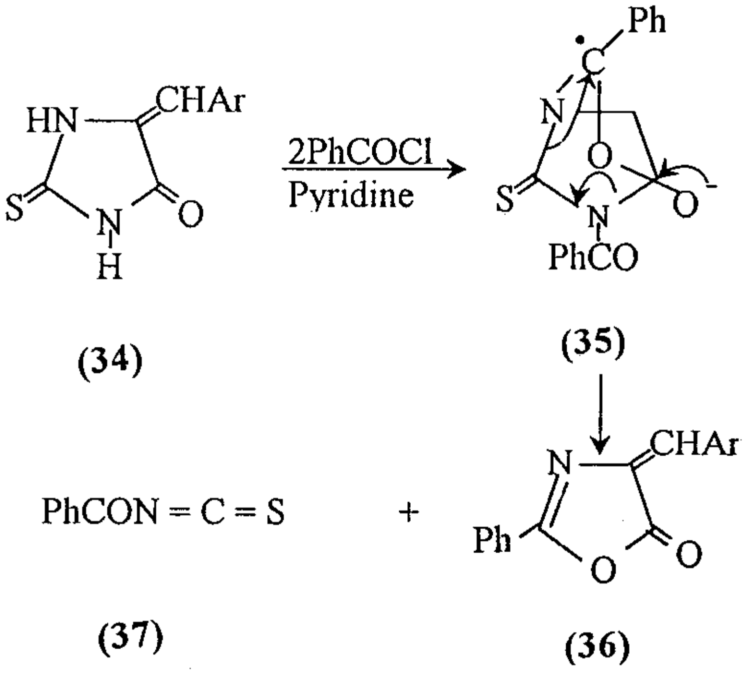
(31) **(32)** **(33)**

A better method of conversion of dithioureas to isocyanates by using 2,4-dichloro-5-nitropyrimidine was also reported.(30) Moreover treatment of metalated thioureas and ureas with carbon disulfide or 1,3-dithiolane-2-thione afforded isothiocyanates in good yields, as shown in the preparation of phenyl isothiocyanats(31)(**33**).

$$PhN^{-}(Li^{+})CSN^{-}(Li^{+})Ph \xrightarrow[\text{THF} \atop \text{heat , 3h}]{2CS_2} \underset{\textbf{(33)}}{2PhN=C=S} + Li_2CS$$

C) Miscellaneous syntheses of isothiocyanates.

Pyrolysis of some cyclic thioureas and thioamides or their reaction with suitable reagents gave isothiocyanates. For instance, the thiohydantion derivative (**34**) with excess of an aroyl chloride furnished aroyl isothiocyanate (**37**) and unsaturated azlactone (**36**) possibly through(32) (**35**). These methods are circuitous and therefore of limited application.



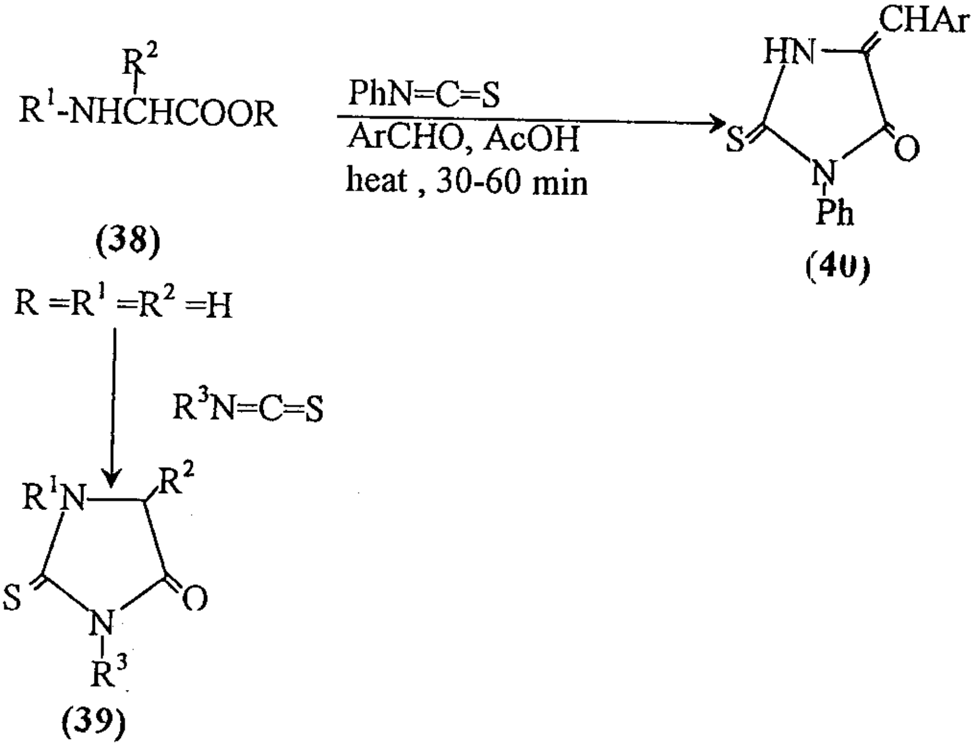
Ar = 3 . $O_2NC_6H_4$

REACTIONS OF ISOTHIOCYANATES

1) Reaction with amino acids and their derivatives:-

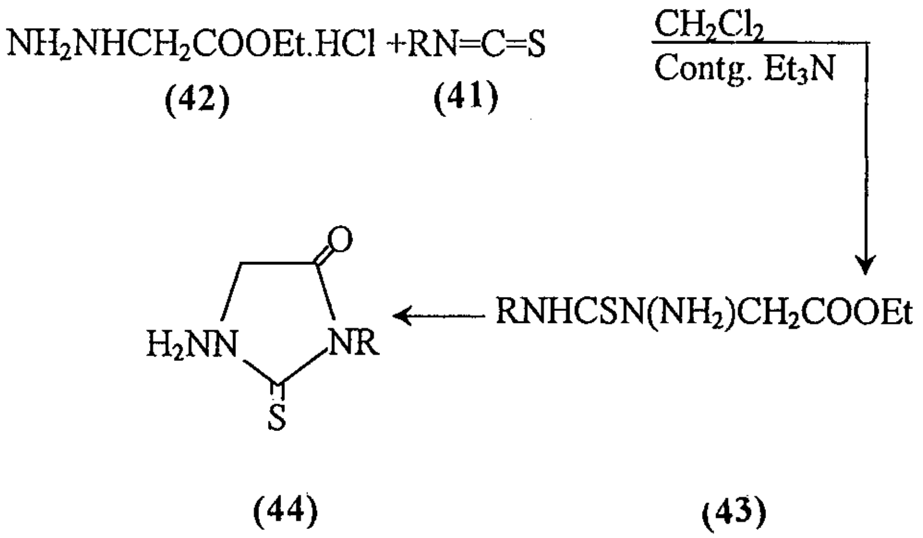
Cyclocondensation of α-amino acids or their esters (**38**) with isothiocyanates was used as a practical method for the synthesis of 2-thiohydantions (**39**) carring different substituents.(33-39) Yields were generally good .

The reaction of glycine with phenyl isothiocyanate in the presence of aromatic aldehydes afforded 5-(arylmethylene)-2-thiohydantions.(40)(**40**)



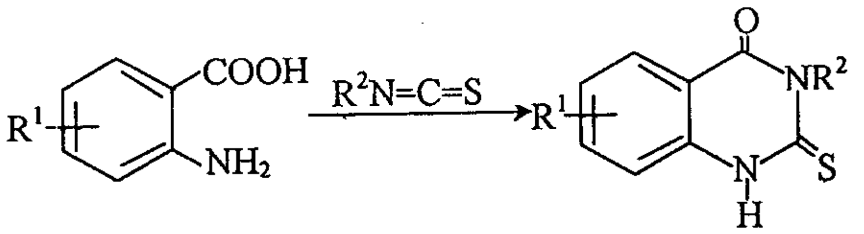
a: $R^1 = R^2 = H$, $R^3 = MeCOCH_2CH_2$.
b: $R^1 = NH_2$, $R^2 = H$, R^3 = alkyl , aryl
c: $R^1 = Me_2N$, $R^2 = CH_2COOMe$, R^3 = alkyl , aryl .
d : $R^1 = CH_2P(OH)_2O$, $R^2 = H$, $R^3 = H$, alkyl , aryl

Similarly, 1-aminothiohydantion derivatives(41)(**44**) were prepared in good yields by the reaction of isothiocyanate (**41**) with amino compound (**42**) in CH_2Cl_2, Et_3-N. The intermediates (**43**) which formed during the reaction and could be transformed into 1-amino thiohydantion derivatives.



R = Me , allyl , cyclohexyl , 4- ClC_6H_4 , CH_2ph , C_6H_5 , $COC_6H_4NO_2$-4 , 2-naphthyl .

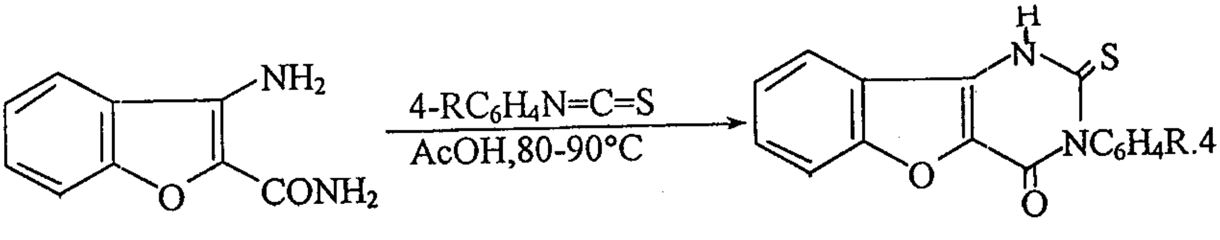
Condensation of anthranilic acids with isothiocyanates gaves 2-thio-4-quinazolones(1,42,43) (**45**).



(45)

R^1 =H ,R^2 = $MeCOCH_2CH_2$

The reaction of 3-aminobenzofuran-2-carboxamide (**46**) with isothiocyanate gave(44) (**47**).



