Reviews

Partially hydrogenated pyridinechalcogenones*

V. P. Litvinov

N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky prosp., 117913 Moscow, Russian Federation. Fax: +7 (095) 135 5328. E-mail: vpl@cacr.ioc.ac.ru

Data on the methods of synthesis, structure, and reactivity of partially hydrogenated 3-cyanopyridin-2(1*H*)-ones, -thiones, and selenones, which are promising substrates in the synthesis of annelated heterocycles containing a partially hydrogenated pyridine ring, are analyzed.

Key words: di- and tetrahydro-3-cyanopyridine-2(1*H*)-chalcogenones, annelated heterocycles, synthesis, structure, properties.

Introduction

Functionally substituted pyridinechalcogenones constitute a class of heterocyclic compounds that has been vigorously studied during the last 15 years; this has been reflected in several reviews.[1—11] This is due, on the one hand, to the theoretical interest in this peculiar class of organic compounds, and, on the other hand, to the fairly broad range of practical applications of their derivatives. Among these compounds, products exhibiting various types of biological activity have been found. In addition, compounds of this class can serve as stabilizing agents for polymers and varnishes, additives to fuels and oils, diazo components in the synthesis of dyes, models for redox coenzymes, and synthons in the syntheses of folic acid, vitamin B_6, and other biologically active products. New prospects for the use of these products appeared when HIV reverse transcriptase inhibitors and highly efficient cardiotonics and tranquilizers were detected among pyridone derivatives.

A special place in this class of compounds is occupied by partially hydrogenated pyridinechalcogenones; in particular, highly efficient cardiotonics have been prepared using these compounds.[1,11] In addition, 4-aryl-1,4-dihydropyridines have been used to develop new types of calcium antagonists[12—14] and CH-antioxidants,[15—17] while substituted spiro[piperidone-4,1′-cyclopentane] was employed in the synthesis of the antidepressant and analgesic "buspirone," which also prevents drug dependence.[18] All this is responsible for considerable interest in the chemistry of hydrogenated pyridinechalcogenones in recent years.

In this review, we analyze for the first time the data accumulated along this line over the last 10—15 years. To provide a fuller representation of the current state of this problem as a whole, in addition to the data obtained in our studies, we also included fundamental results obtained by other groups of researchers. The methods of synthesis are classified according to the types of reagents and substrates used, as was done in the plenary lecture delivered at the XV International Symposium on the

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Organic Chemistry of Sulfur[19] and in the theses devoted to the chemistry of partially hydrogenated pyridinechalcogenones.[20,21]

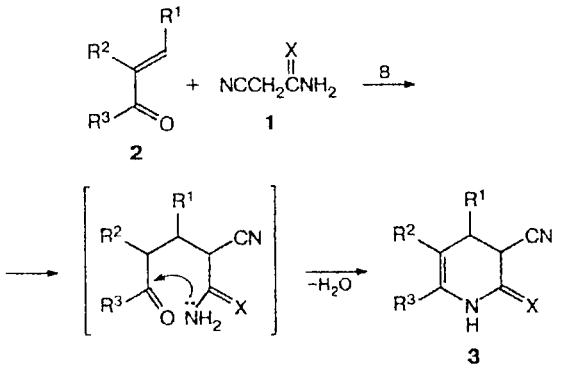
1. Synthesis of partially hydrogenated 3-cyanopyridin-2(1*H*)-ones, 3-cyanopyridine-2(1*H*)-thiones, and 3-cyanopyridine-2(1*H*)-selenones

The main methods used for the synthesis of partially hydrogenated pyridinechalcogenones are based on the same approaches that are used successfully in the syntheses of their aromatic analogs.[1] Among these methods, mention should first be made of the reactions of α,β-unsaturated carbonyl compounds, 1,3-dicarbonyl compounds, their enamines, and α,β-unsaturated nitriles with cyanoacetamide, its thio- and seleno-derivatives (**1a—c**), malononitrile, cyanoacetic acid, and other CH acids. Partially hydrogenated pyridinechalcogenones were not only postulated as intermediates in these reactions; in some cases, they were isolated as stable compounds, whose structure and reactivity have been studied fairly comprehensively.

1.1. Syntheses based on α,β-unsaturated carbonyl compounds

The reactions of α,β-unsaturated carbonyl compounds **2** with CH acids of type **1a—c** in the presence of organic bases (B) follows a mechanism of cascade heterocyclization. The first step gives Michael adducts, which undergo subsequent cyclocondensation to yield 3,4-dihydropyridine-2(1*H*)-chalcogenones (**3**).

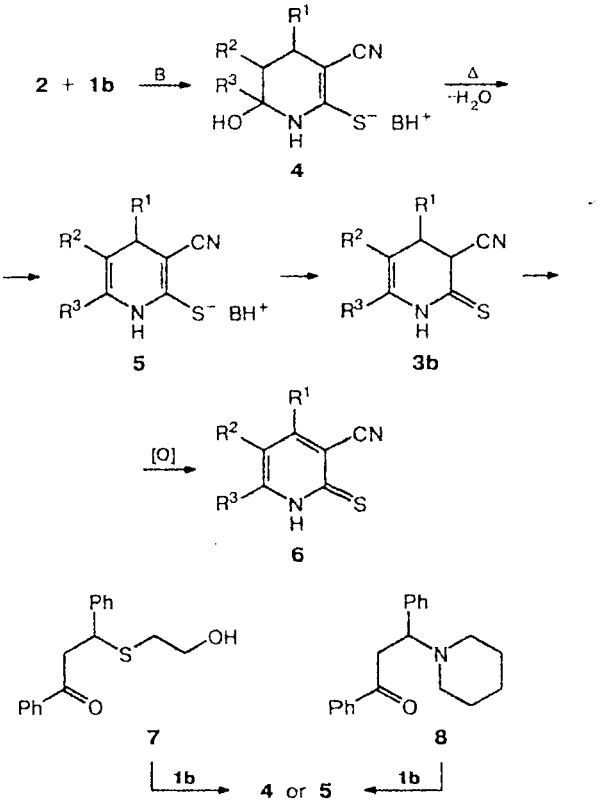
Scheme 1



X = O (**a**), S (**b**), Se (**c**); B is base

The formation of 3,4-dihydropyridine-2(1*H*)-thione (**3b**) derivatives as intermediates in the reaction of arylideneacetone or arylideneacetophenone with cyanothioacetamide (**1b**)[22a] was noted for the first time in 1980. Later, it was found that treatment of compounds **2** with cyanothioacetamide (**1b**) in ethanol in the presence of an equimolar amount of an organic base at 20 °C results in the formation of hydrogenated 3-cyanopyridine-2(1*H*)-thiones (**3b**), which were isolated and characterized as stable salts **4** and **5**.[22b—h] Substituted 3,4-dihydropyridinethiones (**3b**) are formed upon treatment of salt **5** with hydrochloric acid. They are relatively stable; on storage in solution, they are oxidized by atmospheric oxygen to 3-cyanopyridine-2(1*H*)-thiones (**6**). Salts **4** or **5** were also prepared by the reaction of amide **1b** with the adduct of chalcone with 2-mercaptoethanol **7** [23] or with the adduct of chalcone with piperidine **8**.[22b]

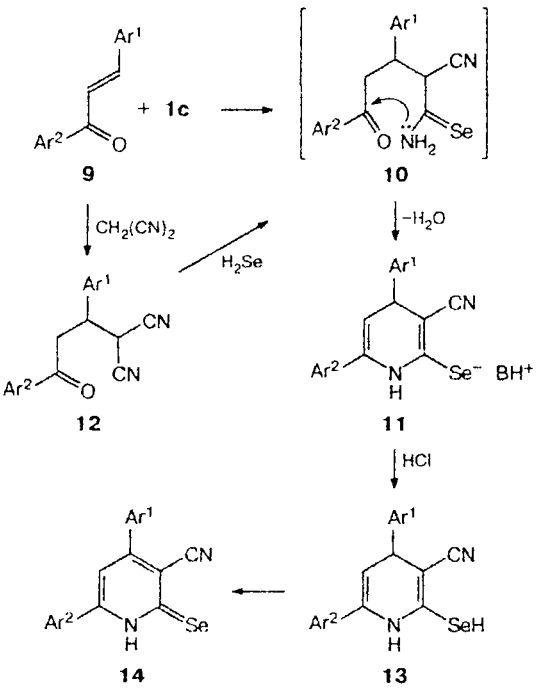
Scheme 2



The reaction of chalcones **9** with cyanoselenoacetamide (**1c**) in the presence of excess *N*-methylmorpholine affords Michael adducts (**10**), which undergo intramolecular cyclization to give 1,4-dihydropyridine-2-selenolates (**11**) in 60—90% yields.[20,21,24,25] The latter were also obtained by the reaction of chalcones **9** with malononitrile followed by the reaction of the δ-oxo dinitriles (**12**) thus formed with hydrogen selenide. Treatment of salts **11** with hydrochloric acid gives rise to 1,4-dihydropyridine-2-selenols (**13**), which are readily oxidized in the presence of atmospheric oxygen to give the corresponding pyridineselenones (**14**) (Scheme 3).

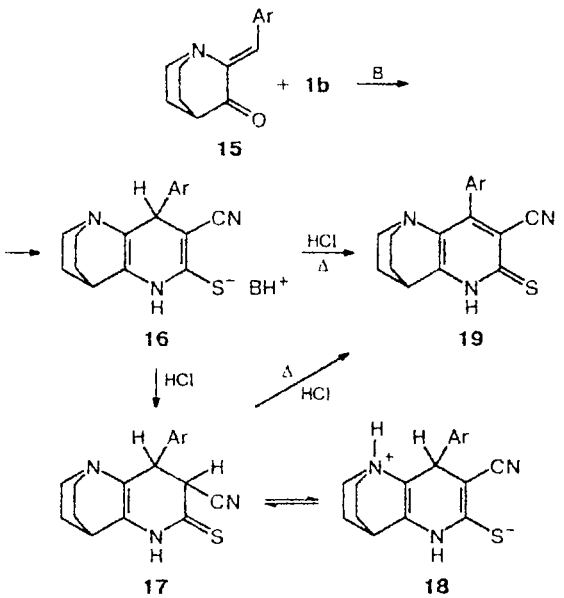
The reactions of 2-arylidenequinuclidin-3-ones (**15**) with cyanothioacetamide (**1b**) in the presence of piperidine result in the formation of piperidinium 4-aryl-3-cyano-5,8-ethano-1,4,5,6,7,8-hexahydro-1,5-naphthiridine-2-thiolates (**16**); on treatment with hydrochlo-

Scheme 3



ric acid, these products are converted into 4-aryl-3-cyano-5,8-ethano-3,4,5,6,7,8-hexahydro-1,5-naphthiridine-2(1*H*)-thiones (**17**), existing in a tautomeric equilibrium with betaines **18**.[26] When salts **16** or betaines **18** are heated in ethanol in the presence of hydrochloric acid, they are dehydrogenated to give naphthiridinethiones **19**.

