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SYNTHESES OF NITROGEN-CONTAINING HETEROCYCLIC COMPOUNDS BASED ON α-HALOKETONES (REVIEW)

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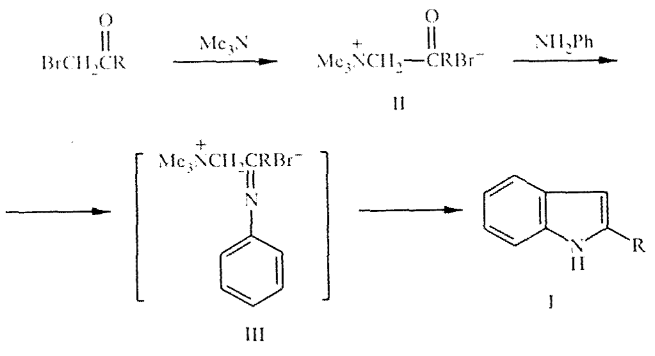
In this review, we systematize data on methods for synthesis of nitrogen-containing heterocyclic compounds cyclocondensation of α-haloketones with different compounds.

α-Halocarbonyl compounds are widely used in synthesis of different heterocycles. The huge amount of data, scattered over countless publications, makes an exhaustive presentation of it impossible.

In this review, we attempt to generalize and systematize literature information over the past 25-30 years on methods for synthesis of heterocyclic compounds based on α-haloketones within the range of nitrogen-containing heterocycles.

1. REACTION OF α-HALOKETONES WITH ANILINE

Cyclocondensation of α-haloketones with aromatic amines is one of the major methods for obtaining substituted indoles [1-2]. α-Haloketones of the aliphatic [3], aromatic [4, 5], and alicyclic [6] series enter into this reaction. The reaction occurs very slowly, at high temperature and in a stream of inert gas; the yields of end products are generally extremely low. S. I. Zav'yalov and coworkers developed a convenient two-step method for obtaining 2-substituted indoles (I) from α-bromoketones of the aliphatic series [7], including synthesis of the intermediate (2-R-2-oxoethyl)trimethyl ammonium bromide (II):



R = CH_2CHMe_2, $(CH_2)_4CH_3$, $(CH_2)_5CH_3$

The authors suggest that cyclocondensation of bromide II with aniline occurs through intermediate formation of the anils (III) followed by ring closure, leading to the products I in 60-90% yields.

It has been shown [8] that upon reaction of α-haloketones with aniline, not only are 2-substituted indoles formed but also 3,5-diaryloxazolium and 3,5-diarylthiazolium salts. By reaction of arylbromoketones with aniline, the aminoketones IV are synthesized; upon acylation of these aminoketones, the corresponding acyl derivatives V are obtained. Ring closure of the latter in the presence of P_2S_5 leads to 3,5-diarylthiazolium salts VI. The use of PCl_4 instead of P_2S_5 makes it possible to obtain 3,5-diaryloxazolium salts VII:

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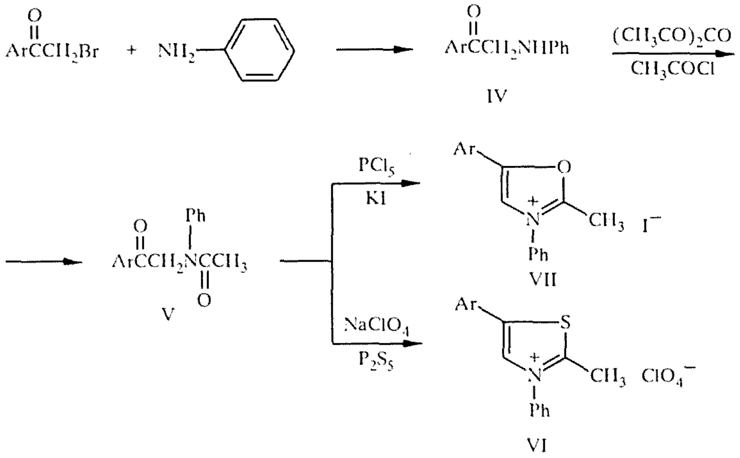
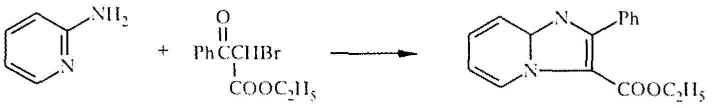

IV V VI VII

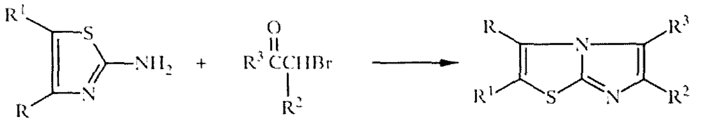
2. REACTION OF α-HALOKETONES WITH AMINOPYRIDINES AND AMINOTHIAZOLES

Heterocycles containing $NH_2C{=}N-$ moieties (2-aminothiazoles, 2-aminobenzothiazoles, 2-aminopyridines, and others) also can react with α-haloketones, which makes it possible to obtain condensed systems: imidazothiazoles, imidazobenzothiazoles, imidazopyridines, etc. [9-58]. Thus, upon reaction of 2-aminopyridine with ethyl-2-benzoyl-2-bromoacetate (VIII) in absolute alcohol, the imidazopyridine derivative IX is obtained in 90% yield [9].