(46)

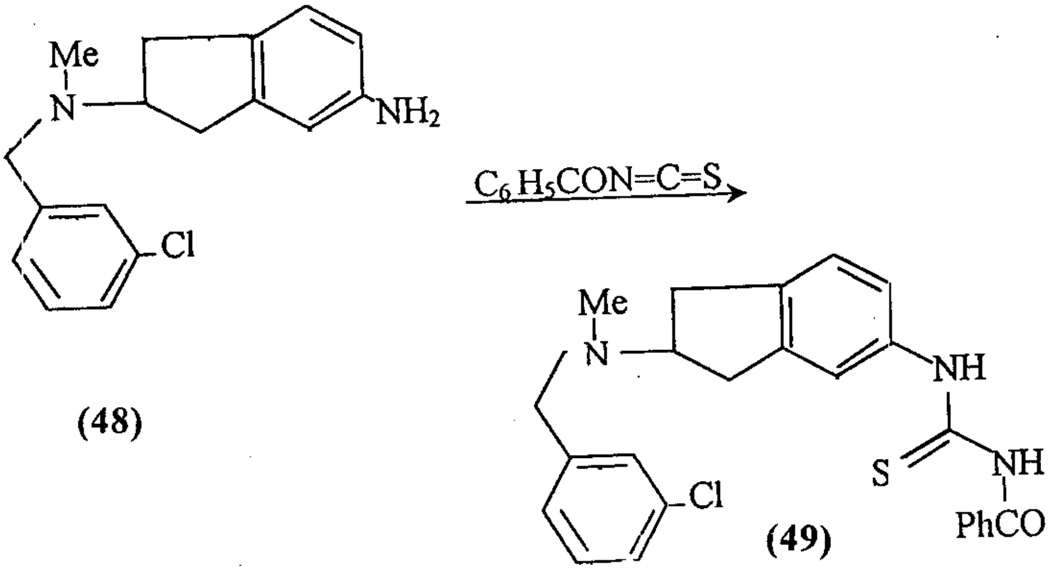
(47)

a : R = Me (42%) .

b: R = Cl (55%) .

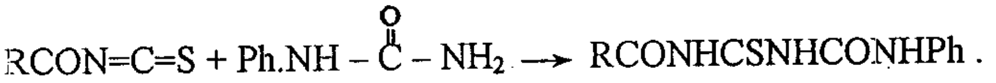
c: R = Br (58%) .

On the other hand, the reaction of 5- aminoindane (**48**) with benzoyl isothiocyanate gave(45)(**49**).



(48) **(49)**

The reaction of phenylurea(**50**)with isothiocyanate(**37**) gave^(46)^(**51**)



$$\underset{(37)}{\mathrm{RCON{=}C{=}S}} + \underset{(50)}{\mathrm{Ph.NH-\overset{O}{\overset{\|}{C}}-NH_2}} \longrightarrow \underset{(51)}{\mathrm{RCONHCSNHCONHPh}}.$$

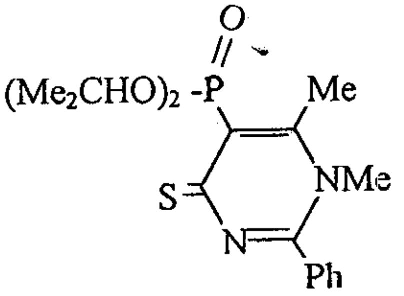
(37) **(50)** **(51)**

R = Ph , substituted Ph , 2-furyl ,
Ph – CH = CH –

Pyrimidine thione dervative **(53)** was prepared by the reaction of benzoyl isothiocyanate with (2-N-methylamino-1-propenyl) phosphonates^(47)^ **(52).**

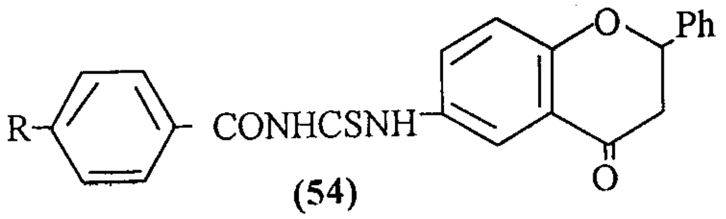
$$(Me_2CHO)_2P(O)CH{=}C(NHMe)\,Me \xrightarrow[\text{benzene 80 °C}]{PhCON{=}C{=}S}$$

(52)



(53)

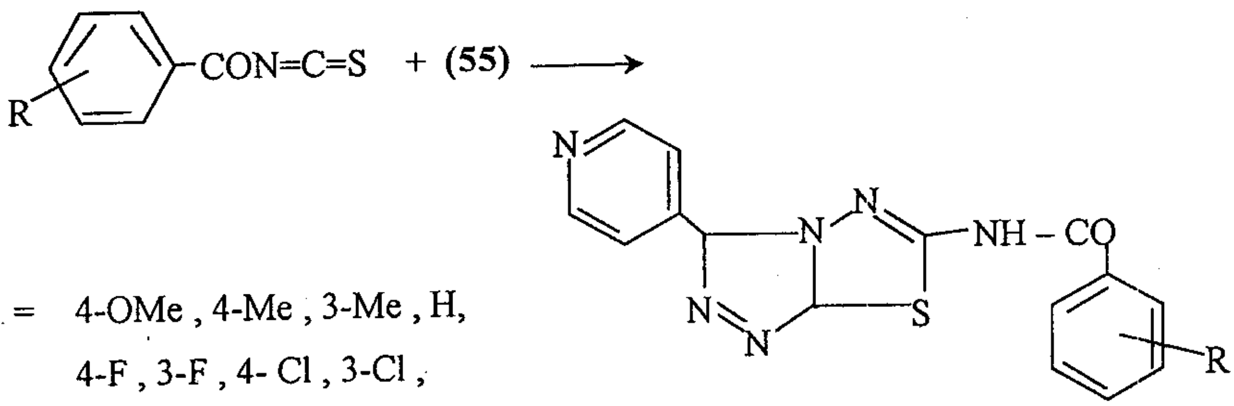
6-Aminoflavone was treated with aroylisothiocyanates in acetone at reflux temperature to yield N-aroyl-N'-(2-phenyl-4-oxo-H-[1]benzopyran-6-yl) thioureas(48)(**54**).



(54)

R = H , Cl , Me , MeO

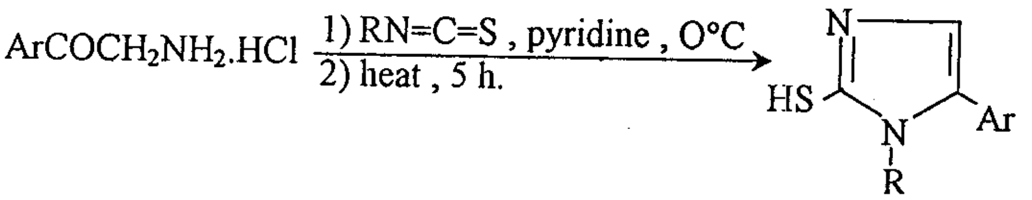
3-(-4'-Pyridyl)-4-amino-5-mercapto-1,2,4-triazole (**55**) reacted with aroyl isothiocyanates to yield twelve novel 3-(4'-pyridyl)-6-aroylamino-S-triazolo [3,4-b]-1,3,4-thiadizoles (49)(**56**).



R = 4-OMe , 4-Me , 3-Me , H,
4-F , 3-F , 4- Cl , 3-Cl ,
4-Br , 3- Br , 4-Iodo , 4-NO_2 .

(56)

Alkyl and aryl isothiocyanates reacted with amino ketone hydrochloride (**57**) in the presence of pyridine affording 2-mercaptoimidazoles(50) (**58**).



(57)

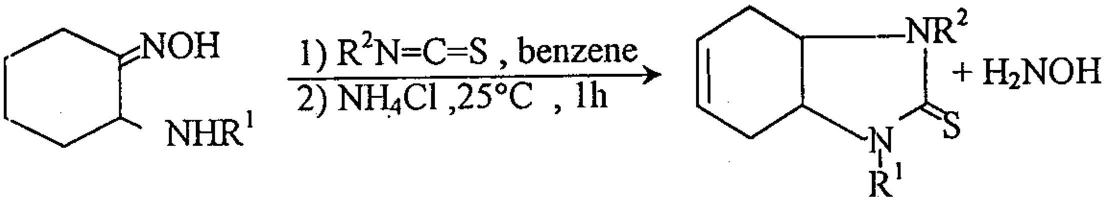
(58)

Ar=4- pyridyl

R = alkyl , aryl

The similar reaction of alkyl and aryl isothiocyanates with 2-amino - 2 - deoxyaldoses,(51) 1- deoxy -1- (methylamino) -D-lyxohexulose(52) and 2-(alkylamino)-2-deoxy-D-glucose(53,54) gave the corresponding imidazole derivatives.

On the other hand, o-aminocyclohexanone oximes (**59**) underwent cyclocondensation with isothiocyanates to give fused thioimidazolone (**60**) as a result of extrusion of hydroxylamine moiety during the cyclization step.(55)

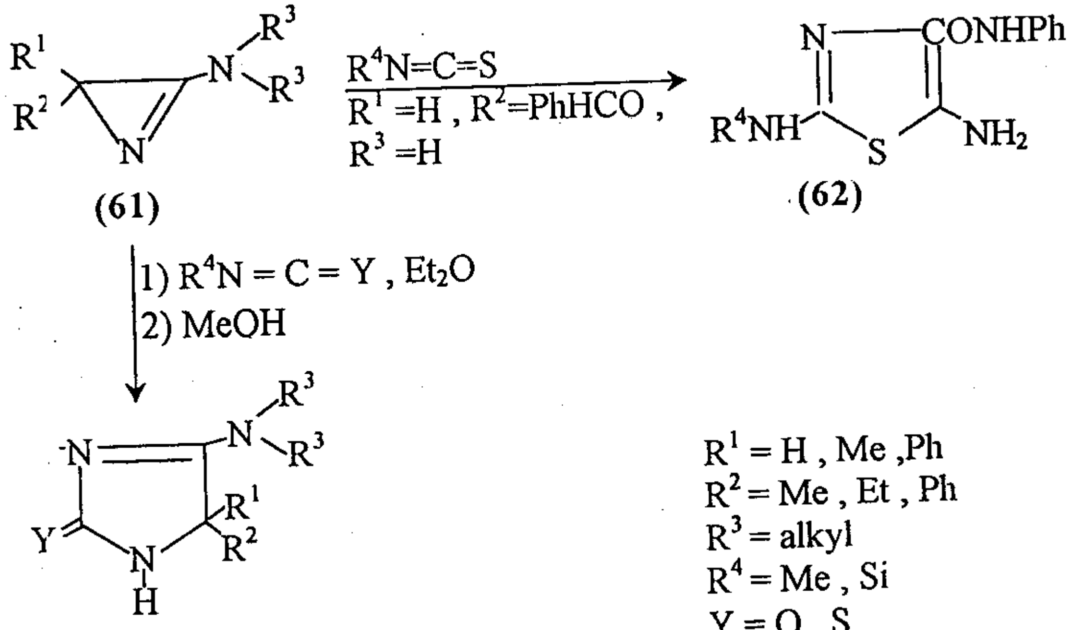


(59) (60)

R^1=H , alkyl , arakyl ;

R^2= alkyl , aryl .