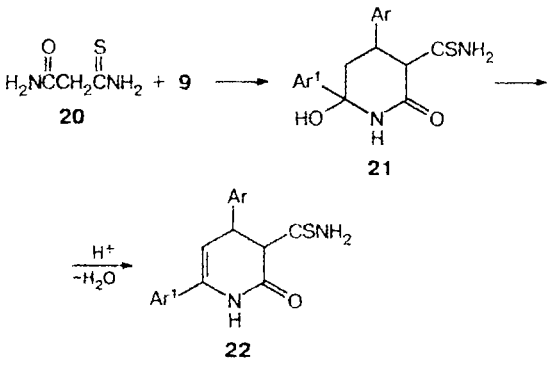
Scheme 4



The reaction of aryl(hetaryl)chalcones with amides **1a** and **1b** in the presence of alkali metal alkoxides or amines as catalysts was also used to prepare a number of 3,4-dihydrogenated derivatives of 3-cyanopyridine-2(1*H*)-ones and -thiones.[27,28]

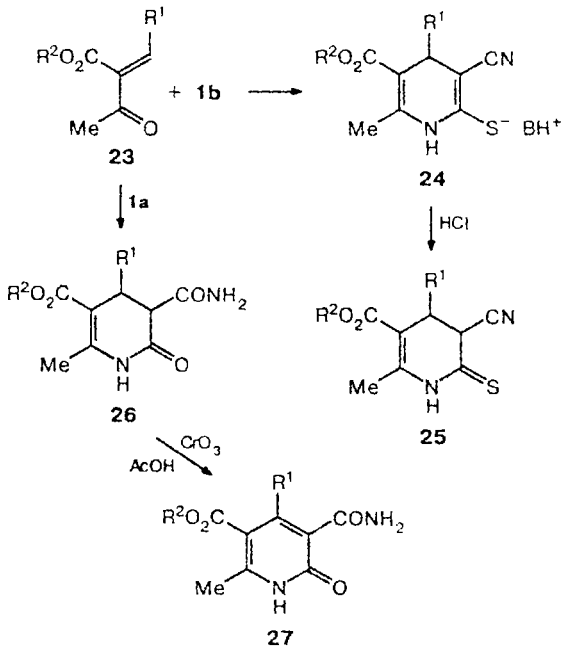
Thiocarbamoylacetamide (**20**) reacts with chalcones **9** in the presence of piperidine or sodium methoxide to yield substituted piperidones **21**, which are converted into 3,4-dihydropyridones **22** in an acidic medium (Scheme 5).[29a,b]

Scheme 5



The reaction of cyanothioacetamide (**1b**) with esters of 2-alkylideneacetoacetic acid (**23**) in ethanol in the presence of excess piperidine at 20 °C affords 1,4-dihydropyridine-2-thiolates (**24**), which were converted

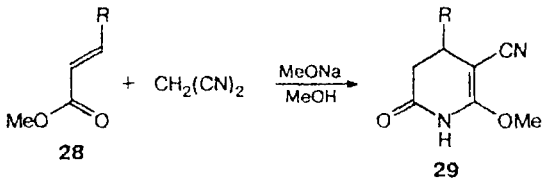
Scheme 6



into thiones **25** by treatment with hydrochloric acid.[30] A similar reaction between esters **23** and cyanoacetamide (**1a**) occurs when the reaction mixture is refluxed in ethanol in the presence of triethylamine; 3,4-dihydro-2(1*H*)-pyridone (**26**) formed in this reaction in 37% yield was oxidized by CrO_3 in acetic acid to the corresponding pyridone **27**.[31]

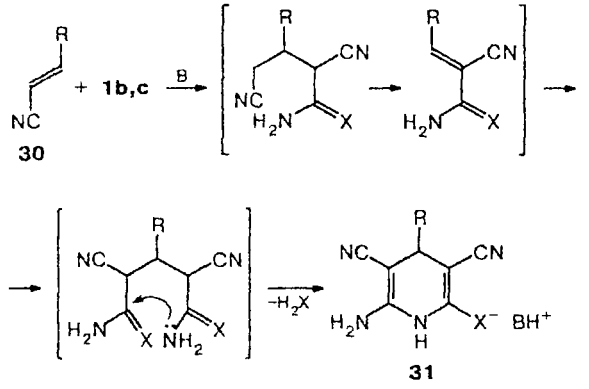
The use of sodium methoxide as the catalyst in the reaction of substituted methyl acrylates **28** with malononitrile results in the formation of 3,4-dihydro-2(1*H*)-pyridones (**29**) in 49—85% yield.[32]

Scheme 7



When 3-phenyl- and 3-(2-furyl)acrylonitriles (**30**) were made to react with cyanothio(seleno)acetamides (**1b,c**) in ethanol at room temperature in the presence of a twofold excess of *N*-methylmorpholine, substituted 1,4-dihydropyridine-2-thiolates and -selenolates **31** were obtained, as a result of the Michael reaction occurring as exchange by methylene components.[20,21,33] The reactions of amides **1b,c** with ethyl 3-phenyl- and 3-(2-furyl)acrylates occur in a similar way.

Scheme 8

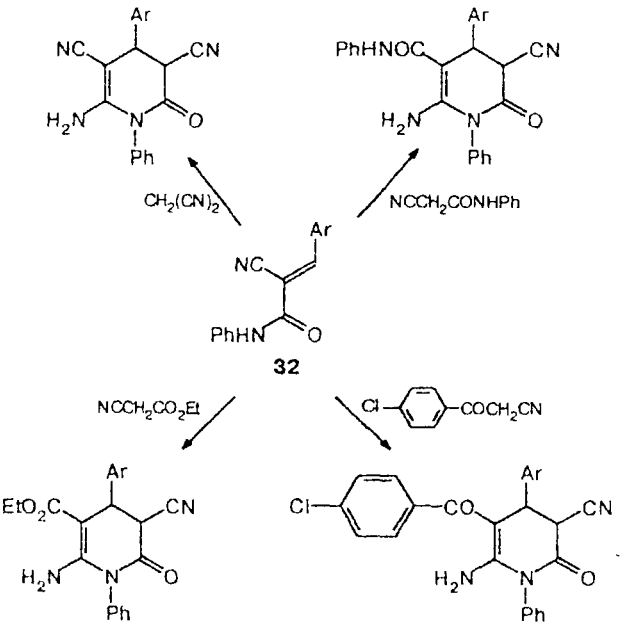


R = Ph, 2-furyl; X = S, Se

The reactions of anilides of 2-cyanocinnamic and other 3-aryl-2-cyanoacrylic acids **32** with CH acids occurring on refluxing in ethanol in the presence of piperidine have been used successfully in the synthesis of diverse 3,4-dihydro-2(1*H*)-pyridones (Scheme 9).[34]

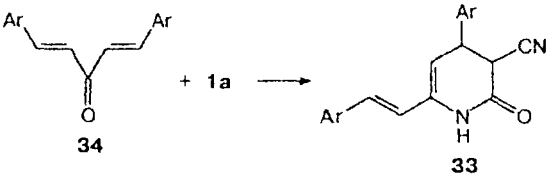
Substituted 3-cyano-3,4-dihydropyridin-2(1*H*)-ones (**33**) have been synthesized by heating a mixture of pentadienone **34** and cyanoacetamide (**1a**) in a sealed

Scheme 9



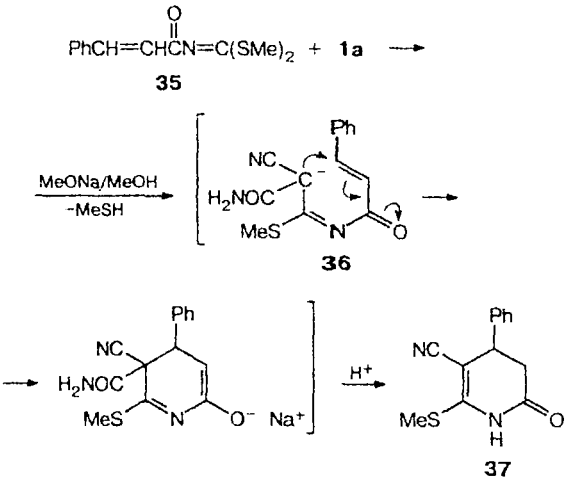
tube at 170 °C without a solvent; the yields of 3,4-dihydro-2(1*H*)-pyridones (**33**) reach 32—37% (Scheme 10).[35]

Scheme 10



Condensation of α,β-unsaturated carbonyl compound **35**, containing active di(methylthio)methylene group,

Scheme 11

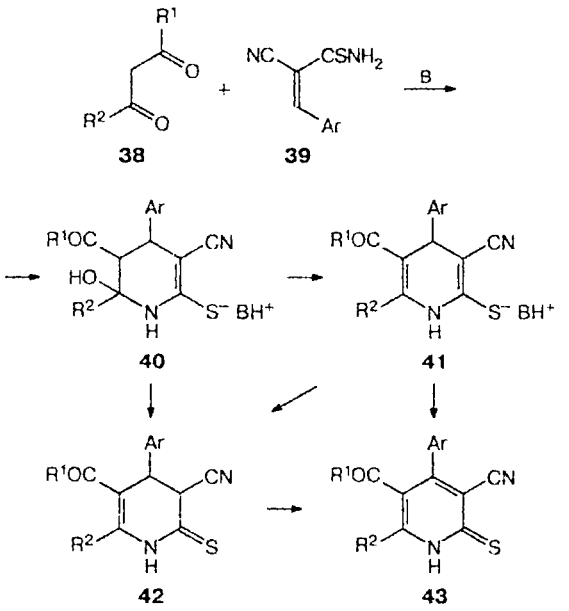


with amide **1a** in methanol in the presence of sodium methoxide occurs with elimination of methanethiol and intermediate formation of Michael adducts **36** and affords 5-cyano-6-methylthio-4-phenyl-3,4-dihydropyridine-2(1*H*)-one (**37**) (yield 70%) (Scheme 11).[36]

1.2. Syntheses based on 1,3-dicarbonyl compounds

Like α,β-unsaturated analogs, 1,3-dicarbonyl compounds **38** or their enamines react with arylidenecyanothioacetamides (**39**) giving rise to hydrogenated 3-cyanopyridine-2(1*H*)-thiones or their salts.[6,30,37] In some cases, tetrahydropyridine-2-thiolates (**40**) can be isolated; subsequent elimination of water results in the formation of 3-cyano-1,4-dihydropyridine-2-thiolates (**41**). Acidification of salts **40** and **41** gave 3-cyano-3,4-dihydropyridine-2(1*H*)-thiones (**42**), which are dehydrogenated in solution to afford 3-cyanopyridine-2(1*H*)-thiones (**43**), although the yields of these products are low. β-Enamino derivatives of 1,3-dicarbonyl compounds can also react with amides **39** without basic catalysts to give the corresponding 1,4-dihydropyridine-2-thiolates in high yields.