VIII IX

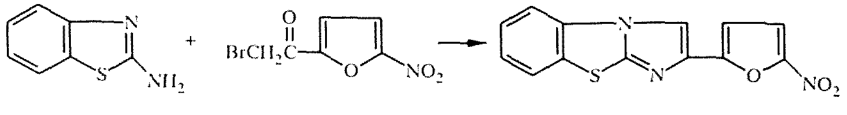
Imidazo[2,1-a]isoquinolines are similarly synthesized from 2-aminoisoquinoline and α-haloketones of the aromatic series [10]. Under the same conditions, imidazopyrimidines are formed from 2-aminopyrimidine [11-13], and the 3-acetyl derivative of imidazoquinoline is formed from 1-acetylaminoquinoline [14].

The reaction of 2-aminothiazoles with α-haloketones is widely used to obtain imidazothiazoles [15-26]:

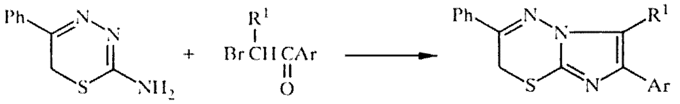


$R = R^1 = R^2 = H$, $R^3 = Me$ [15], Ar [16, 18, 20, 21], Het [22], CH_2COOEt [25]; $R = R^1 = H$, $R^2 = Me$, $R^3 =$ furyl-2 [24]; $R = R^3 = H$, $R^2 = Me$, $R^3 = MeC_6H_4$ [19]; $R = R^1 = Me$, $R^2 = H$, $R^3 = Ph$ [17]; $R = Me$, $R^1 = R^2 = H$, $R^3 =$ 5-Me-furyl-2 [23]; $R = CH_2COOEt$, $R^1 = Et$, $R^2 = H$, $R^3 = Ph$ [26]

The imidazobenzothiazole (XII) is synthesized by reaction of 2-aminobenzothiazole (X) with bromomethyl-5-nitro-2-furylketone (XI) [27]:

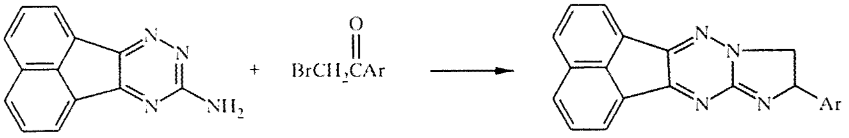

X XI XII

Introducing cyclic compounds with several heteroatoms into the reaction makes it possible to obtain complex condensed systems. The reaction of (α-bromoalkyl)arylketones with 2-amino-5-phenyl-1,3,4-thiadiazine (XIII) leads to the aryl-substituted 2H-imidazo[2,1-b][1,3,4]thiadiazine (CIV) [41, 42]:



XIII XIV

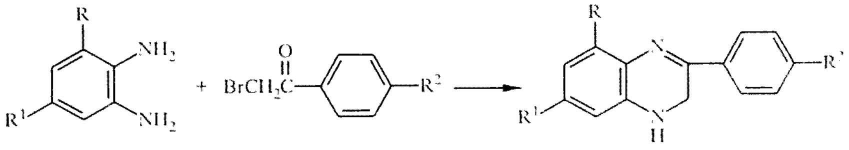
2-Aryl-substituted imidazo[2,1-b]acenaphtheno[1,2-c]-1,2,4-triazines (CVI) are obtained upon heating 3-amino-acenaphtheno[1,2-c]-1,2,4-triazine (XV) with (α-bromomethyl)arylketones [47]:



XV XVI

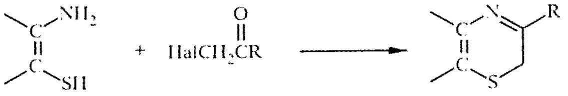
3. REACTION OF α-HALOKETONES WITH AROMATIC AND HETEROCYCLIC COMPOUNDS CONTAINING VICINAL AMINO GROUPS OR A VICINAL AMINO GROUP AND THIO GROUP

A series of heterocyclic derivatives have been described which are obtained by reaction of α-haloketones and compounds containing two amino groups on adjacent carbon atoms [59-61]. For example, 3-aryl-1,2-dihydroquinoxalines (XVII) have been obtained from o-phenylenediamine and its 4-chloro- and 3,5-dichloro derivatives in methanol [61]:

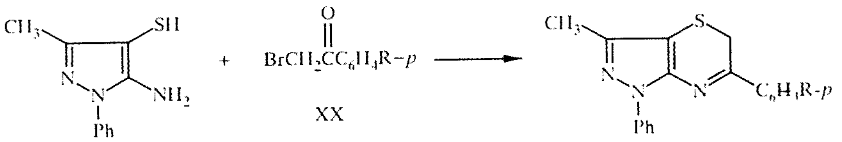


XVII

If a compound with vicinal SH and NH_2 groups is introduced into the reaction with α-haloketones, then the reaction occurs according to the scheme presented below [62-75].