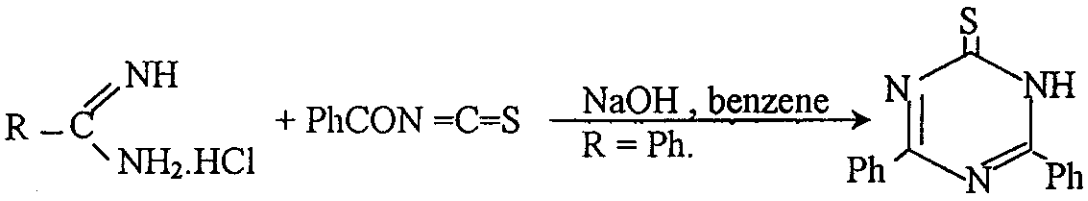
In the ring expansion of azirines (**61**) to five-membered heterocycles , the exocyclic amino group activated the ring nitrogen , thereby enanbling it to initiate the reaction which proceeded through a cycloadduct intermediate.(56,57)



(61) (62) (63)

R^1 = H , Me ,Ph
R^2 = Me , Et , Ph
R^3 = alkyl
R^4 = Me , Si
Y = O , S

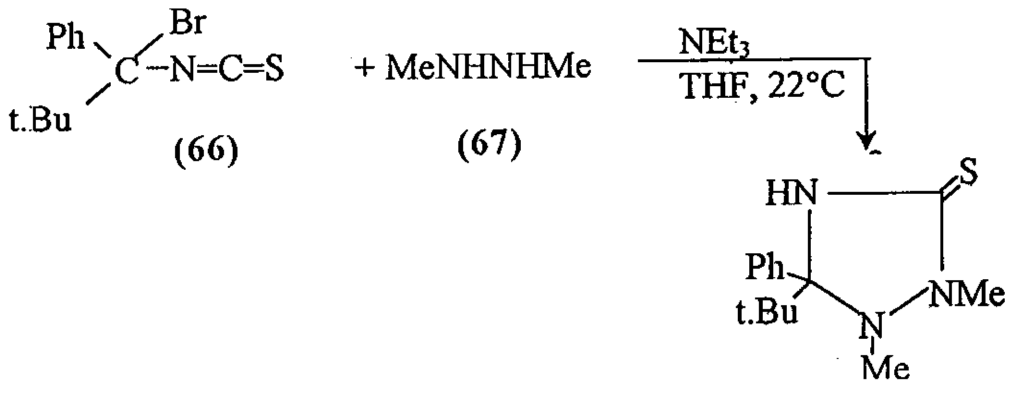
On the other hand , benzoyl isothiocyanate reacted with **(64)** to give triazine thiones(58-60) **(65)**.



(64) (65)

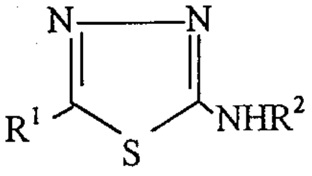
2) Reaction with hydrazines and hydrazides:-

The condensation of hydrazines with isothiocyanates led to different products depending on the nature of the reactants. Intereaction of the 1,3-bielectrophile **(66)** and N,N-dimethylhydrazine **(67)** furnished triazolidine derivative(61) **(68)**.



(68)

On the other hand , reaction of isothiocyanates with hydrazides furnished thioureas which gave thiadiazoles **(69)** under acidic conditions , whereas the use of dilute sodium hydroxide afforded triazoles(62-67) **(70)**.



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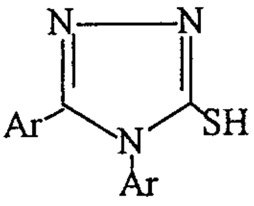
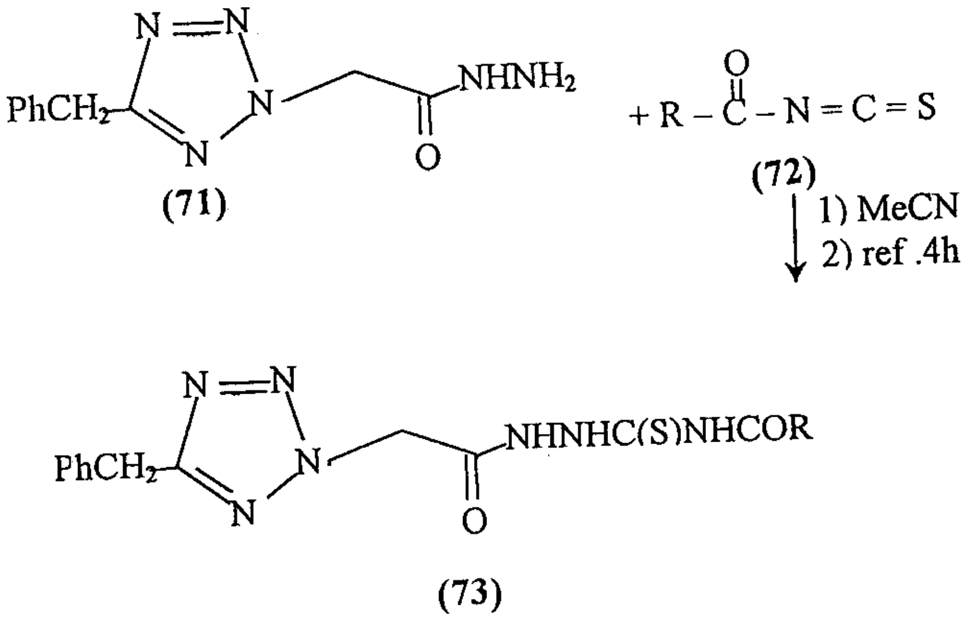
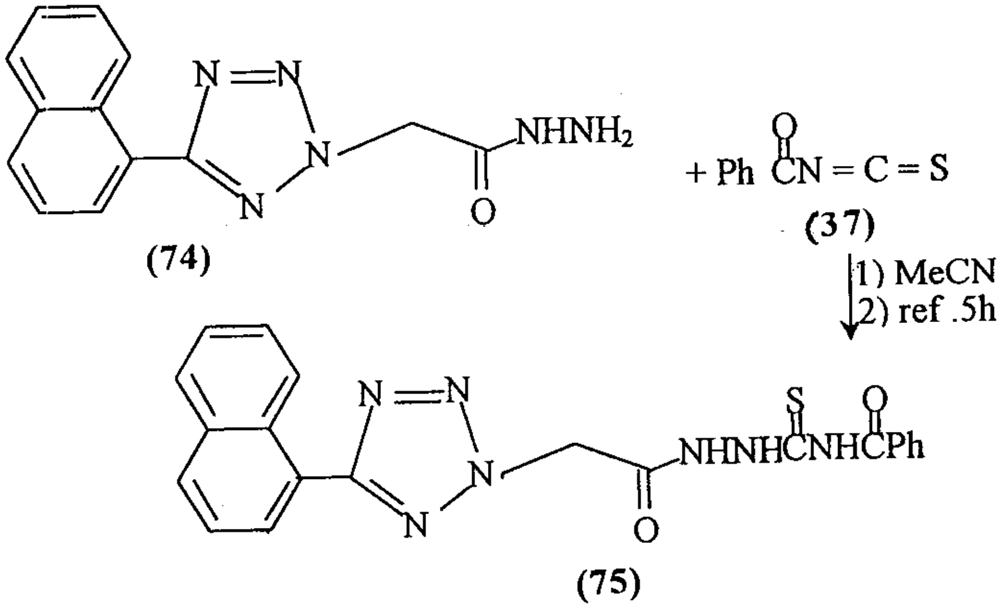


(70)

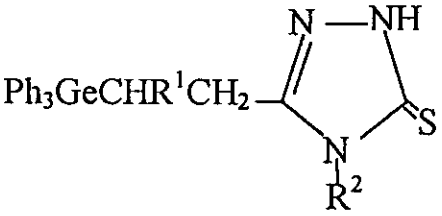
1-(5-Benzylteterazol-2-ylacetyl)- 4- aroylthiosemicarbazides (68) **(73)** were prepared by refluxing (5-benzyltetrazol-2-yl) acetyl hydrazine **(71)** with isothiocyanates **(72)** in MeCN for 4 h .



Similarly , 1-[5-(α-naphthyl)-2H-tetrazol-2-ylacetyl]-4-aroyl-thiosemicarbazides⁽⁶⁹⁾ **(75)** were prepared by refluxing 5-(α-naphthyl)-2H-tetrazol-2-ylacetyl hydrazine **(74)** with PhCONCS **(37)** in MeCN for 5h .



The reaction of R^2NCS (R^2 = Ph, p-MePh, o-MePh, p-ClPh) with Ph_3GeCHR^1 $CH_2CONHNH_2$ (R^1 = H,Ph) in $CHCl_3$ gave compound$^{(70)}$ (**76**).