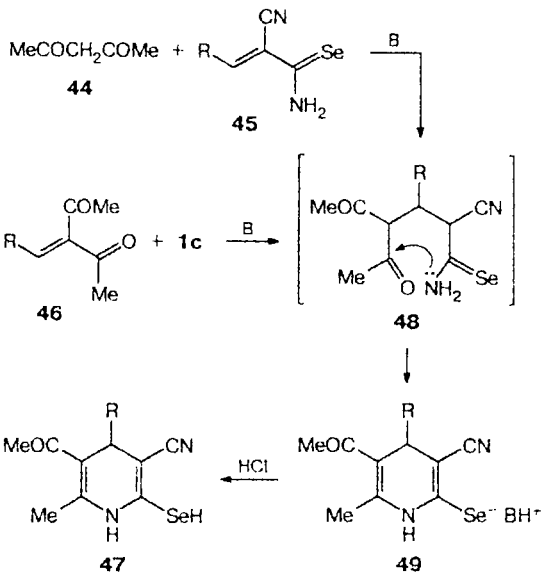
Scheme 12



The reaction of acetylacetone (**44**) with 2-furfurylidenecyanoselenoacetamide (**45**) or 2-furfurylideneacetylacetone (**46**) with cyanoselenoacetamide (**1c**) affords 5-acetyl-3-cyano-4-(2-furyl)-6-methyl-1,4-dihydropyridine-2-selenol (**47**) in ~80% yield.[21,38] The process includes the intermediate formation of Michael adduct **48** and stable salt **49**, whose acidification gives rise to selenol **47**. Amide **45** reacts with ethyl acetoacetate or its enamine in a similar way to give 5-ethoxycarbonyl-substituted selenol of type **47** (Scheme 13).

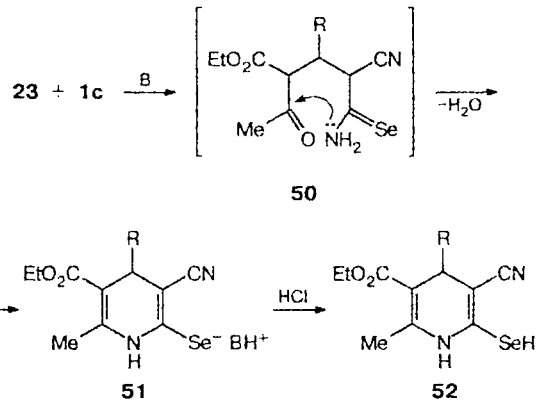
Similarly to 2-furfurylideneacetylacetone (**46**), benzylidene- and 2-furfurylideneacetoacetic esters (**23**) react with amide **1c** under mild conditions (anhydrous ethanol, room temperature); the ratio of the reactants plays an important role in this reaction.[21,39–41] Thus when the ratio is 1 : 1, the reaction yields adducts **50**, which cyclize to 1,4-dihydropyridine-2-selenolates (**51**). The latter were converted into 1,4-dihydropyridine-2-selenols (**52**) by treatment with hydrochloric acid.

Scheme 13



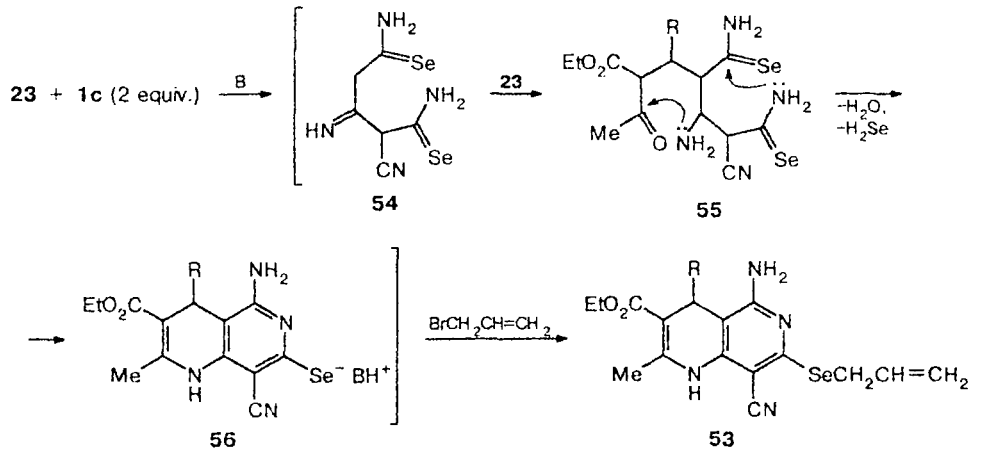
R = 2-furyl

Scheme 14



Condensation of a twofold excess of amide **1c** with 2-furfurylideneacetoacetic ester (**23**) under the same conditions followed by treatment of the reaction mixture with allyl bromide gave unexpectedly 1,4-dihydro-1,6-naphthiridine (**53**), whose structure was confirmed by X-ray diffraction analysis.[41] The first step of the reaction is Thorpe dimerization of cyanoselenoacetamide

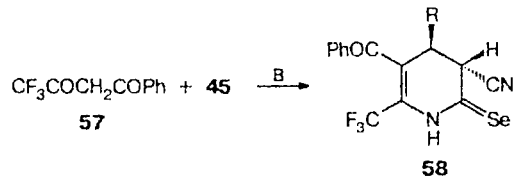
Scheme 15



(**1c**) giving iminonitrile **54**, which adds to ester **23** according to Michael to afford adduct **55**. The latter undergoes cyclocondensation under the reaction conditions yielding 1,4-dihydro-1,6-naphthiridine-7-selenolate (**56**); alkylation of this product with allyl bromide results in the formation of naphthiridine **53** (Scheme 15).

The reaction of benzoyltrifluoroacetone (**57**) with 2-thenylidenecyanoselenoacetamide (**45**) in the presence of *N*-methylmorpholine in anhydrous ethanol under argon at 20 °C gave *trans*-5-benzoyl-3-cyano-4-(2-thienyl)-6-trifluoromethyl-3,4-dihydropyridine-2(1*H*)-selenone (**58**) in 78% yield.[37f]

Scheme 16

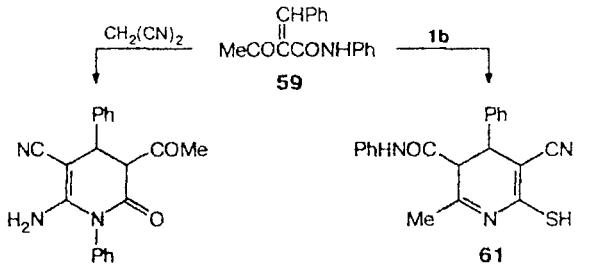


R = 2-thienyl

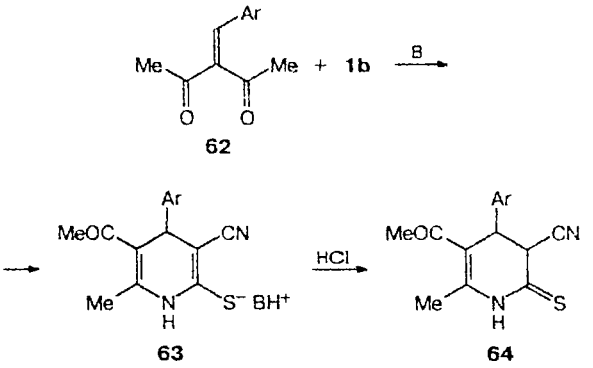
The addition of malononitrile and cyanothioacetamide (**1b**) to 3-benzylideneacetoacetanilide (**59**) in ethanol in the presence of a catalytic amount of piperidine has been studied.[37l] The authors believe that in the former case, pyridone **60** is formed as a result of intramolecular interaction between the cyano and phenylcarbamoyl groups in the Michael adduct arising *in situ*, and in the latter case, the acetyl group reacts with the thiocarbamoyl group according to the intramolecular condensation pattern to give thiol **61** (Scheme 17), although the existence of this prototropic tautomer for this class of compounds is relatively unlikely.[1]

When 3-arylideneacetylacetone (**62**) reacts with amide **1b** under similar conditions, thiolates **63** are produced. On treatment of the reaction mixture with hydrochloric acid, products **63** are converted into thiones **64**, which exhibit neurotropic activity.[22g]

Scheme 17

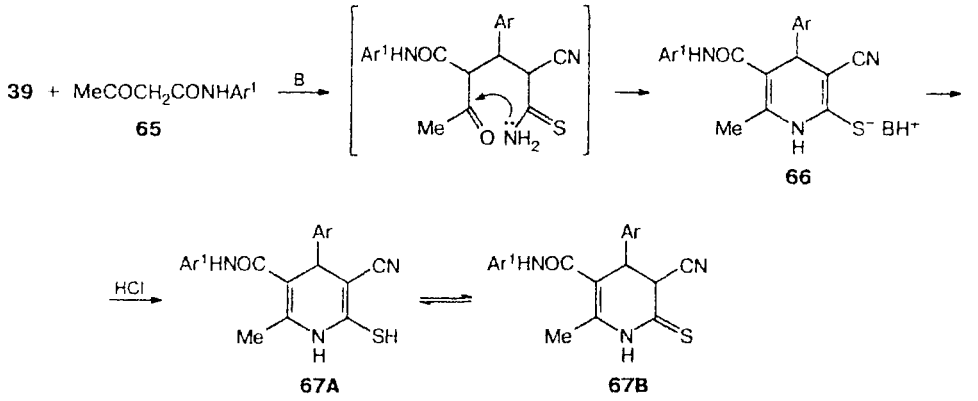


Scheme 18



The reactions of aryl(hetaryl)idenecyanothioacetamides (**39**) with acetoacetanilides **65** can also be assigned to the type of reactions under discussion. Thus owing to the presence of an activated double bond, amides **39** readily enter into the Michael reaction with anilides **65** to give substituted 1,4-dihydropyridine-2-thiolates (**66**); on treatment with hydrochloric acid, these products are converted into thiols **67**, which exist