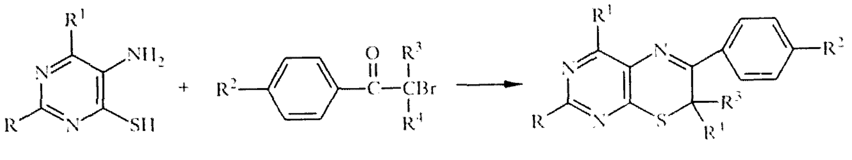
The authors of [64] report that derivatives of pyrazolo[4,5-b]-1,4-thiazine (XVIII) can be obtained by reaction of 5-amino-4-mercapto-3-methyl-1-phenylpyrazole (XIX) with ω-bromo-4-R-acetophenones (XX):



XIX XX XVIII

R = H, Cl, NO_2

Derivatives of pyrimido-1,4-thiazine (XXII) are obtained by reaction of 5-amino-6-mercaptopyrimidines (XXI) with (α-bromoalkyl)arylketones [71, 72]:



XXI XXII

$R = H, NH_2; R^1 = Cl, CH_3; R^2 = NO_2, Br, H, CH_3; R^3 = R^4 = H, CH_3$

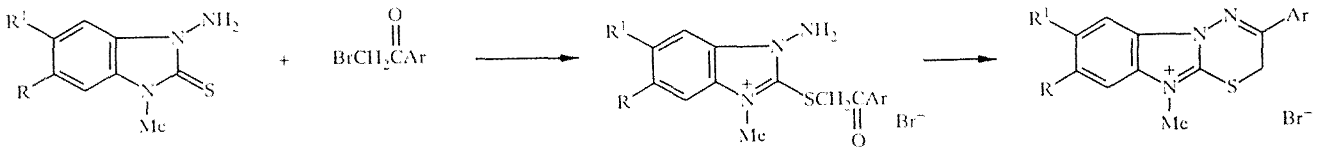
Similarly, 3-amino-2-mercapto-5,6-diphenylpyrazines when heated for 4-6 h in alcohol with phenacyl halides form derivatives of pyrazinothiazines in 70-90% yields [74].

The reactions can occur not only between α-haloketones and compounds containing SH and NH_2 groups, but also with compounds containing the SH group and heterocyclic nitrogen on the same carbon atom [37, 39, 40, 76-101]:

Thus the reaction of α-haloketones of the aliphatic and aromatic series with 2-mercaptoimidazoles leads to imidazothiazoles [85, 86], and reaction with 2-mercaptobenzimidazoles leads to thiazolobenzimidazoles [87-89]. The use of 2-mercaptoimidazolines in this reaction leads to 2-thiazoloimidazolines [90]. α-Haloketones reacted with 2-mercapto-3,4,5,6-tetrahydropyrimidine form thiazolotetrahydropyrimidine [91], and when reacted with 2-mercapto-sym-triazole they form 2-thiazolo-sym-triazole [92].

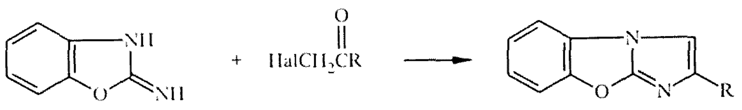
In addition to mercapto derivatives of heterocyclic compounds, thiones also may react with α-haloketones [101-111]:

Upon reaction of 1-amino-3-methylbenzimidazoline-2-thiones (XXIII) with phenacylbromides, a series of new 3-aryl-10-methyl-2H-1,3,4-thiadiazino[3,2-a]benzimidazolium bromides (XXIV) is obtained. It has been established that with a decrease in the reaction time, the intermediate salt of type XXV can be isolated:

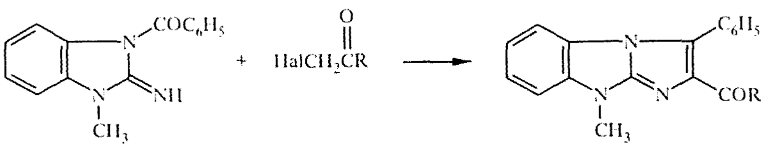


XXIII XXV XXIV

In the case of compounds in which the carbon atom is double-bonded to an imino group, the imidazobenzoxazoles XXVI [112, 113] and imidazobenzimidazoles XXVII [114, 115] can be obtained:



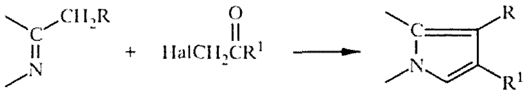
XXVI



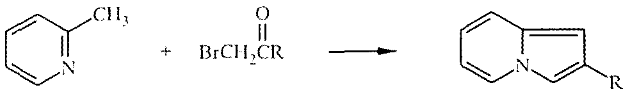
XXVII

4. REACTIONS OF α-HALOKETONES WITH 2-ALKYLPYRIDINES

Compounds containing an alkyl group in the α-position relative to the heterocyclic nitrogen also can undergo cyclocondensation with α-haloketones [84, 116-140]:



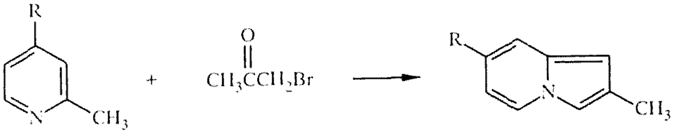
This reaction is one route to obtaining derivatives of indolizine with different substituents. Thus 2-picoline reacted with (bromomethyl)arylketones in absolute alcohol forms 2-R-indolizines (XXVIII) [119, 120]:



XXVIII

R = Ph, 4-ClC$_6$H$_4$, 4-MeC$_6$H$_4$

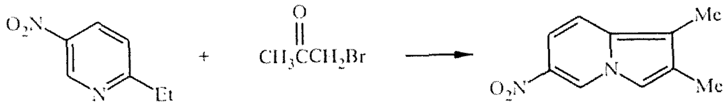
Similarly, the indolizines XXIX are formed from derivatives of α-picoline and bromoacetone [125, 126]:



XXIX

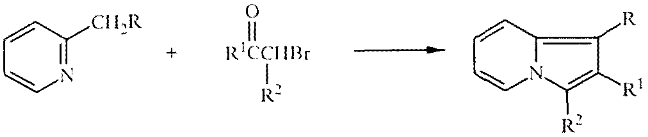
R = CH$_3$, NO$_2$

By changing the type and position of the substituents on the pyridine, different derivatives of indolizine can be obtained, such as 1,2-dimethyl-6-nitroindolizine XXX [131]:



XXX

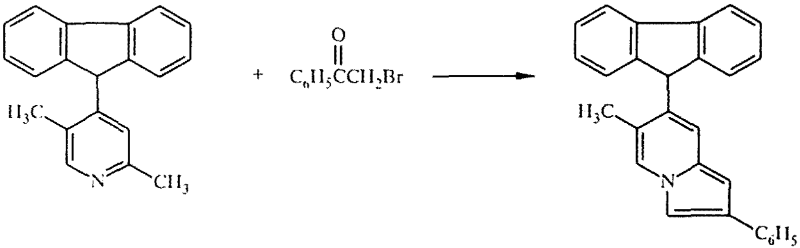
The authors of [135] describe the synthesis under similar conditions of a series of other indolizines XXXI [135]:



XXXI

R = H, CN, C$_6$H$_5$O; R^1 = CH$_3$, C$_6$H$_5$; R^2 = CH$_3$, C$_6$H$_5$

Upon reaction with bromoacetophenone, 9-(2′,5′-dimethylpyridyl-4′)fluorene (XXXII) is converted to 6-methyl-2-phenyl-7-(fluorenyl-9′)indolizine (XXXIII) [138]:



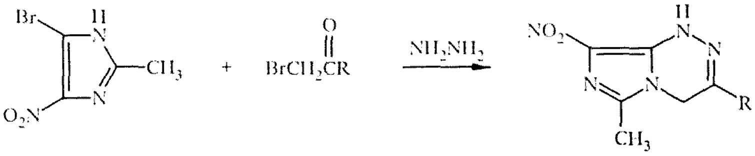
XXXII

XXXIII

A number of reactions involving three-component cyclocondensation of α-haloketones with $R-NH_2$ and heterocyclic compounds containing the $-NH-C-Hal$ moiety have been reported in [141-147]. Thus reaction of 4,5-diphenyl-2-bromoimidazole (XXXIV) with α-bromoketones and amines leads to imidazo[1,2-a]imidazole (XXXV) [143]:

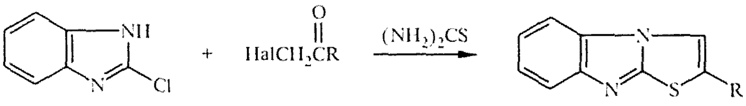
XXXIV XXXV

In turn, 2-methyl-4(5)-bromo-5(4)-nitroimidazole (XXXVI) reacted with α-haloketones and hydrazine hydrate forms imidazo[3,4-c]-1,2,4-triazines (XXXVII) [144]:



XXXVI XXXVII

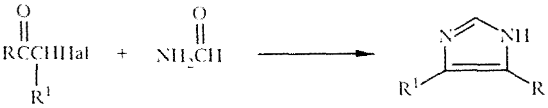
Reaction of 2-chlorobenzimidazole (XXXVIII) with α-haloketone and thiourea leads to a heterocyclic compound with three condensed rings (XXXIX) [145]:



XXXVIII XXXIX

5. SYNTHESIS OF IMIDAZOLES AND OXAZOLES

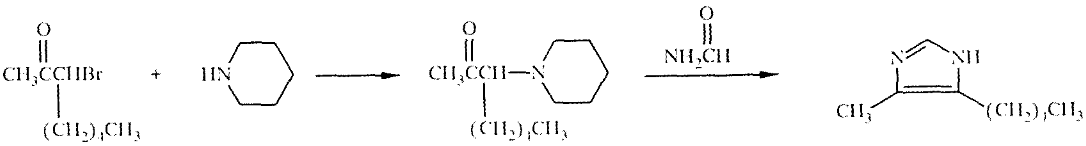
α-Galoketones can be converted to 4-mono- and 4,5-disubstituted imidazoles XL upon treatment with an excess of formamide, which fulfills the role of both an aminating and formylating agent:



XL

$R = Me$, $R^1 = H$ [148]; $R = p\text{-}NO_2C_6H_4$, $R^1 = H$ [149]