(76)

1-(3,4,5 -Trimethoxybenzoyl) - 4 - aroylthiosemicarbazides (**78**) were prepared by condensation of (**77**) with isothiocyanates$^{(71)}$ (**37**)

$$3,4,5\text{-}(MeO)_3C_6H_2CONHNH_2 + R\overset{O}{\overset{\|}{C}}-N{=}C{=}S$$

(77) (37)

R = Ph , substituted
Ph , PhCH : CH ,
1-naphthylmethyl
, furyl

↓

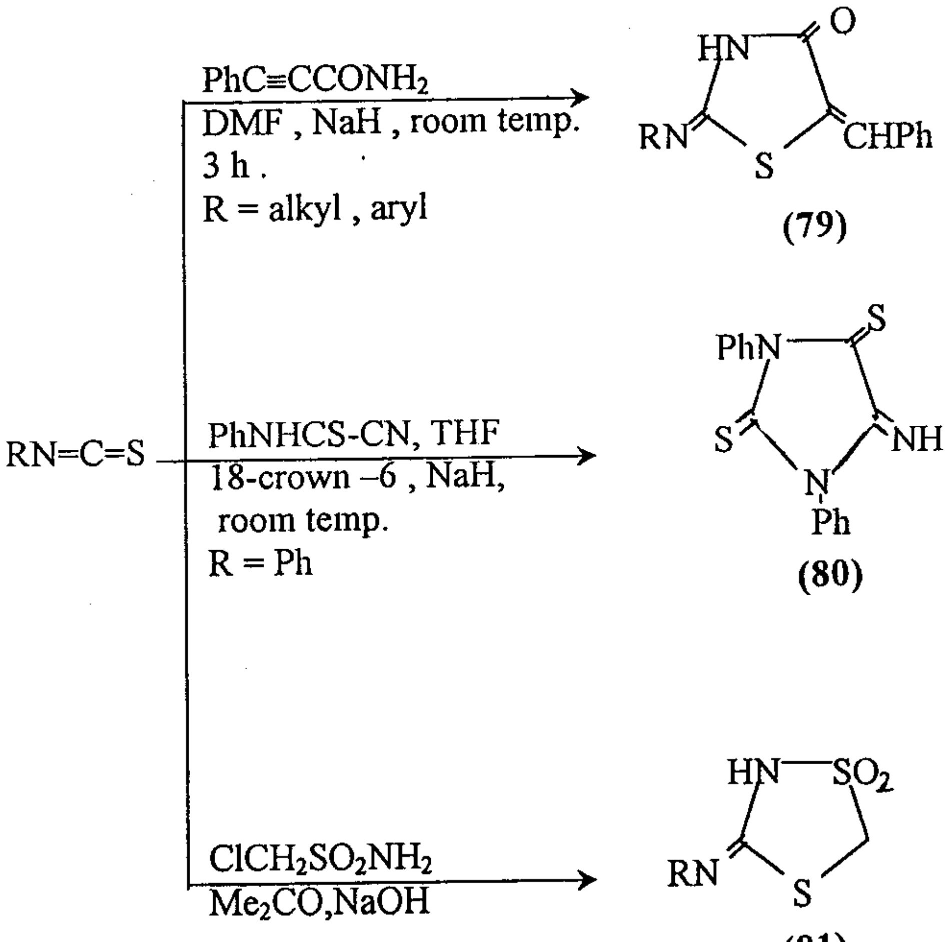
$3,4,5\text{-}(MeO)_3\ C_6H_2CONHNHC(S)NHCOR$

(78)

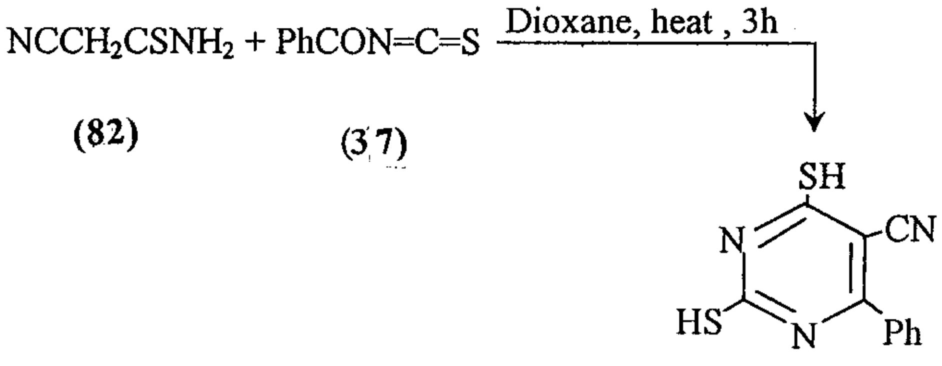
On the other hand , thiadiazolopyridazines were prepared via reaction of a pyridazinecarboxylic acid hydrazide with isothiocyanates.$^{(72)}$

3) Reaction with amides:

Azolidines (**79**)$^{(73,74)}$, (**80**)$^{(75-77)}$, and thiazolidine$^{(78)}$(**81**) were prepared by reaction of primary and secondary amides, thioamides and sulfoamides carring an electrophilic center within the molecule with isothiocyanates which in turn underwent ring closure to give heterocycles.

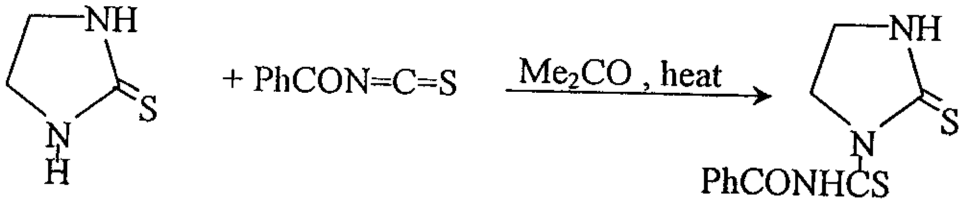


On the other hand, the condensation of cyanothioacetamide (**82**) with benzoyl isothiocyanate (**37**) led to the formation of the pyrimidinedithiol derivative(79) (**83**).



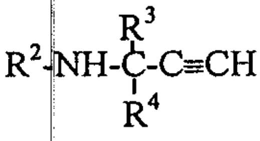
(82) (37) **(83)**

Imidazolidine-2- thione (**84**) reacted with benzoyl isothiocyanate (37) to give N- thiocarbamyl derivative(80) (**85**).



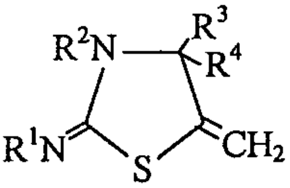
(84) (37) **(85)**

On the other hand, the addition of alkyl and aryl isothiocyanates to aminoalkyl acetylenes (**86**) led to the formation of 2- imino-5- methylene thiazolidines (**87**) through the involvement of the sulfur nucleophile. Compounds (**87**) exhibited hypotensive, antiinflammatory, and analgesic properties.(81)



$R^2\text{-}NH\text{-}\underset{R^4}{\overset{R^3}{C}}\text{-}C\equiv CH$

(86)



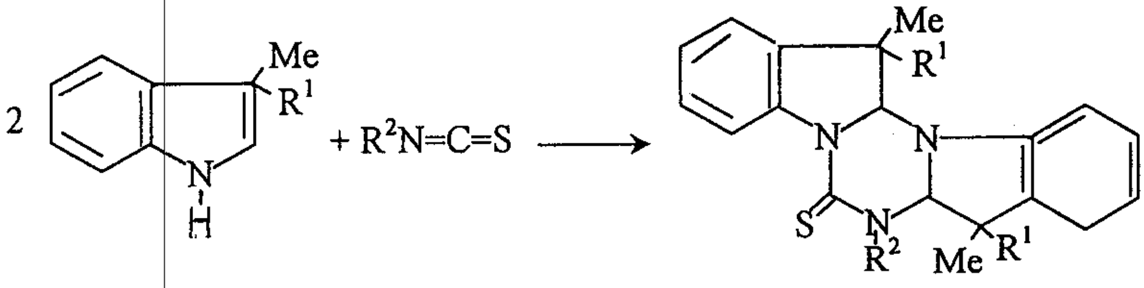
(87)

4) Cycloaddition reactions:-

The addition could take place at the C=S or at the C=N bond of the heteroallene moiety, and it could also lead to polymerization. The reaction of acyl or thioacyl isothiocyanates often led to the formation of 1,4-cycloadducts.(82)

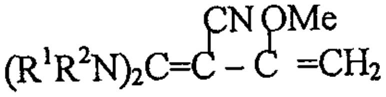
A) (2+2) Cycloaddition:-

The reacion of isothiocyanates with some imines furnished a six-membered triazine ring(1,22) as shown in the synthesis of **(89)** by the interaction of alkyl isothiocyanates and 3H- indoles(83) **(88)**.



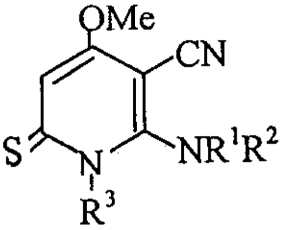
(88) **(89)**

The presence of a methoxy group at C=C bond of **(90)** enabled it to form an adduct **(91)**, possibly via cyclo addition, which underwent thermal cyclization to 5-cyano-6-aminopyridine-2-thiones **(92)** in good yields.(84)

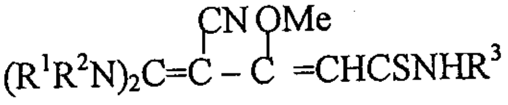


$(R^1R^2N)_2C=\overset{CN}{C}-\overset{OMe}{C}=CH_2$

(90)



(92)

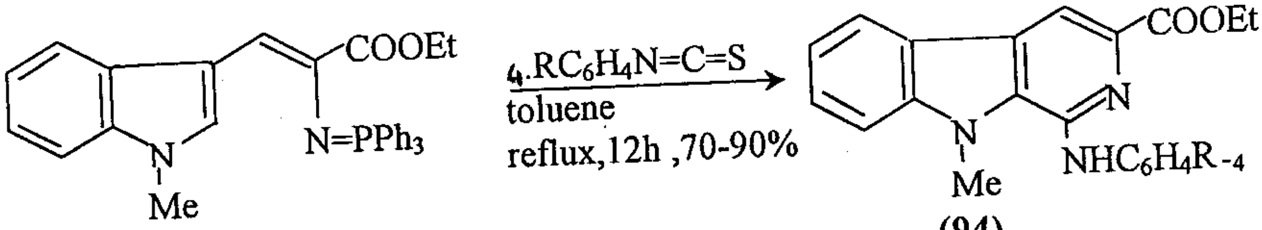


$(R^1R^2N)_2C=\overset{CN}{C}-\overset{OMe}{C}=CHCSNHR^3$

(91)

R^1R^2N = heterocyclic

On the other hand, (**93**) was converted into β-carboline (**94**) by heating with aryl isothiocyanates apparently via the carbodiimide intermediate.(85)

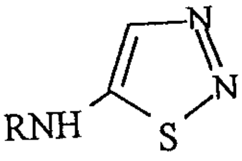


(93) **(94)**

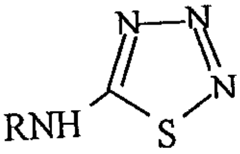
R = H , Me , MeO,Cl

B) [3+2]Cycloaddition:-

Diazomethane(86,87) and hydrazoic acid(86) added to isothiocyanates, furnishing thiadiazoles **(95)** and thiatriazoles **(96)**, respectively.