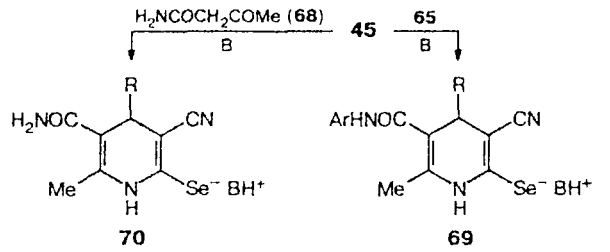
Scheme 19



in DMSO solutions as mixtures of tautomers **67A** and **67B** (Scheme 19).[21,42]

Condensation of hetarylidenecyanoselenoacetamides (**45**) with acetoacetanilide (**65**) or acetoacetamide (**68**) also occurs regioselectively and yields the corresponding 1,4-dihydropyridine-2-selenolates **69** or **70**.[21,43]

Scheme 20

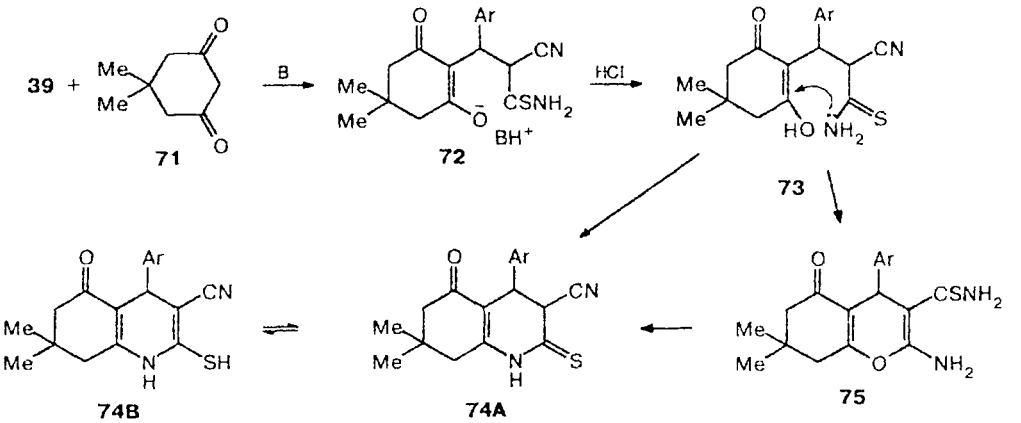


The introduction of cyclic 1,3-dicarbonyl compound, dimedone (**71**), into the reaction with arylidenecyanothioacetamides (**39**) has revealed some aspects of its mechanism.[19b,21,37i,44] In particular, this made it possible to isolate the intermediate Michael adducts as stable salts **72** and to study some of their properties. Acidification of salts **72** gives rise to adducts **73**; heating of these products (or directly salts **72** with subsequent acidification) in ethanol in the presence of an organic base affords dihydropyridine-2-thione derivatives (**74**). The use of dimedone (**71**) in the reactions with amides **39** made it possible to isolate a large number of intermediate compounds, which are ultimately converted into more stable products, thiones **74**.[44] It should also be mentioned that in a nonpolar solvent (benzene) in the presence of triethylamine, adducts **73** are transformed into substituted 5,6,7,8-tetrahydro-4*H*-benzo[*b*]pyrans (**75**), which are kinetically controlled products, and in a polar solvent (ethanol) they are converted into thermodynamically controlled dihydropyridine-2-thiones **74A** occurring in a tautomeric equilibrium with the thiol form **74B** (Scheme 21).

Similar reactions of dimedone (**71**) and its analogs with arylidenemalononitriles or arylidenecyanoacetic esters result in the formation of stable tetrahydro-4*H*-benzo[*b*]pyrans **75**.[45]

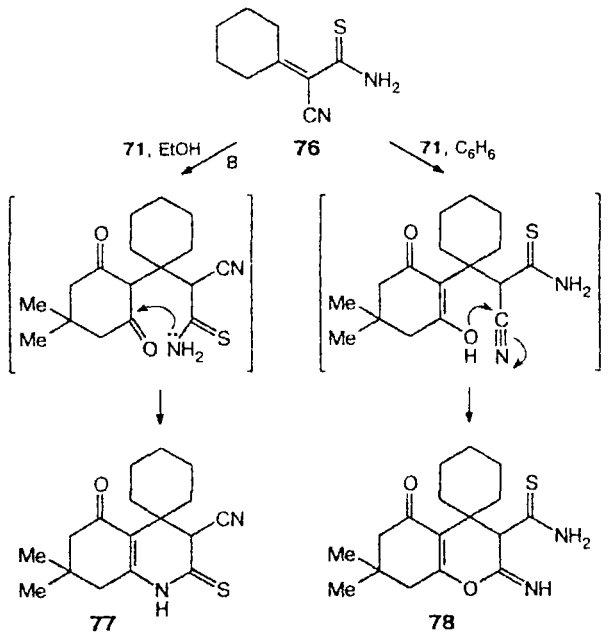
Functionally substituted 3,4,5,6,7,8-hexahydroquinoline-4-spiro-1′-cyclohexane-2(1*H*)-thione (**77**) was obtained in 68% yield by the reaction of dimedone (**71**)

Scheme 21



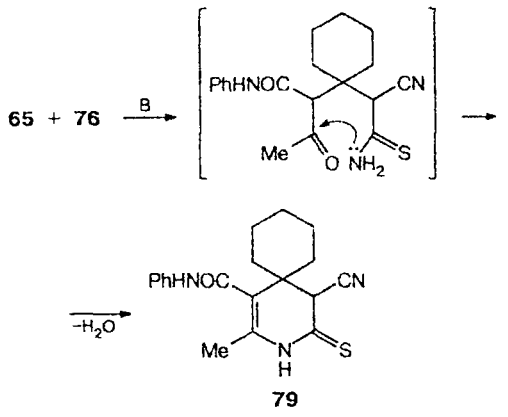
with cyclohexylidenecyanothioacetamide (**76**) in the presence of morpholine in ethanol.[21,46] The use of benzene as the solvent changes the reaction route; in this case, 2-iminopyran (**78**) is formed.

Scheme 22



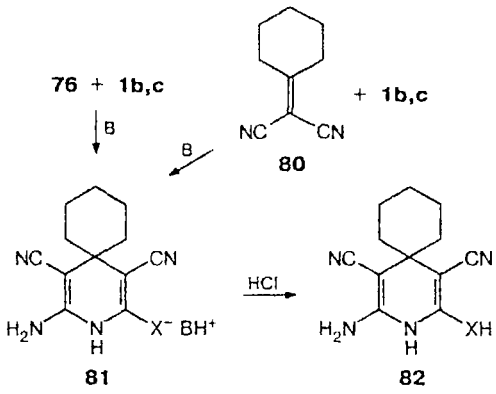
In turn, 3,4-dihydropyridine-4-spiro-1′-cyclohexane-2(1*H*)-thione (**79**) was synthesized in 87% yield by the reaction of amide **76** with acetoacetanilide (**65**) in ethanol in the presence of *N*-methylmorpholine[21,47].

Scheme 23



Spiro-fused partially hydrogenated pyridine-2-thiolate(selenolates) have also been prepared using some other reagents. Thus the reaction of cyclohexylidenemalononitrile (**80**) or cyclohexylidenecyanothioacetamide (**76**) with cyanoselenoacetamide (**1c**) and, correspondingly, the reaction of amide **76** with cyanothioacetamide (**1b**) or malononitrile in ethanol in the presence of *N*-methylmorpholine give salts **81**, which are converted into 6-amino-3,5-dicyano-1,4-dihydropyridine-4-spiro-1′-cyclohexane-2-thiolate (-selenolate) (**82**) by treatment with hydrochloric acid.[21,48–50]

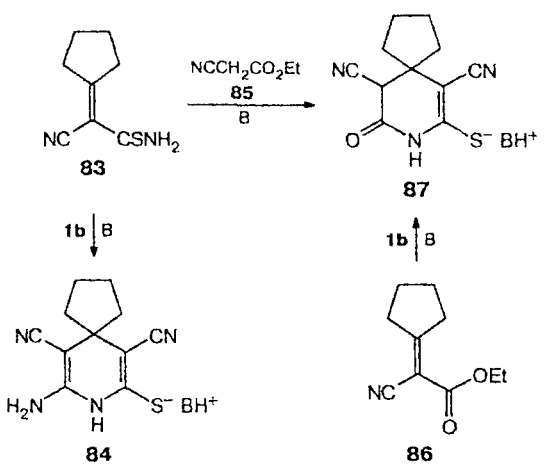
Scheme 24



X = S, Se

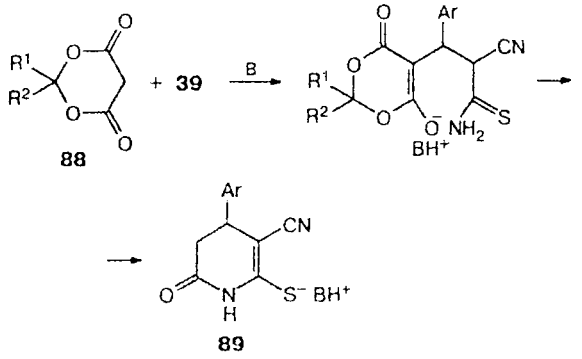
The reaction of cyclopentylidenecyanothioacetamide (**83**) with cyanothioacetamide (**1b**) carried out in a similar way resulted in the synthesis of 6-amino-3,5-dicyano-1,4-dihydropyridine-4-spiro-1′-cyclopentane-2-thiolate (**84**),[21,51] and the reactions of amide **83** with ethyl cyanoacetate (**85**) or amide **1b** with ethyl cyclohexylidenecyanoacetate (**86**) gave 3,5-dicyano-6-oxo-1,4,5,6-tetrahydropyridine-4-spiro-1′-cyclopentane-2-thiolate (**87**).[21,52]

Scheme 25



The use of Meldrum's acid (**88**) as a cyclic 1,3-dicarbonyl compound in the reaction with arylidenecyanothioacetamides (**39**) occurring on refluxing in ethanol led to 4-aryl-3-cyano-6-oxo-1,4,5,6-tetrahydropyridine-2-thiolates (**89**) in a yield of up to 85%.[53] When this reaction was carried out in benzene, the yield of thiolates **89** decreased to 30%.