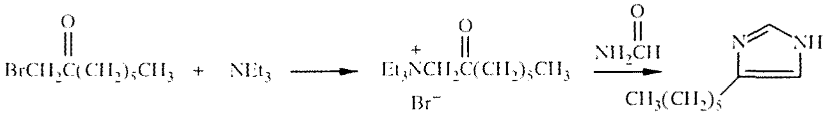
Thus 4,5-dialkylimidazoles are formed upon reaction of alkyl-α-bromoalkylketones with formamide [150]. The reaction is accompanied by significant tar formation. Tar formation is prevented and high stable yields of the target products can be ensured by addition of silica gel to the reaction mass [151]. With the goal of decreasing tar formation and increasing the yields of imidazoles, a two-step scheme for obtaining them has also been proposed in [150]. The first step includes synthesis of the aminoketone XLI; the second step involves its cyclization to the imidazole XLII:



XLII

XLI

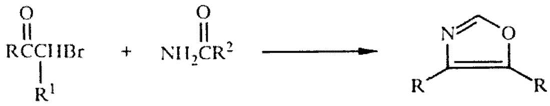
By such a route, it was possible to increase the yields of imidazole to 52%. Triethylamine can be used instead of piperidine. The quaternary salt XLIII formed in this case in the second step of the reaction with formamide is converted to 4-hexylimidazole (XLIV) in 40% yield [151, 152]:



XLIII XLIV

At the same time, 4-(adamantyl-1)imidazole is obtained in good yield from bromomethyl(adamantyl-1)ketone [6], which probably is connected with the high stability of the adamantane ring.

Carrying out the reaction of bromoketones with formamide in a sulfuric acid medium leads to the oxazoles XLV:

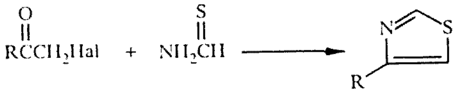


XLV

R = C_6H_5, p-$CH_3C_6H_4$; R^1 = H, CH_3; R^2 = H [153]; R = Ar, R^1 = H, R^2 = Ph [154]

6. SYNTHESIS OF THIAZOLES

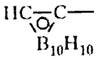
The reaction of α-haloketones with thioformamide is extremely interesting. This reaction makes it possible to obtain 4-substituted thiazoles XLVI [155, 156]:



XLVI

R = Me [157]; R = CH_2COOEt [158]; R = p-MeC_6H_4 [159]; R = p-ClC_6H_4 [160]; R = indolyl [161]; R = tropolyl [162]; R = adamantyl [165]

R =



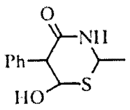
[163, 164]

Other thioamides also can be used, which leads to 4,5-disubstituted thiazoles XLVII:

XLVII

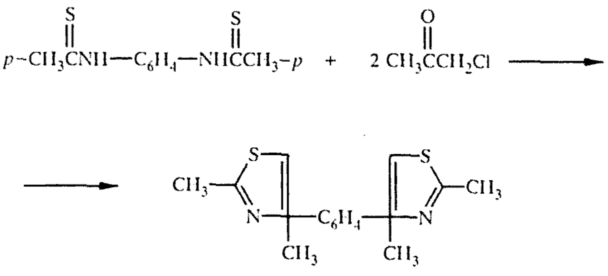
R = C_6H_5CO, R^1 = Me [166]; R = pyridyl -2, R^1 = Me [167]; R = indolyl 3, R^1 = Me [168]; R = tropolyl , R^1 = Me [162]; R = adamantyl-1, R^1 = Me [165]; R = Me, R^1 = t-Bu [169]; R = tropolyl R^1 = Ph [162]; R = 5- nitrofur -2-yl , R^1 = CH_2Ph [170]; R = Ph, R^1 = ArNHNC(COR) [171]; R = Alk, R^1 = res. of N-alkylamide of benzoic acid [172]; R = Ph, R^1 = pyridyl [153]; R = Ph, R^1 = indolyl -3 [173]; R = 4-ClC_6H_4, R^1 = pyridyl-4-yl [174]; R = Ph, R^1 = phenylindolyl[175]; R = quinol-8-yl , R^1 = methylimidazolyl 176]; R = Ph, R^1 = benzoylcyanomethyl [177]; R = Ph, R^1 = 1,5 dimetnyicyanopyrrolyl [178]; R = Ph [179],

R^1 =



[179]

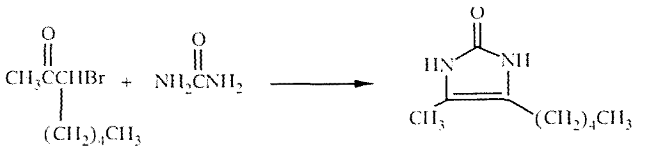
The use of bis derivatives of thioamides leads to the bis derivatives of thiazole XLVIII [180]:



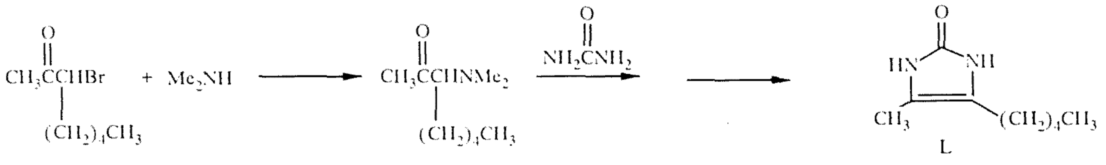
XLVIII

7. REACTION OF α-HALOKETONES WITH UREA AND THIOUREA

The reaction of α-haloketones with urea leads to imidazolin-2-ones. The latter can be synthesized both in a single step and through intermediate synthesis of aminoketones, which makes it possible to increase the yield and to reduce tar formation. One-step cyclocondensation could be accomplished for 3-bromoctan-2-one only in boiling ethyleneglycol and with an excess of K_2CO_3, with small amounts of the starting materials [181]:



A two-step synthesis is accomplished by reaction in the first step of α-bromoketones with dimethylamine or piperidine. The α-aminoketone formed (XLIX) upon cyclocondensation with urea is converted to imidazolin-2-one (L) [181-183]:

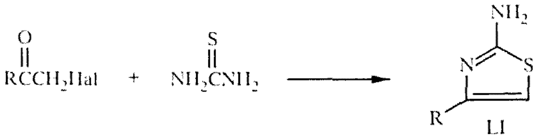


XLIX

L

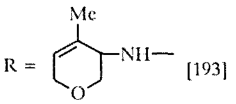
Intermediate formation of the aminoketone XLIX makes it possible to synthesize imidazolin-2-one (L) in up to 60% yields.

2-Aminothiazoles (LI) are formed upon reaction of α-haloketones with thiourea:

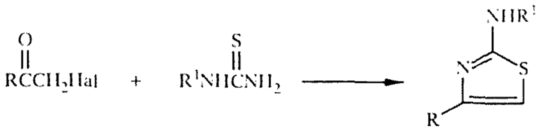


LI

R = hexyl [152], R = Ph [184], R = p-MeC$_6$H$_4$ [185], R = p-IC$_6$H$_4$ [186], R = p-methoxyphenyl [187], R = indolyl -3 [161, 168], R = pyridyl -2 [188], R = alkylthiophenyl [189], R = 5-bromofuryl -2 [190]; R = tropolyl [162], R = fluorenyl [191], R = coumarinyl-3 [192], R = adamantyl [165, 194],



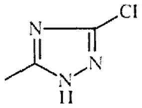
If the reaction is carried out with N-substituted derivatives of thiourea, then N-substituted 2-aminothiazoles (LII) are formed:



LII

R = tropolyl R^1 = Me [162]; R = p-IC_6H_4, R^1 = $NNHCOCH_3$ [195]; R = p-MeC_6H_4, R^1 = Ph [196]; R = p-FC_6H_4, R^1 = p-BrC_6H_4 [197]; R = p-ClC_6H_4, R^1 = $-N{=}CHC_6H_4Me$ [198]; R = p-BrC_6H_4, R^1 = $=CH{=}C(CN)Ph$ [199]; R = Ph, R^1 = $ArSO_2C(CONH_2){=}NNHAr$ [200]; R = imidazolyl , R^1 = 3,5- dimethylizoxazolyl -4 [202]

R = Ph. R^1 =



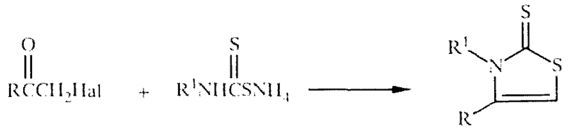
[201]

The reaction of bromoketones with bisthiourea LIII is of the same type. As a result of this reaction, bisthiazolylamine LIV is obtained [203]:

LIII LIV

R = Ph, Ar; R^1 = H, Me, Ph

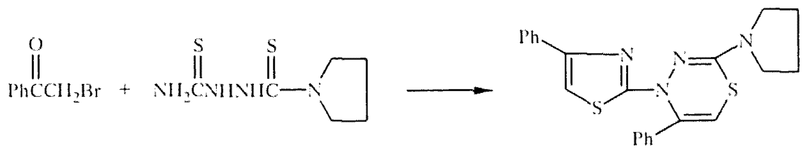
Thiazole derivatives, 2-mercaptothiazole (thiazole-2-thiones) (LV), can be obtained by reaction of α-haloketones with dithiocarbamates LVI:



LVI LV

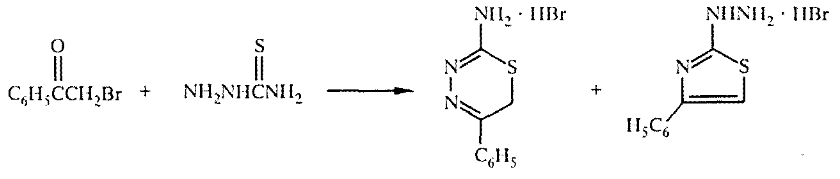
R = CH_3, R^1 = CH_2COOH [203]; R = Ph, R^1 = H [204]; R = Ar, R^1 = H [205]; R = methoxyphenyl R^1 = H [187]; R = indolyl , R^1 = H [161]; R = tropolyl , R^1 = H [162]

The biheterocycles LVIII are obtained by reaction of α-haloketones with dithiourea LVII [207]:



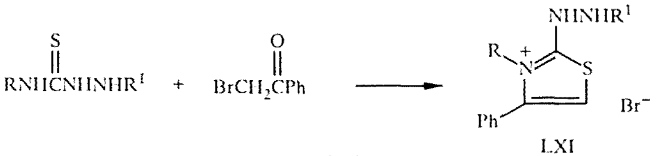
LVII LVIII

The reaction of α-haloketones with thiosemicarbazide is interesting. This reaction makes it possible to obtain a whole series of cyclic products whose structure is determined by the reaction conditions [156, 208]. Thus when phenacylbromide is heated with thiosemicarbazide in alcohol [207] in concentrated HCl [209] or HBr [210], two products are obtained:



LIX LX

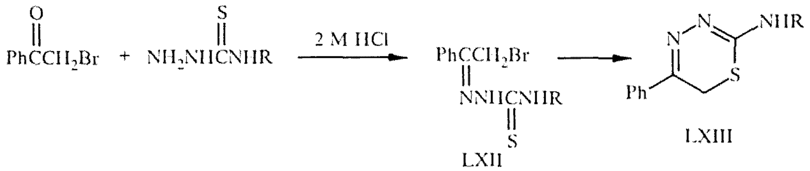
It has been found that prolonged heating of α-haloketones with thiosemicarbazide in alcohol leads to a mixture of thiadiazines, thiazoles, and thiazolines [206, 211]. It is interesting to note that upon condensation of α-bromoacetylpyridines [212-215] or ω-bromoacetophenones [216] in alcohol, only the corresponding thiadiazines are formed. The reaction of semicarbazones of substituted methyl-5-thiazolylketones with α-haloketones leads to 2-thiazolylhydrazones of the mentioned methyl-5-thiazolylketones [217]. Bromoketones reacted with 4-substituted thiosemicarbazides in alcohol yield thiazoles [218]; the thiazolium salts LXI are obtained when using 1,4-disubstituted thiosemicarbazides [219].



LXI

R and R^1 = Ph, p-substituted Ph

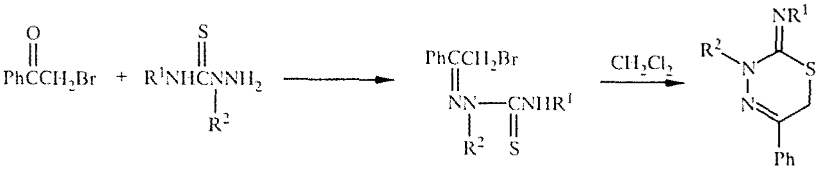
The corresponding thiosemicarbazones LXII are obtained from α-haloketones and 4-substituted thiosemicarbazides in 2 M hydrochloric acid; these thiosemicarbazones when heated in alcohol go to the thiadiazines LXIII [210]:



LXII LXIII

R = Me, Ph

Similarly, thiosemicarbazones LXIV are synthesized by reaction of ω-bromoacetophenones with 2,4-disubstituted thiosemicarbazides in ethanol. These thiosemicarbazones are converted by heating in dichloroethane to the thiadiazines LXV [220-222]:



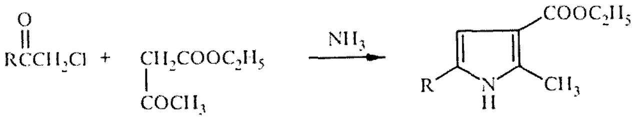
LXIV LXV

R = R^1 = Me, Ph

8. OTHER REACTIONS

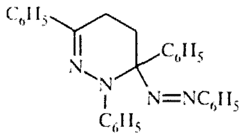
A number of reactions of α-haloketones do not fit into any of the schemes mentioned above. However, they are important and interesting from the standpoint of obtaining biologically active substances and intermediates for organic synthesis. Accordingly, it is advisable to separate such reactions into their own individual section.

The Hansch reaction serves as one method for synthesis of compounds of the pyrrole series (LXVI) [223]: reaction of α-haloketones with the acetoacetic ester LXVII in the presence of ammonia:



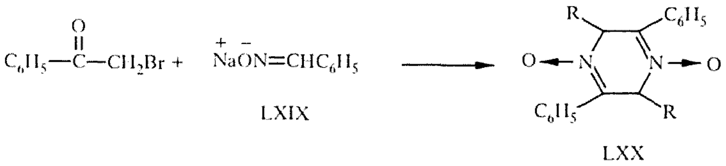
LXVII LXVI

α-Haloacetophenone reacts with phenylhydrazine to form 6-phenylazo-1,3,6-triphenyl-1,4,5,6-tetrahydropyridazine (LXVIII) instead of the expected hydrazone [224]:

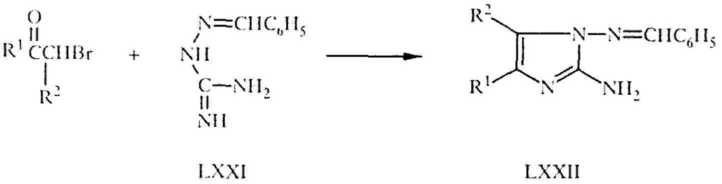


LXVIII

3,6-Diphenyl-2,5-dihydropyridazine-N,N-dioxide (LXX) is obtained by reaction of α-haloketones with the sodium salt of anti-benzaldoxime (LXIX) [225]

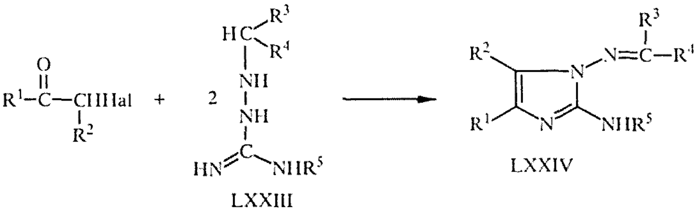


The reaction of benzaldehyde quanylhydrazone (LXXI) with α-bromoalkylarylketones leads to 2-amino-1-benzylideneaminoimidazoles (LXXII) [226]:



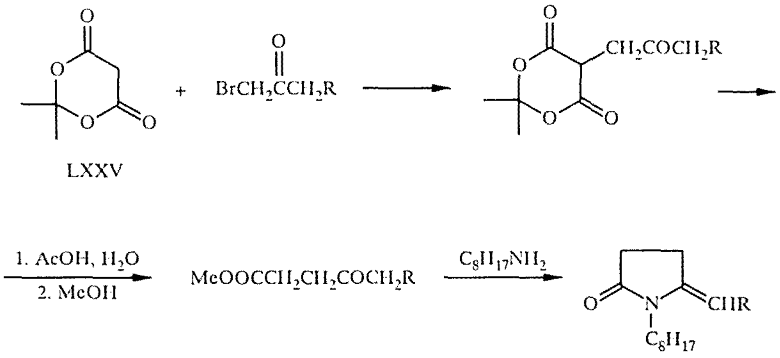
R^1 = Ar, R^2 = Alk

Similarly, interesting derivatives of imidazole LXXIV are obtained upon reaction of α-haloketones with two molecules of 4-alkylguanylhydrazone (LXXIII) [227]:



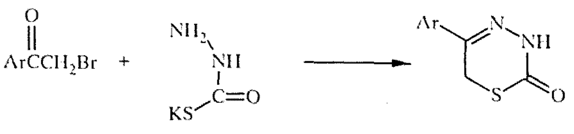
R^1 = Ar, R^2 = H, Ph, R^3 = Alk, R^4 = Ar, R^5 = Alk

Bromoalkanones monoalkylate Meldrum's acid (LXXV). Condensation of the monoalkylation product with octylamine yields 5-alkylidenepyrrolidin-2-ones [228]:



R = $(CH_2)_nCH_3$, n = 3, 4, 9

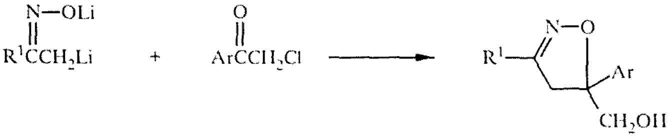
Cyclization of salts of thiocarbazinic acid with bromoacetylarenes leads to 5-aryl-3,6-dihydro-1,3,4-thiadiazin-2-ones (LXXVII) [206]:



LXXVI LXXVII

Diketosulfide, easily formed upon reaction of bromomethyl(1-adamantyl)ketone with sodium sulfide, in the presence of $TiCl_4$ easily undergoes ring closure and upon subsequent dehydration yields 3,4-diadamantylthiophene [229].

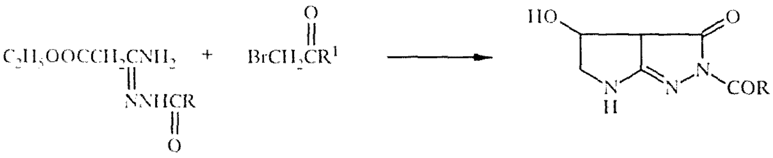
The reaction of dianions of oximes (LXXVIII) with chloroketones leads to 5-(hydroxymethyl-Δ^2-isoxazolines (LXXIX) [230]:



LXXVIII LXXIX

R^1 = Ph, p-MeC_6H_4, p-ClC_6H_4

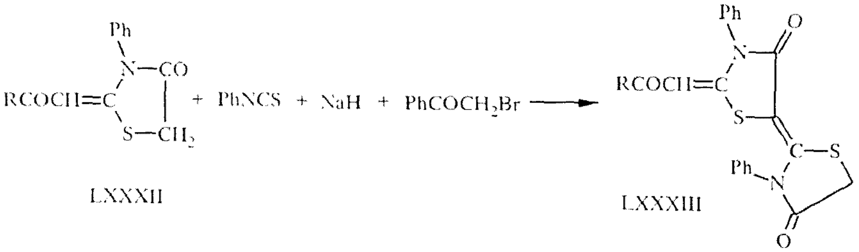
N-Acyl-2-ethoxycarbonylacetamidrazone (LXXX) reacted with α-haloketones forms pyrrolidino[2,3-c]pyrazol-3-ones (LXXXI) [231]:



LXXX LXXXI

R = CH_3, OC_2H_5, Ar; R^1 = CH_3, Ph

2-(R-carbonylmethylidene)-3-phenyl-5-(3-phenylthiazolidin-2-ylidene)thiazolid-4-ones (LXXXIII) are synthesized by reaction of 2-(R-carbonylmethylidene)-3-phenylthiazolidinones (LXXXII), PhNCS, NaH, and $PhCOCH_2Br$ [232]:



LXXXII LXXXIII

The data presented in this review suggest that the prospects are good for using α-haloketones of the aliphatic, alicyclic, aromatic, and heterocyclic series in synthesis of different heterocyclic compounds. Obviously the possibilities for synthesis based on these compounds have been far from exhausted, and it is advisable to continue investigations in this direction.

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