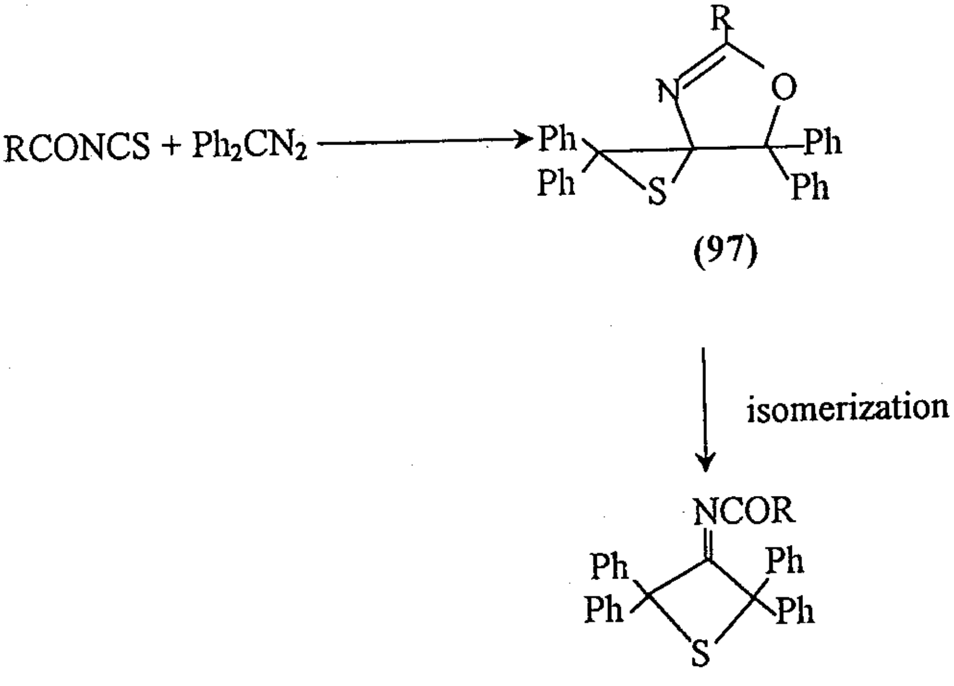


(95)



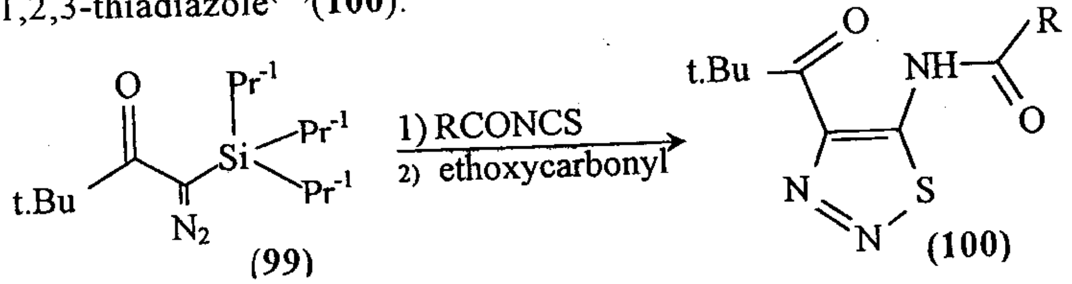
(96)

Similarly when acyl isothiocyanates reacted with diphenyl diazomethane it gave 4,5-dihydro-1,3-oxazole-4-spiro-2'-thiiranes (**97**) which isomerize in solution to the thietanes(88)(**98**). This isomerization also occurs when the spiro compounds are heated to their m.ps, but here the desulfurized products are also formed in some cases.

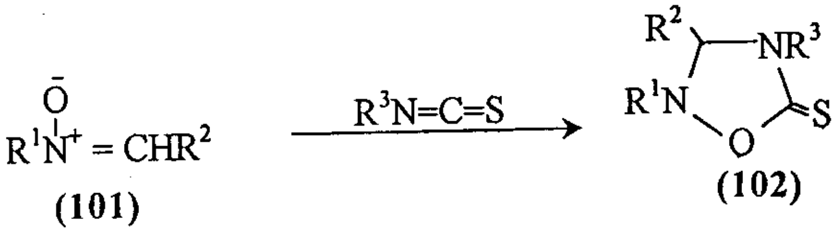


(98)

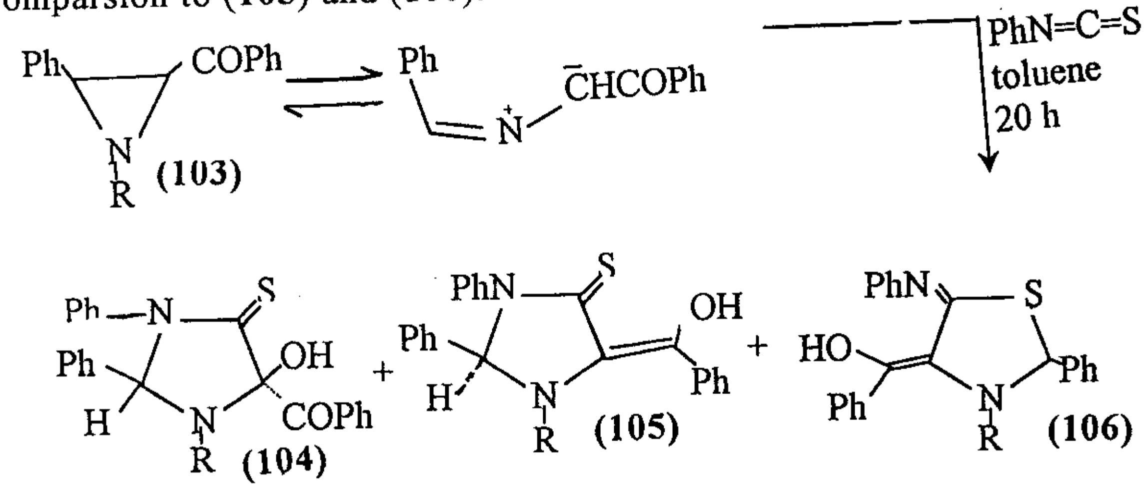
Also, 1-diazo-2-oxoalkylsilanes (**99**) reacted with acyl isothiocyanates and ethoxycarbonyl to give 4-acyl-5-acylamino-1,2,3-thiadiazole(89)(**100**).



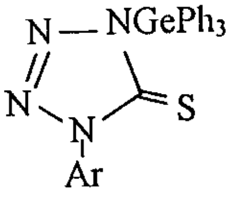
On the other hand,addition of nitrones (**101**)to alkyl and aryl isothiocyanates furnished oxadiazolidinethiones(90)(**102**) in variable yields.



1-substituted 2-benzolylaziridine (**103**) was converted into azomethine ylided and trapped with phenyl isothiocyanate to give imidazolidinethione derivatives (**104**) and (**105**), and iminothiazolidine (**106**). Yields were dependent on the reaction temperature and also on the period of heating. the shorter duration of heating either in benzene or toluene gave (**104**) in better yields in comparsion to (**105**) and (**106**).(91)



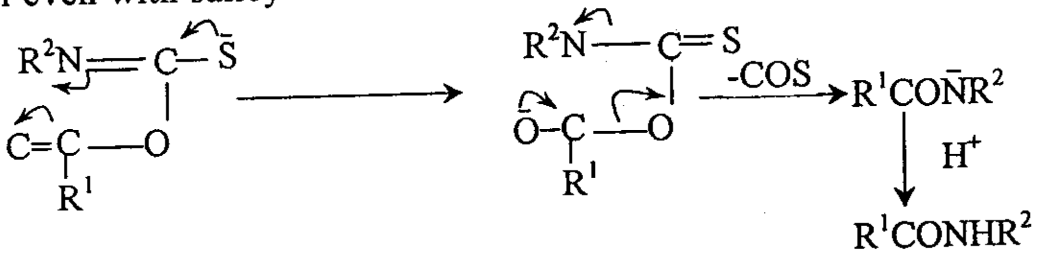
Germanium substituted tetrazole (**107**) was obtained by the interaction of aryl isothiocyanates and triphenylgermanium azide.(92)



(107)

5) Condensation with carboxylic acids:-

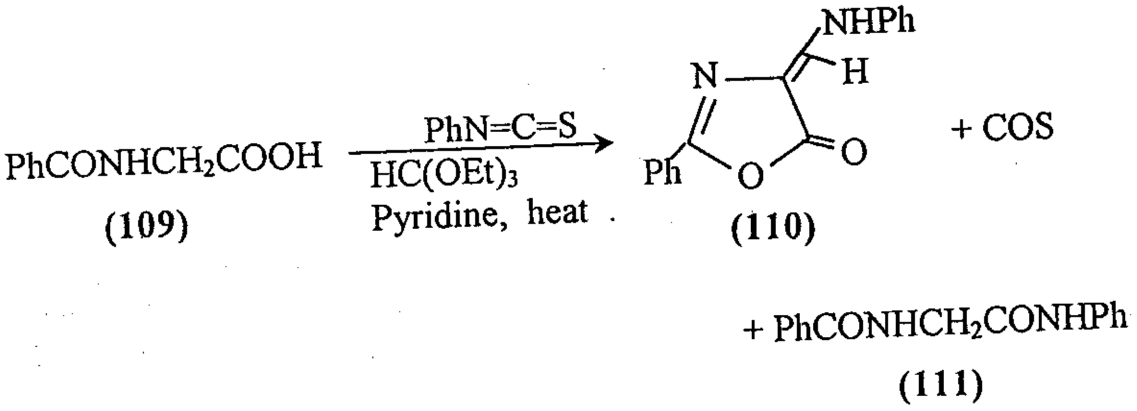
The condensation of carboxylic acids with isothiocyanates proceeded through addition-elimination reactions to yield amides (**108**). Pyridine as a catalyst facilitated the reaction, and it was successful even with salicylic acid.(93)