Scheme 26



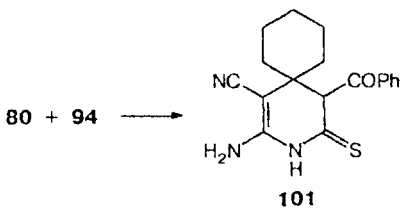
1.3. Syntheses based on α,β-unsaturated nitriles

Due to their high reactivity and accessibility,[7] α,β-unsaturated nitriles are widely employed in the synthesis of hydrogenated pyridinechalcogenones. A fairly large number of examples of using these compounds for this purpose are presented in the previous section, because arylidenecyanothioacetamides (**39**), 2-hetarylidenecyanoselenoacetamides (**45**), cyclohexylidenecyanothioacetamide (**76**), cyclohexylidenemalononitrile (**80**), cyclopentylidenecyanothioacetamide (**83**), and ethyl cyclopentylidenecyanoacetate (**86**), which belong to the class of α,β-unsaturated nitriles, have been employed as the CH acid components in the synthesis of hydrogenated pyridinechalcogenones based on 1,3-dicarbonyl compounds.

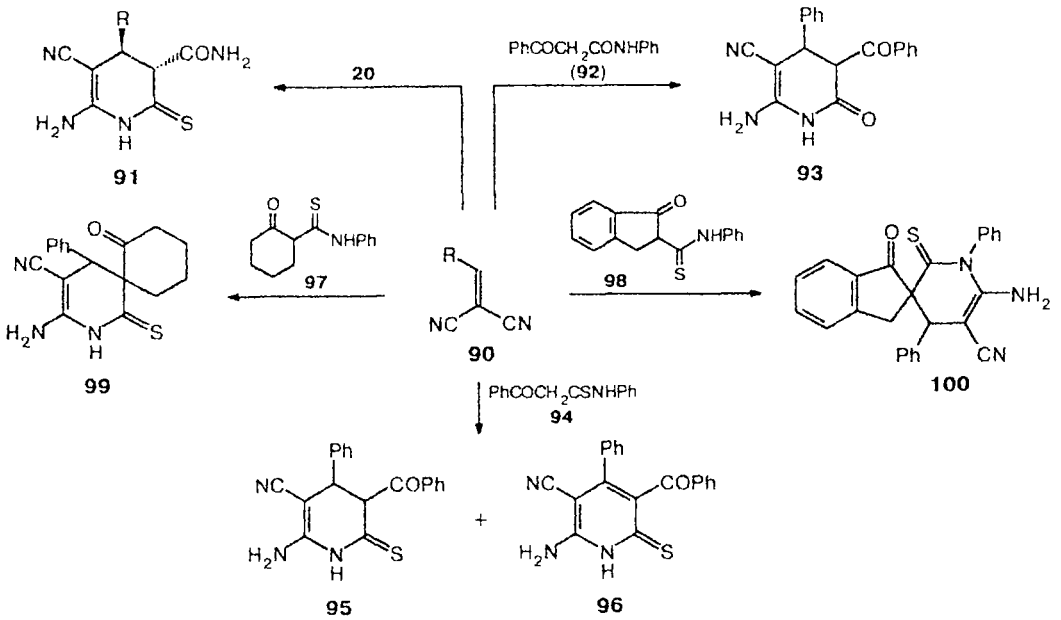
As other examples, it should be noted that the regioselective reaction of thiocarbamoylacetamide (**20**) with dinitriles **90** affords *trans*-3,4-dihydropyridine-2(1*H*)-thiones (**91**),[54a,b] and when *N*-phenylbenzoylacetamide (**92**) is used in this reaction as the CH acid, dihydropyridone **93** is formed.[54a] *N*-Phenylbenzoylthioacetamide (**94**) reacts in a similar way, although in this case pyridinethione **96** is formed in minor amounts, in addition to dihydropyridinethione **95**.[55] In turn, 2-oxocyclohexanethiocarbanilide (**97**) or 1-oxoindane-2-thiocarbanilide (**98**) react with benzalmalononitrile (**90**, R = Ph) to give spiro-fused pyridinethiones **99** and **100**, respectively (Scheme 27).[56]

Spiro-fused pyridinethione **101** was prepared by the reaction of nitrile **80** with anilide **94**.[57]

Scheme 28

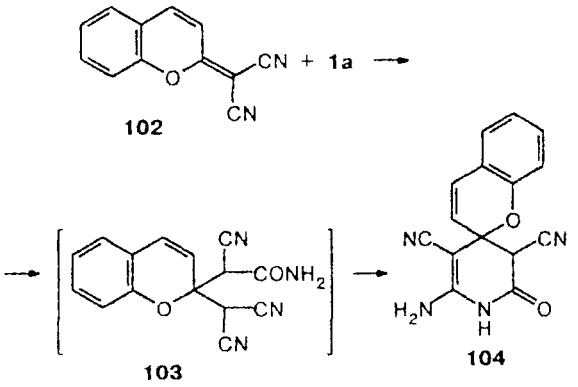


Scheme 27



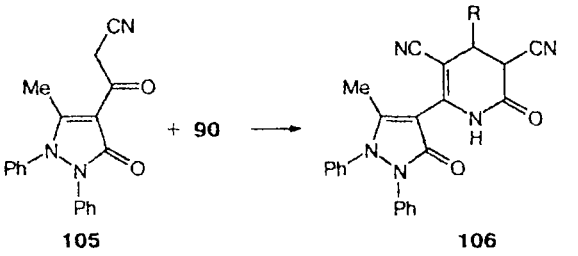
On refluxing in ethanol in the presence of piperidine, cyanoacetamide (**1a**) adds to cumarinylidenemalononitrile (**102**) to give adduct **103**, which undergoes intramolecular cyclization *in situ* yielding a spiro-fused pyridone **104**.[58]

Scheme 29



The reaction of pyrazolinone **105** with nitrile **90** afforded substituted 3,4-dihydropyridine-2(1*H*)-ones (**106**) in 70—75% yield.[59]

Scheme 30

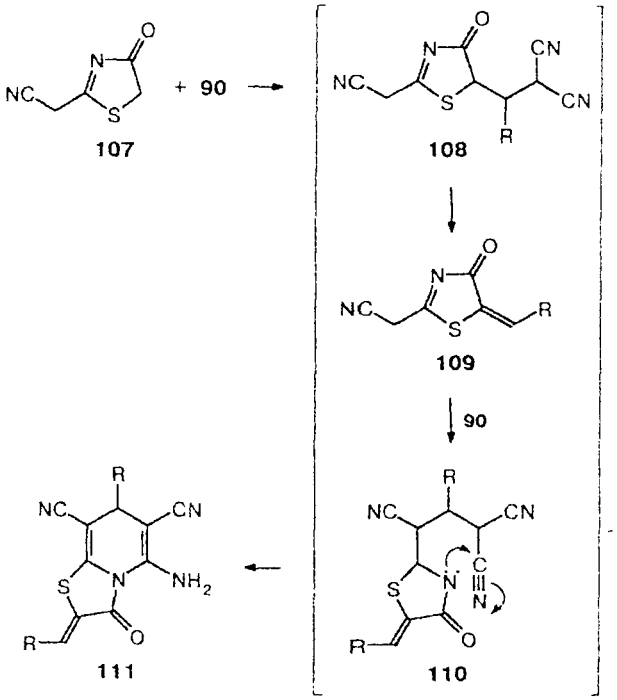


The interaction of nitriles **90** with 2-cyanomethylthiazol-4-one (**107**) follows an interesting pathway, which includes several steps.[60] First, Michael addition occurs giving rise to adducts **108**, which are transformed into thiazolones **109** upon elimination of malononitrile. Products **109** react with nitrile **90** to give adducts **110**, which undergo intramolecular cyclization to thiazolo[3,2-*a*]pyridines (**111**). (Scheme 31).

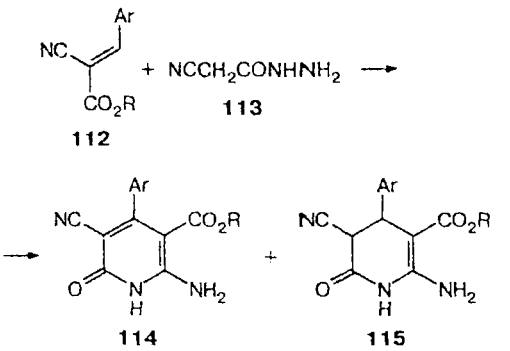
Arylidenecyanoacetates (**112**) react with cyanoacetohydrazide (**113**) on refluxing in ethanol in the presence of triethylamine or piperidine to give pyridones **114** in 80—83% yield;[61] the same reaction at room temperature affords a mixture of products, which contains, in addition to pyridones **114**, 3,4-dihydropyridin-2(1*H*)-ones (**115**) (Scheme 32).[62—64]

Substituted pyridone **118**, which has cardiovascular activity is formed in 52% yield upon nucleophilic addition of enamine of ethyl acetoacetate (**116**) to nitrile **117** in boiling methanol (Scheme 33).[65]

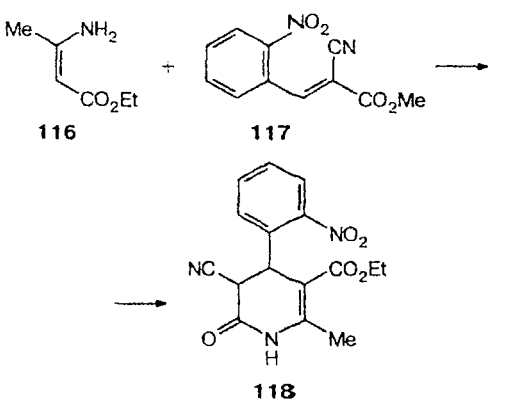
Scheme 31



Scheme 32



Scheme 33



1.4. Other methods of synthesis

Among other methods used to prepare partially hydrogenated pyridinechalcogenones, we would like to mention some procedures proposed for the first time in our studies, namely, three-component condensation, reactions with pyridinium ylides, and recyclization of eaminonitriles of the heterocyclic series.

1.4.1. Three-component condensation

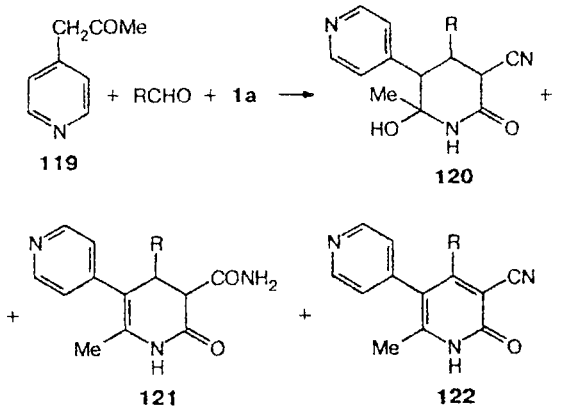
Three-component condensation, which has been used successfully in the synthesis of substituted 3-cyanopyridine-2(1*H*)-thiones,[1] proved to be also quite promising in the synthesis of hydrogenated analogs. This method consists of the reaction of aliphatic, aromatic, or heteroaromatic aldehydes with 1,3-diketones or acetoacetanilides and cyanothioacetamide (**1b**). It is significant that this method does not require a preliminary step of preparation of arylidenecyanothioacetamides (**39**).[19b,20,21,66–70]

Thus condensation of an aromatic or heteroaromatic aldehyde, cyanothioacetamide (**1b**), and acetoacetanilide (**65**) in anhydrous ethanol in the presence of *N*-methylmorpholine at room temperature resulted in the synthesis of 4-aryl(hetaryl)-3-cyano-6-methyl-1,4-dihydropyridine-2-thiolates (**67**) in 77–89% yield.[66–68]

Similar reaction of aliphatic aldehydes, amide **1b**, and dimedone (**71**) results in the formation of thiones **74** in a high yield,[69] and in the case of aromatic or heteroaromatic aldehydes, amide **1b**, and Meldrum's acid (**88**), the corresponding thiolates **89** are produced.[70]

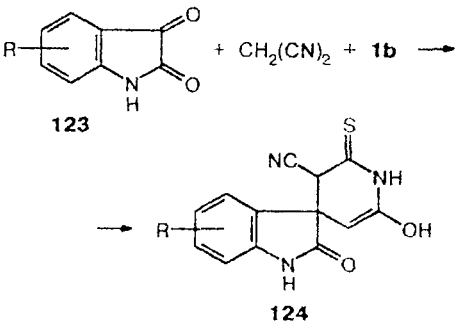
Three-component condensation of 4-pyridylacetone (**119**), cyanoacetamide (**1a**), and aromatic or heteroaromatic aldehyde gives rise to a mixture of pyridones **120**–**122**, which were separated by recrystallization.[71]

Scheme 34



Of other examples of using the three-component condensation in the synthesis of hydrogenated pyridinechalcogenones, note the interaction of diketone **123**, cyanothioacetamide (**1b**), and malononitrile in ethanol in the presence of piperidine, which yields spiro-fused pyridinethione **124**.[72]

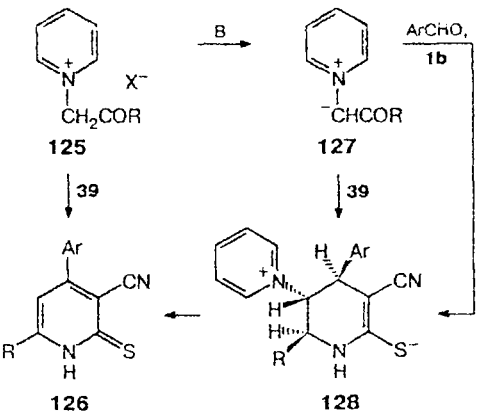
Scheme 35



1.4.2. Pyridinium ylides in the synthesis of 1hydrogenated pyridinechalcogenones

Pyridinium ylides generated *in situ* from quaternized pyridines are used advantageously in fine organic synthesis;[73] in particular, they are used for the preparation of 4,6-disubstituted 3-cyanopyridine-2(1*H*)-thiones.[73d,74–82] The reaction conditions were found to influence the nature of the products formed. Thus heating of arylidenecyanothioacetamides (**39**) with *N*-phenacylpyridinium salts (**125**) in the presence of ammonium acetate in acetic acid gives 4,6-diaryl-3-cyanopyridine-2(1*H*)-thiones (**126**),[75] whereas the reaction of pyridinium ylides **127**, generated from salts **125**, with amides **39** performed in ethanol in the presence of triethylamine is highly regio- and stereoselective and affords *trans,trans*-1,4,5,6-tetrahydropyridine-2-thiolates (**128**), which were isolated and characterized as stable crystalline salts.[74–77,79] They are converted into thiones

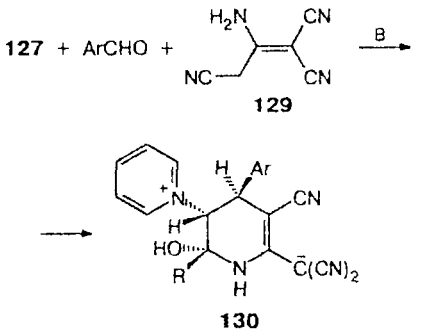
Scheme 36



126 on heating in acetic acid in the presence of ammonium acetate. Thiolates **128** have also been synthesized without preliminary preparation of thioamides **39** by three-component condensation of pyridinium ylides **127**, cyanothioacetamide (**1b**), and aromatic aldehydes.[75,76,79]

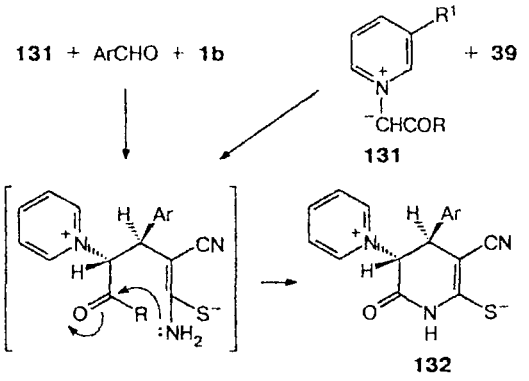
Stereoselective three-component condensation of pyridinium ylides **127**, aromatic aldehydes, and 2-amino-1,1,3-tricyanopropene (**129**) gave *trans,trans*-6-R-4-aryl-3-cyano-5-pyridinio-1,4,5,6-tetrahydropyridine-2-dicyanomethylides (**130**), which are difficult to obtain by other methods.[74,75]

Scheme 37



The reactions of pyridinium ylides **131** with amides **39** occur regio- and stereoselectively and give *trans*-4-aryl-3-cyano-6-oxo-5-pyridinio-1,4,5,6-tetrahydropyridine-2-thiolates (**132**).[37r,75,81–83]

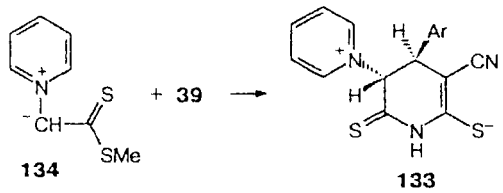
Scheme 38



Various pyridinium ylides have been used in the syntheses of analogous 4-aryl-3-cyano-6-oxo-5-pyridinio-1,4,5,6-tetrahydropyridin-2-olates.[75,81–85]

Substituted *trans*-6-thioxo-1,4,5,6-tetrahydropyridine-2-thiolates (**133**) were synthesized by the reaction of pyridinium ylide **134** with amides **39**.[37r,75,81,83]

Scheme 39



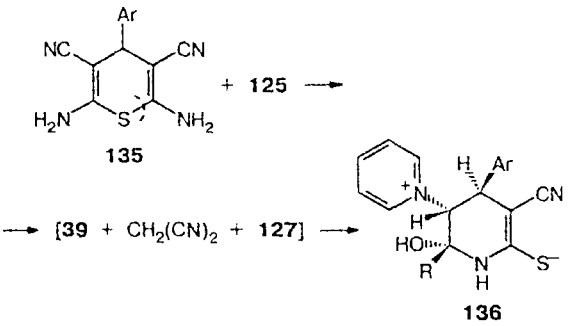
Isoquinolinium ylides[77] have also found application in the synthesis of thiolates **128**.

1.4.3. Recyclization of enaminonitriles of the heterocyclic series

Recyclization of enaminonitriles of six-membered heterocycles, *viz.*, 4*H*-pyran, 4*H*-thiopyran, 4*H*-selenopyran, and 1,3-dithiacyclohexene, has been used fairly widely in the synthesis of substituted 3-cyanopyridine-2(1*H*)-chalcogenones.[1,19b,20,37a,i,86] The intermediate formation of the corresponding partially hydrogenated pyridinechalcogenones was not merely postulated; in some cases,[87,88] these compounds were isolated as stable crystalline compounds.

In order to study the mechanism of recyclization of enamines of the 4*H*-thiopyran series, the structure of these compounds and their interaction with pyridinium ylides have been studied.[86j–n] Thus it was found that upon heating of a mixture of 4*H*-thiopyran **135** and pyridinium salt **125**, the thiopyran ring is cleaved, and further decomposition gives arylidenecyanothioacetamides (**39**) and malononitrile. The subsequent predominant interaction of the thioacetamides **39** thus formed with pyridinium ylides **127** (cross-recyclization) results in the formation of stable 1,4,5,6-tetrahydropyridine-2-thiolates (**136**).

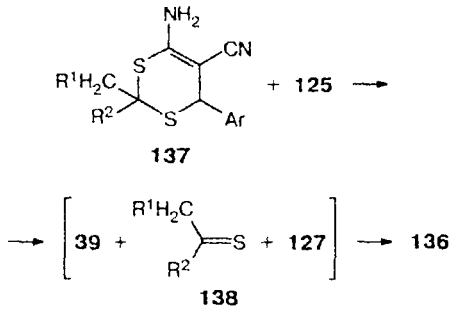
Scheme 40



The reaction of 1,3-dithiacyclohex-4-enes (**137**) with salt **125** follows a similar pathway resulting in the formation of unsaturated thioamides **39**, thioketones **138**, and pyridinium ylides **127**. The subsequent predominant interaction of ylides **127** with thioamides **39** (rather than

with thioketones **138**) occurs stereoselectively to give thiolates **136**.

Scheme 41



2. Chemical properties of partially hydrogenated pyridinechalcogenones

Study of the chemical properties of hydrogenated pyridinechalcogenones led ultimately to the development of methods for the synthesis of hitherto unknown partially hydrogenated functionally substituted annelated heterocyclic systems: furo(thieno, selenopheno)pyridines, pyrazolopyridines, pyridothienopyridines, and pyridothienopyrimidines, which potentially possess a wide range of biological activities.