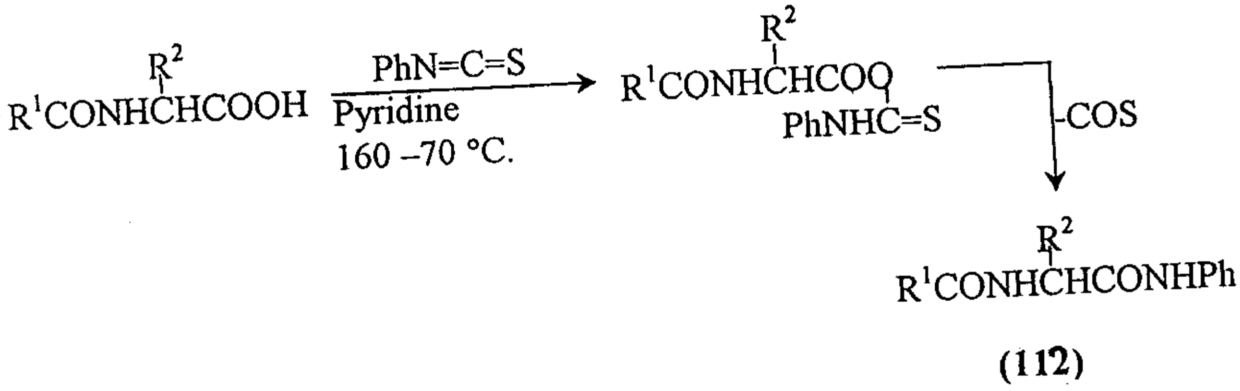
(108)

Compd.	R^1	R^1	Yield%
108			
a	Me	Ph	60
b	n-Pr	Ph	75
c	$PhCH_2$	n-Bu	63
d	$4-O_2NC_6H_4$	n-Bu	60
e	$2-HOC_6H_4$	Ph	41
f	$2-O_2NC_6H_4$	Ph	35

Also, N-benzoylglycine (**109**) condensed with phenyl isothiocyanate in the presence of triethyl orthoformate and gave 4-(anilino methylene)-2- phenyl-2-oxazolin-5-one(94) (**110**).



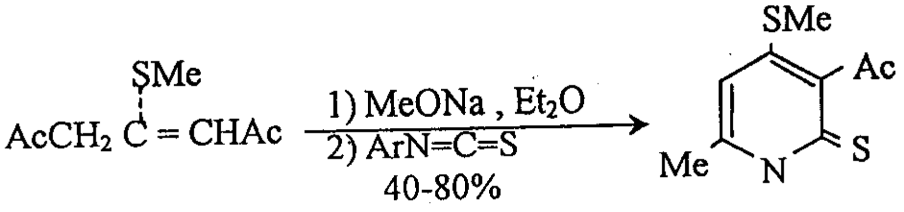
Similarly, the condensation of α-N-acylamino acids with phenyl isothiocyanate afforded the corresponding anilides(94) (**112**).



Also isothiocyanates reacted with N-acylglycines (**113**) in the presence of salicylaldehyde, led to the formation of 3-(N-acylamino) cumarins (**114**) as the major product along with a small amount of 2-substituted 4-(2-hydroxybenzylidene)-1 phenyl-2-imidazolin-5-one(95) (**115**).

7) Reaction with Carbonanion:-

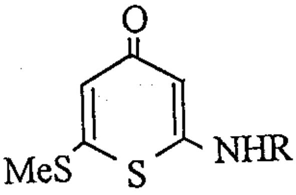
The condensation of aryl isothiocyanates with anion of diketone (**118**) furnished pyridinethiones(98) (**119**).



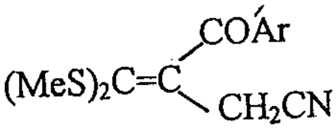
(**118**) (**119**)

Also, Ketene dithioacetal derived from acetone, underwent sodiun hydride aided condensation with alkyl and aryl isothiocyanates to give thiopyrones(99) (**120**).

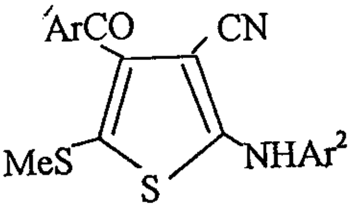
Similarly, anions from the polarized ketene dithioacetals (**121**) in step-wise regioselective cyclocondensation with aryl isothiocyanates gave the corresponding 4-aroyl-2- (arylamino)-5- (methylthio) thiophene-3-carbonitriles(100) (**122**).



(**120**)



(**121**)

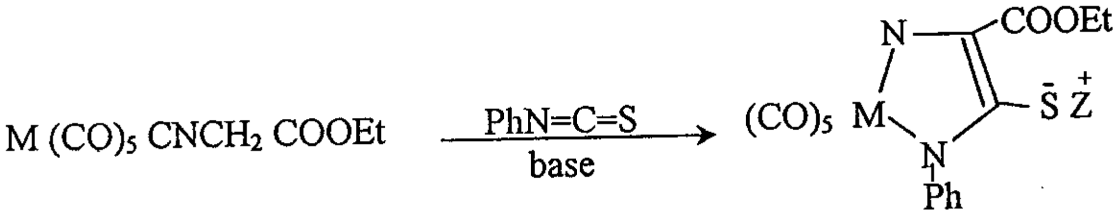


(**122**)

Ár =Ph , 4-MeOC$_6$H$_4$
Ar^2 = Ph , 4-MeC$_6$H$_4$

Also, the reaction of ketene with acyl isothiocyanates 4-RC$_6$H$_4$CONCS (R=MeO, H, Cl, NO$_2$) gave oxazinones (101) (**123**).

On the other hand, phenyl isothiocyanate reacted with the metal complexes of isocyanoacetate (**128**), in the presence of a base to give imidazole derivatives(104) (**129**).

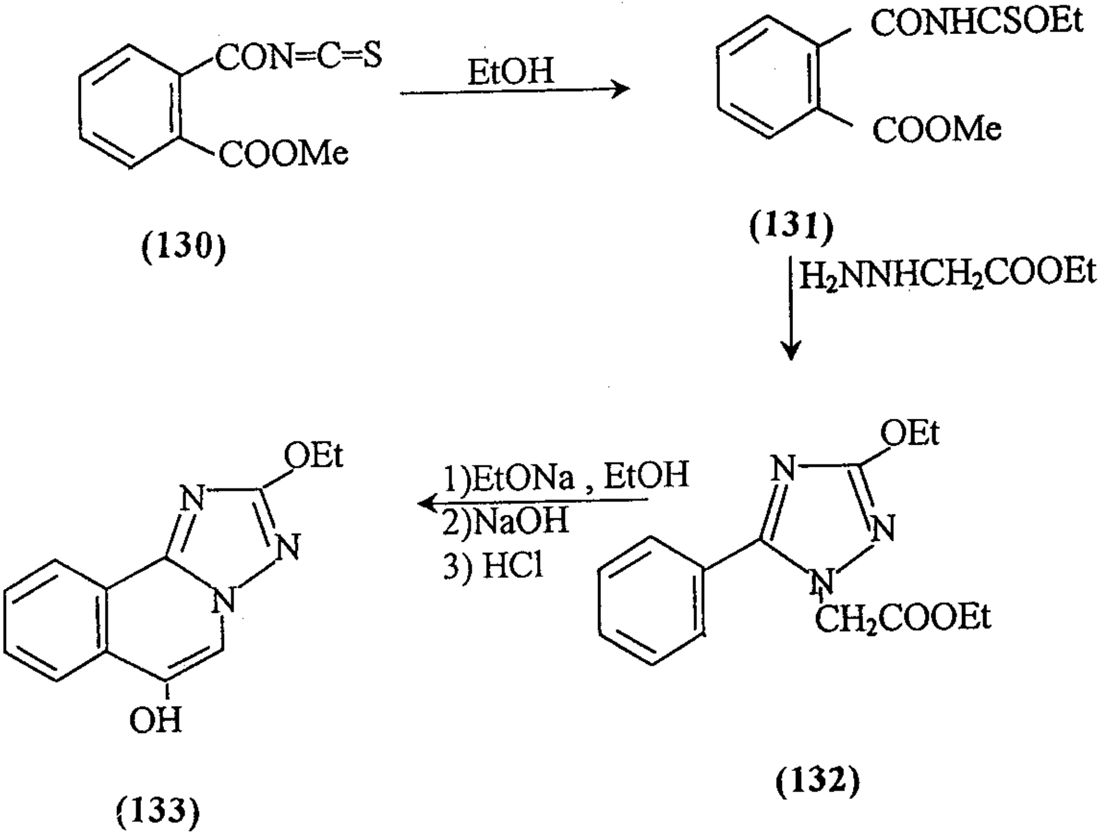


(128)
M=Cr.W

(129)
Z= Et_3NH , Li , K

8) Formation of thiocarbamate or dithiocarbamate and its cyclization:-

2-(Methoxycarbonyl) benzoyl isothiocyanates (**130**) was converted to (**131**) which on reaction with ethyl hydrazinoacetate gave triazole (**132**), the Dieckmann cyclization of which afforded triazoloisoquinoline(105) (**133**).