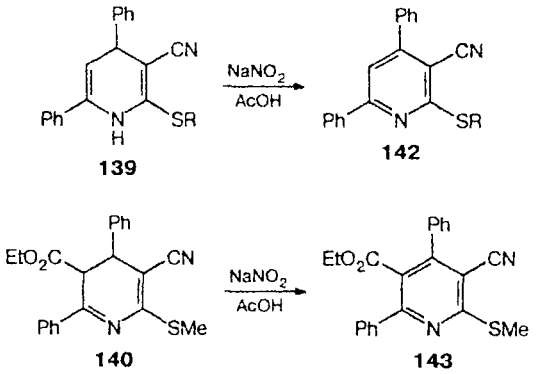
2.1. Dehydrogenation

The most typical feature of the chemical reactions of partially hydrogenated pyridinechalcogenones is the ease of dehydrogenation, which is indicated, in particular, by the large number of examples in which these species have been postulated as intermediates in the synthesis of 3-cyanopyridine-2(1*H*)-chalcogenones by the Michael reaction.[1,19b,20,21] In addition, it was found that stable salts **31**, **66** [20,21,43,66–68,87,88] and betaines **136** [75–78a,84,89,90] are dehydrated fairly easily on treatment with acids to give the corresponding aromatic pyridine-2(1*H*)-thiones. Oxidation of dihydropyridines **139** and **140** with sodium nitrite in acetic acid affords the corresponding aromatic analogs **142** and **143** (Scheme 42).[91,92]

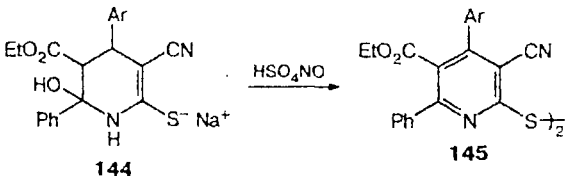
When salts **144** are treated with nitrosylsulfuric acid, oxidation of the thiolate anion yielding disulfides **145** occurs in parallel with dehydrogenation (Scheme 43).[22d]

Substituted piperidone **146** undergoes interesting transformations depending on the pH of the medium. Thus on treatment with hydrochloric acid, it is dehydrated to dihydropyridones **147**, and upon refluxing in an aqueous solution of KOH, it undergoes intramolecular cyclocondensation to give 2,6-diaza-1,8-diphenylbicyclo[2.2.2]octane-3,5-dione (**148**) (Scheme 44).[93]

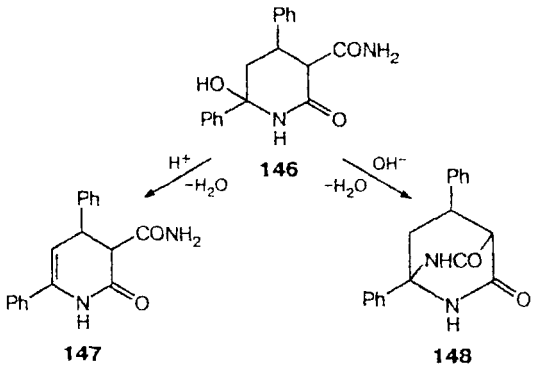
Scheme 42



Scheme 43



Scheme 44

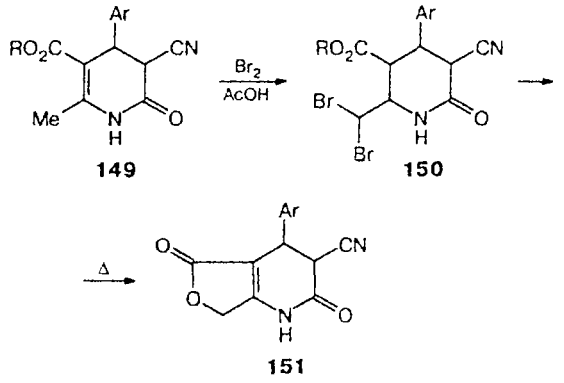


2.2. Halogenation

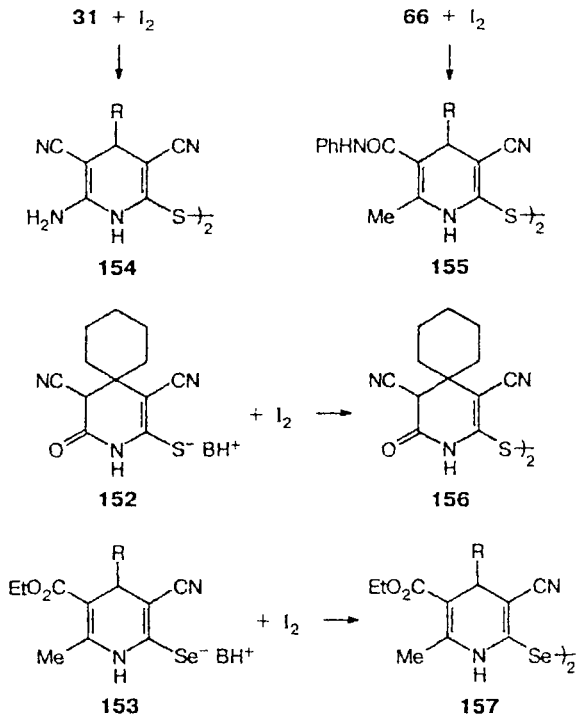
In a study of the reaction of substituted 3,4-dihydropyridin-2(1*H*)-ones (**149**) with bromine in acetic acid, it has been shown that this process yields dibromo derivatives **150**, which undergo intramolecular cyclization to furopyridines **151** at 170—180 °C (Scheme 45).[94]

However, thiolates **31** (X = S, R = 2-ClC_6H_4), **66** (Ar = Ph, R = 2-ClC_6H_4, 2-furyl), spiro-fused thiolate **152**, and selenolate **153** are oxidized by iodine in ethanol in the presence of an alkali to give the corresponding disulfides **154**—**156** or diselenide **157** in high yields, together with minor amounts of the products of their dehydrogenation (Scheme 46).[20,21,66,88]

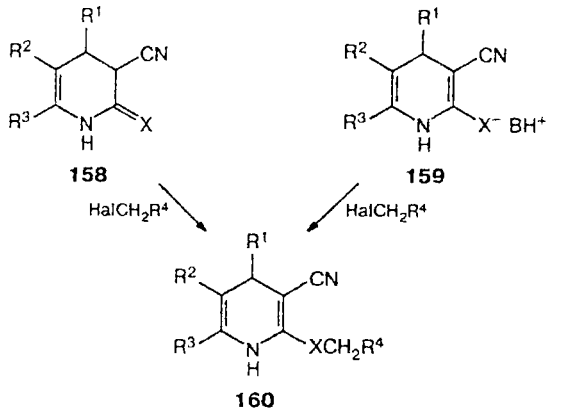
Scheme 45



Scheme 46



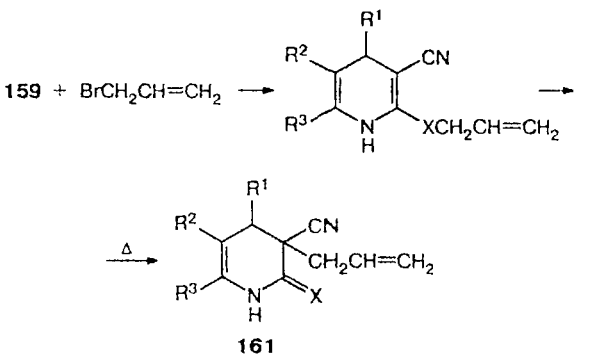
Scheme 47



X = S, Se

rangement of 2-allylthio(seleno)-1,4-dihydropyridines to 3-allyl-3,4-dihydropyridinechalcogenones (**161**);[19b,20,21,40,51,53,96] this rearrangement, unlike those of aromatic allyloxy(thio)pyridines and quinolines,[97—99] occurs regiospecifically and affords compounds **161** (in a yield of up to 98%).

Scheme 48



X = S, Se

2.3. Alkylation

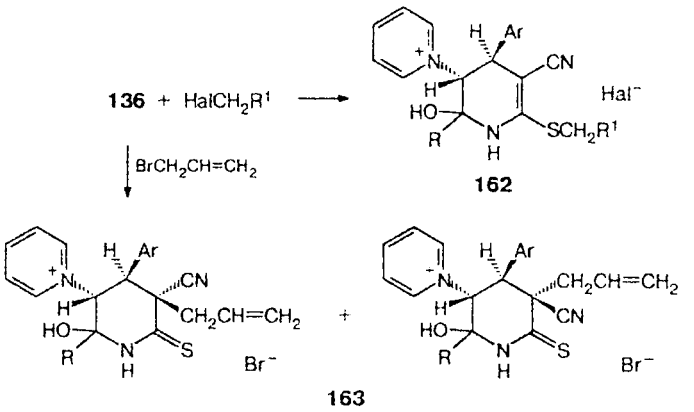
Alkylation of partially hydrogenated pyridine-2(1*H*)-thiones and -selenones has been studied in more detail. As in the case of their aromatic analogs, alkylation of 3,4-dihydropyridinechalcogenones (**158**) or salts **159** with alkyl halides (Scheme 47) occurs regioselectively to give 2-alkylthio(seleno)-1,4-dihydropyridines (**160**).[19b,20,21,37f,i,j,p,r,38,43,46,47,50—53,66—70,88,95]

The alkylation of salts **159** with allyl bromide was found to be accompanied by [3,3]-sigmatropic rearrangement of 2-allylthio(seleno)-1,4-dihydropyridines to 3-allyl-3,4-dihydropyridinechalcogenones (**161**);[19b,20,21,40,51,53,96] this rearrangement, unlike those of aromatic allyloxy(thio)pyridines and quinolines,[97—99] occurs regiospecifically and affords compounds **161** (in a yield of up to 98%).

The alkylation of betaines **136** in ethanol or DMF without additional bases present is also regioselective and gives 6-alkylthiotetrahydropyridines **162**.[37p,r] The alkylation of betaines **136** with allyl bromide in ethanol, heating of the reaction mixture, and prolonged keeping at room temperature result in the formation of the [3,3]-sigmatropic rearrangement product **163** as a mixture of two isomers (Scheme 49).