Also, o-(2-propenyl)N-acylmonothiocarbamates (**135**), prepared by the condensation of acyl isothiocyanates with allylic alcohol (**134**), underwent cope rearrangement to give S-(2-propenyl) N-acylmono-thiocarbamates(106) (**136**).

$$\mathrm{RCON{=}C{=}S + HO\text{-}CH_2\text{-}CH{=}CH_2 \longrightarrow RCONHCSOCH_2CH{=}CH_2}$$

(134) **(135)**

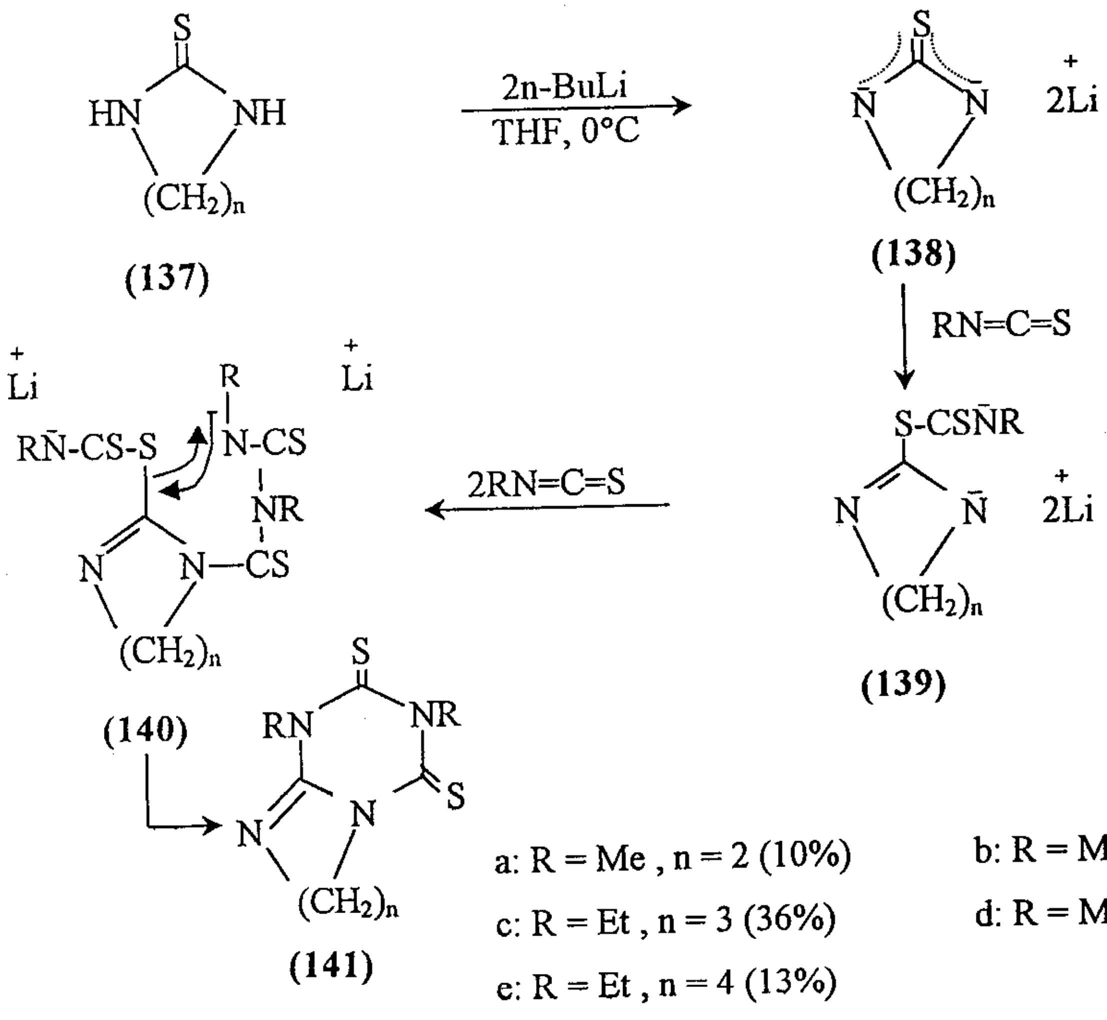
$\downarrow$ benzene 50-98%

$$\mathrm{RCONHCOSCH_2CH{=}CH_2}$$

(136)

R= aryl , heteroaryl

On the other hand, the condensation of isothiocyanates with anions generated from cyclic thioureas (**137**) was initiated by addition of the sulfur nucleophile.(107)



a: R = Me , n = 2 (10%)

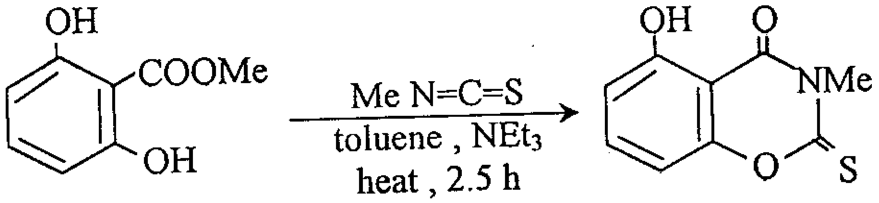
b: R = Me , n = 3 (61%)

c: R = Et , n = 3 (36%)

d: R = Me , n =4 (22%)

e: R = Et , n = 4 (13%)

Aromatic ketoximes reacted with isothiocyanates, whereas phenols of comparable acidity gave no adducts. However, triethylamine-aided condensation of **(142)** with methyl isothiocyanate gave the benzoxazine derivative **(143)** which exhibited fungicidal property.(108)

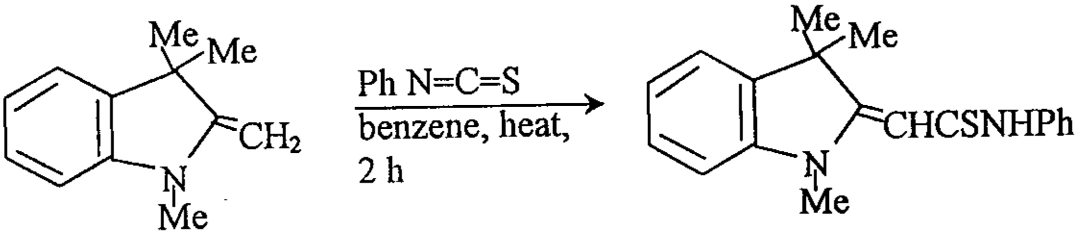


(142) **(143)**

9) Reactions involving an activated C=C bond:-

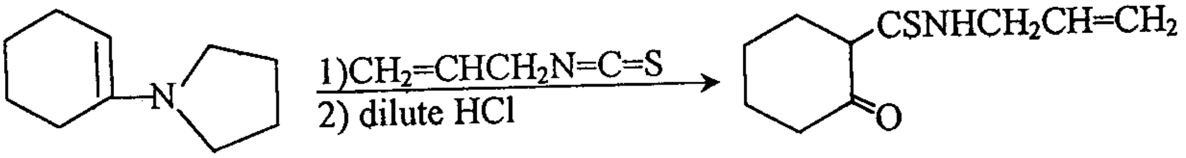
Compounds with C=C bond reacted as nucleophiles provided they carried an activating group, such as amino, alkoxy, alkylthio, etc., directly linked to the olefinic carbon atom.

The Fischer base **(144)** afforded E/Z isomers of(109) **(145)**.



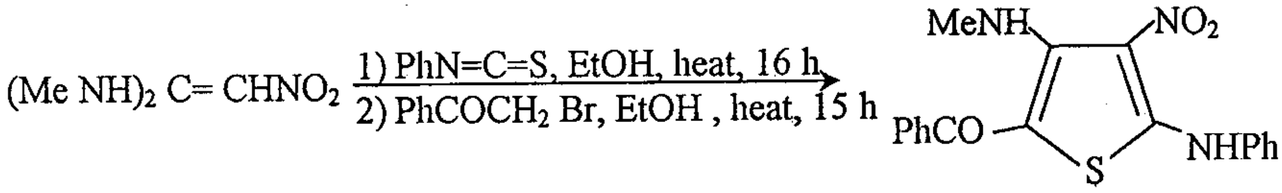
(144) **(145)**

Also, cyclopentanone and cyclohexanone were converted into the corresponding α-thioamido ketones via their enamines as shown in the preparation of(110) **(147)**.



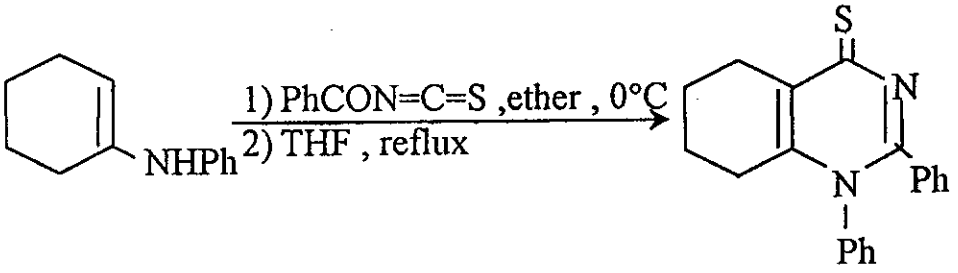
(146) **(147)**

Nitroketene aminal (**148**) with phenyl isothiocyanate formed an adduct in 28% yield which was converted into thiophene (**149**). This was the first direct synthesis of a nitroheterocycle from a nitro enamine.(111)



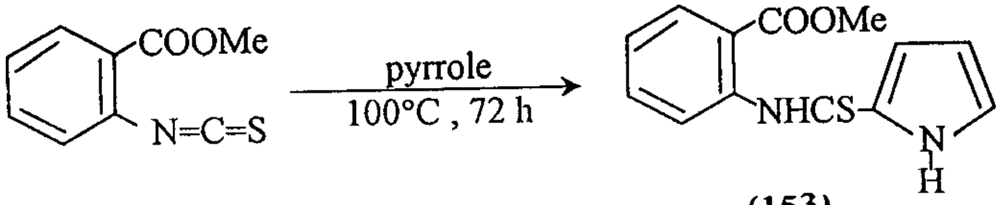
(148) **(149)**

Isothiocyanate bielectrophile reacted with secondary enamines to give heterocycles, as shown in the synhesis of(112) (**151**).



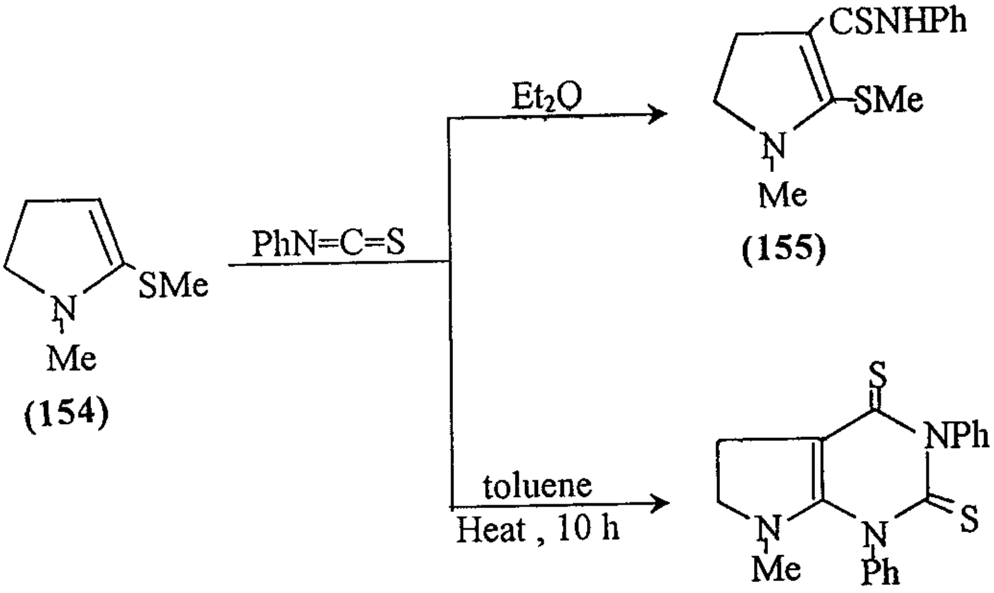
(150) **(151)**

On the other hand, pyrrole reacted with methyl-2-isothiocyanatobenzoate (**152**) and gave (113) (**153**).



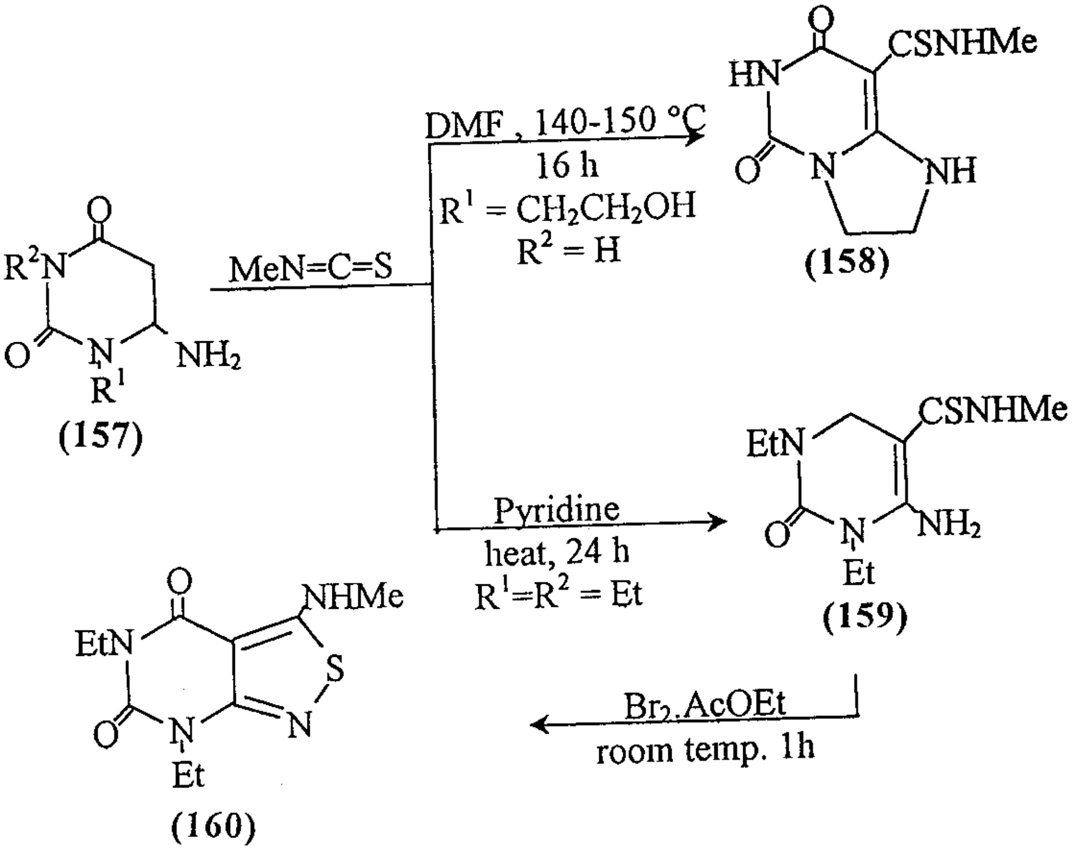
(152) (153)

Also, the semicyclic ketene S, N-acetals, for example **(154)**, reacted with an aryl isothiocyanate to give **(155)** or **(156)**, depending on the reaction conditions.(114,115)



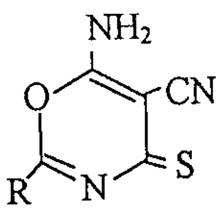
(154) (155) (156)

Similarly, 6-aminouracils **(157)** with isothiocyanates furnished 5-substituted thiocarbamoyl derivatives which were manipulated to yield various fused pyrimidine some of which found to be biologically active.(116,118)

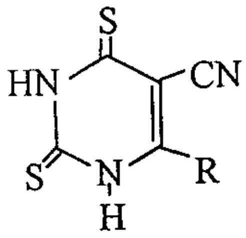


10) Other reactions:

Active methylene compounds, for example malononitrile reacted with aroyl isothiocyanate, ethylacetoacetate and/or cyanothioacetamide to give oxazines (**161**) or pyrimidines (119) (**162**).



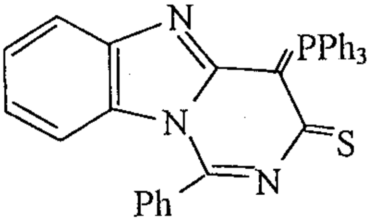
(161)



(162)

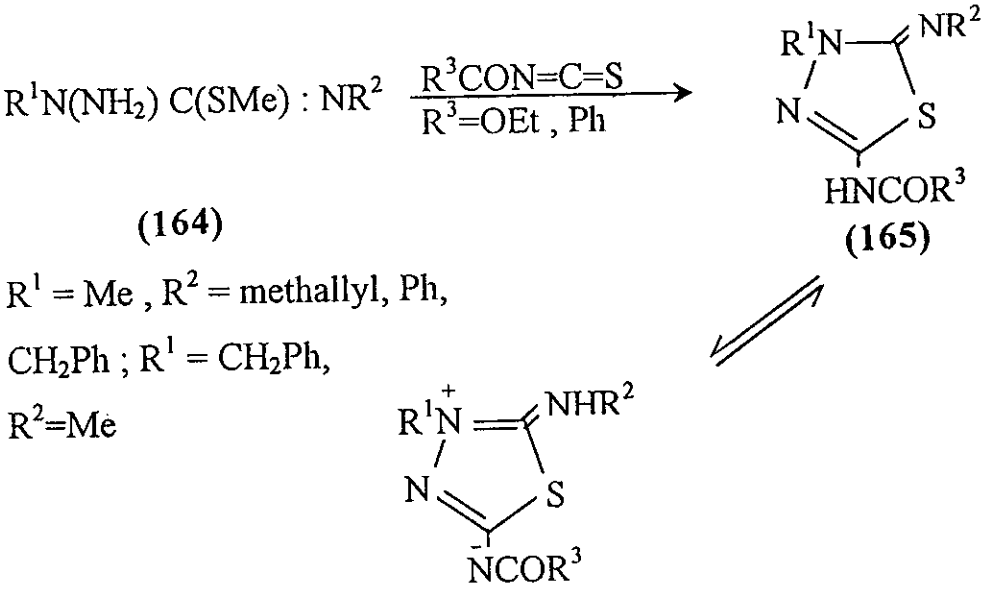
R = Ph , C$_6$H$_4$Cl-4 , C$_6$H$_4$OMe-4

(2-Benzimidazolylmethyl) triphenylphosphonium chloride condensed with benzoyl isothiocyanate in the presence of Et_3N to give pyrimidobenzimidazole derivative (120) **(163)**.



(163)

Isothiosemicarbazides **(164)** reacted with acylisothiocyanates under addition-cyclization to yield 1,3,4- thiadiazoline imines **(165)** as well as the isomeric 2-amino-substituted 1,3,4- thiadiazolium-5 acrylaminides (121) **(166)**.



(164)

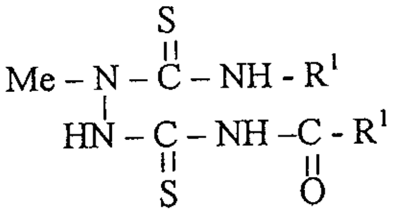
$R^1 = Me$, $R^2 =$ methallyl, Ph,
CH_2Ph ; $R^1 = CH_2Ph$,
R^2=Me

(165)

(166)

$R^1 = Me$, $R^2 =$ methallyl, $R^3 = Ph$; $R^1 = Me$
$R^2 = CH_2Ph$, $R^3 = Ph$; $R^1 = CH_2Ph$, R^2 Me , $R^3 = Ph$

Disubstituted thiosemicarbazides reacted with acylisothiocyanates to give bithioureas (**167**) which by the action of sodium ethanolate cyclize to 1,2,4- triazoline-3 thiones.(122)



$$\begin{array}{l} \text{Me}-\underset{|}{\text{N}}-\overset{\text{S}}{\overset{\|}{\text{C}}}-\text{NH}-\text{R}^1 \\ \quad\ \text{HN}-\underset{\text{S}}{\underset{\|}{\text{C}}}-\text{NH}-\underset{\text{O}}{\underset{\|}{\text{C}}}-\text{R}^1 \end{array}$$

(167)

R^1 = Me , Ph , methallyl ; R^2 =ethoxy , Ph