Study of the alkylation of thiolates **81**, **87**, and **152** with 1,2-dibromoethane in DMF at room temperature made it possible to develop a method for the preparation

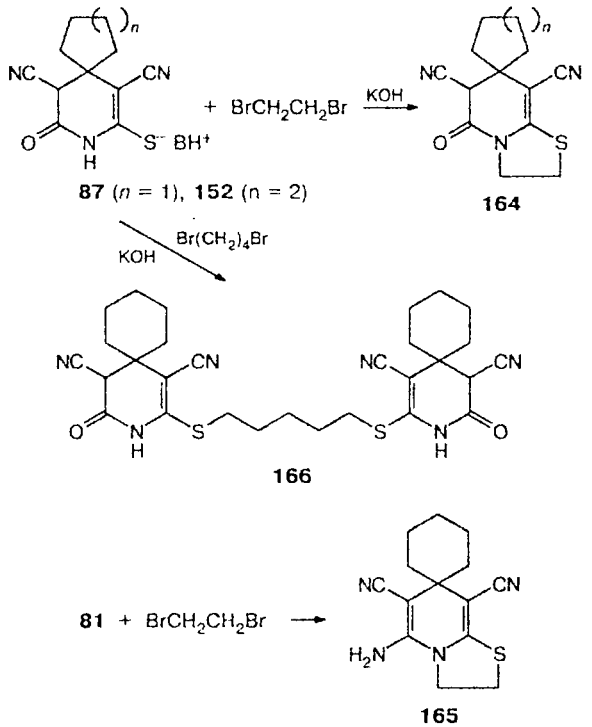
Scheme 49



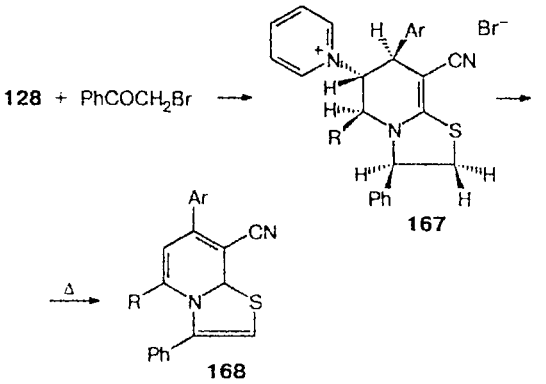
of previously unknown spiro-fused heterocyclic systems, *viz.*, 6,8-dicyano-5-oxo-2,3,6,7-tetrahydro-5*H*-thiazolo[3,2-*a*]pyridine-7-spiro-1′-cyclopentane and -cyclohexane (**164**) and 5-amino-7*H*-thiazolo[3,2-*a*]pyridine **165** having a similar structure.[20,21,49,52,100] A similar reaction of thiolate **152** with 1,4-bromobutane resulted in the formation of bis-sulfide **166**.

Thiazolo[3,2-*a*]pyridines have also been prepared from thiolates **128**. Thus heating a mixture of thiolate **128** and phenacyl bromide in ethanol to the boiling temperature yields 8-cyano-2,3,6,7-tetrahydro-5*H*-thiazolo[3,2-*a*]pyridine salts (**167**).[81] A more prolonged heating of the reaction mixture affords 8a*H*-thiazolo[3,2-*a*]pyridines (**168**).

Scheme 50

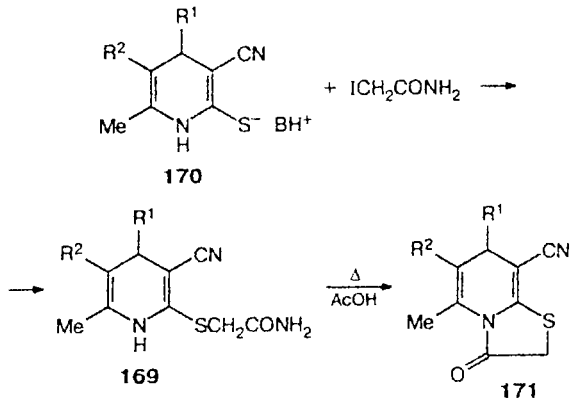


Scheme 51



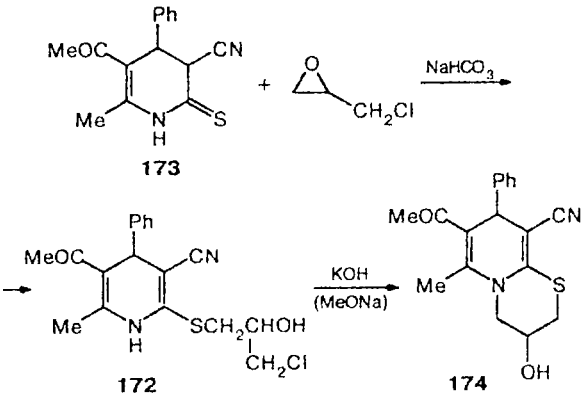
Sulfides **169** prepared by alkylation of salts **170** with iodoacetamide in ethanol undergo intramolecular cyclocondensation on refluxing in acetic acid to give 3-oxo-2,3-dihydro-7*H*-thiazolo[3,2-*a*]pyridines (**171**), whose yield depends on the nature of the substituent in position 4 of the dihydropyridine nucleus.[101] Thus in the presence of the pyridyl substituent, thiazolopyridines **171** can be obtained in 70—85% yields, while replacement of the pyridyl by an aryl group decreases the yield to 25%.

Scheme 52



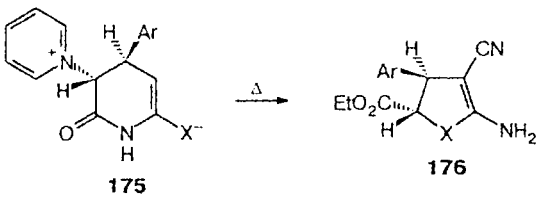
In turn, sulfide **172**, prepared by alkylation of thione **173** with epichlorohydrin, is converted into substituted pyridothiazine **174** on treatment with an alcoholic solution of KOH or MeONa.[102]

Scheme 53



An original pathway to functionally substituted hydrogenated furans and thiophenes, based on the use of 6-oxo-1,4,5,6-tetrahydropyridin-2-olates(thiolates) (**175**) as the substrates, has been developed. Thus on heating in ethanol, the starting tetrahydropyridines **175** undergo alkoholysis and recyclization to give *trans*-dihydrofurans and *trans*-dihydrothiophenes **176** in high yields (Scheme 54).[78a,82,103]

Scheme 54



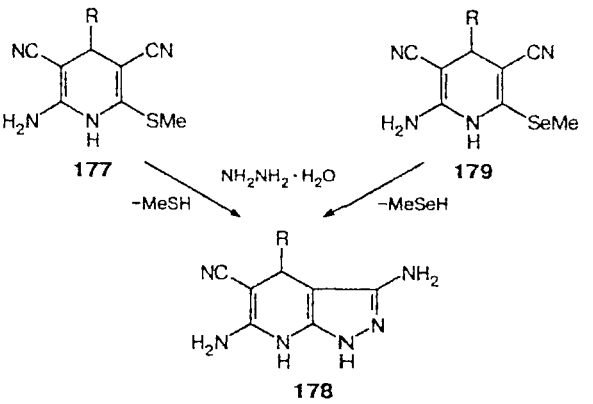
X = O, S

2.4. Cyclization

Cyclization of various vicinal derivatives of hydrogenated pyridinechalcogenones has been used successfully in the development of methods for the synthesis of previously unknown annelated heterocycles containing a partially hydrogenated pyridine ring.

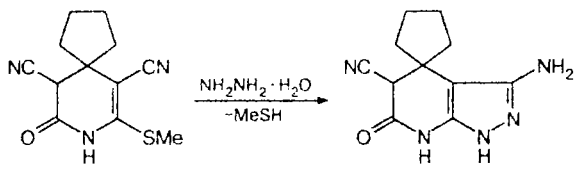
Among other properties, the ability of alkylthio groups in pyridinechalcogenones to enter into substitution reactions with nucleophiles has been used for this purpose.[20,21,104] Thus refluxing of 2-methylthio-1,4-dihydropyridine **177** with hydrazine hydrate in ethanol (40 min) gave 3,6-diamino-4-(2-chlorophenyl)-5-cyano-4,7-dihydro-1*H*-pyrazolo[3,4-*b*]pyridine (**178**) in 74% yield. This product is formed even more readily (refluxing for 10 min, yield 87%) from 2-methylseleno-1,4-dihydropyridine **179**.

Scheme 55



Under similar conditions, spiro-fused 2-(methylthio)tetrahydropyridine **180** is converted in 63% yield into 3-amino-5-cyano-6-oxo-4,5,6,7-tetrahydro-1*H*-pyrazolo[3,4-*b*]pyridine-4-spiro-1′-cyclopentane (**181**) (Scheme 56).

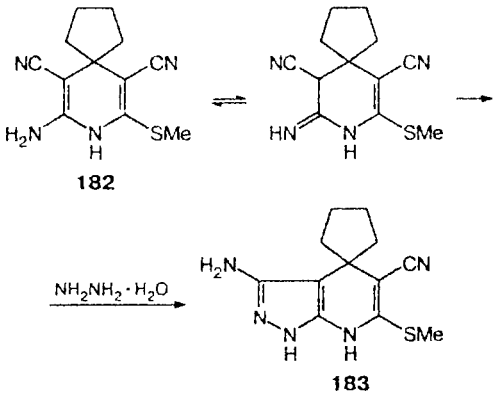
Scheme 56



180 181

However, in the case of 2-amino-3,5-dicyano-1,4-dihydropyridine-4-spiro-1′-cyclopentane (**182**), under the same conditions, 3-amino-5-cyano-6-methylthio-4,7-dihydro-1*H*-pyrazolo[3,4-*b*]pyridine (**183**) is produced.[20,21,51,52]

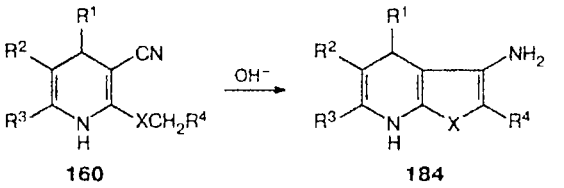
Scheme 57



182 183

Easily accessible functionally substituted 1,4-dihydropyridines **160** are remarkable substrates in the preparation of various 3-amino-4,7-dihydrothieno(selenopheno)[2,3-*b*]pyridines (**184**), which possess a great synthetic potential in the synthesis of biologically active compounds with a broad range of activity. For example, treatment of these substrates with alkali or alkali metal alkoxides in ethanol or DMF at room temperature or with short-term heating gives functionally substituted dihydrothienopyridines **184** in high yields.[19b,20,21,37a,f,i,j,r,43,47,49,53,67,68,70,88,95,100,105]

Scheme 58

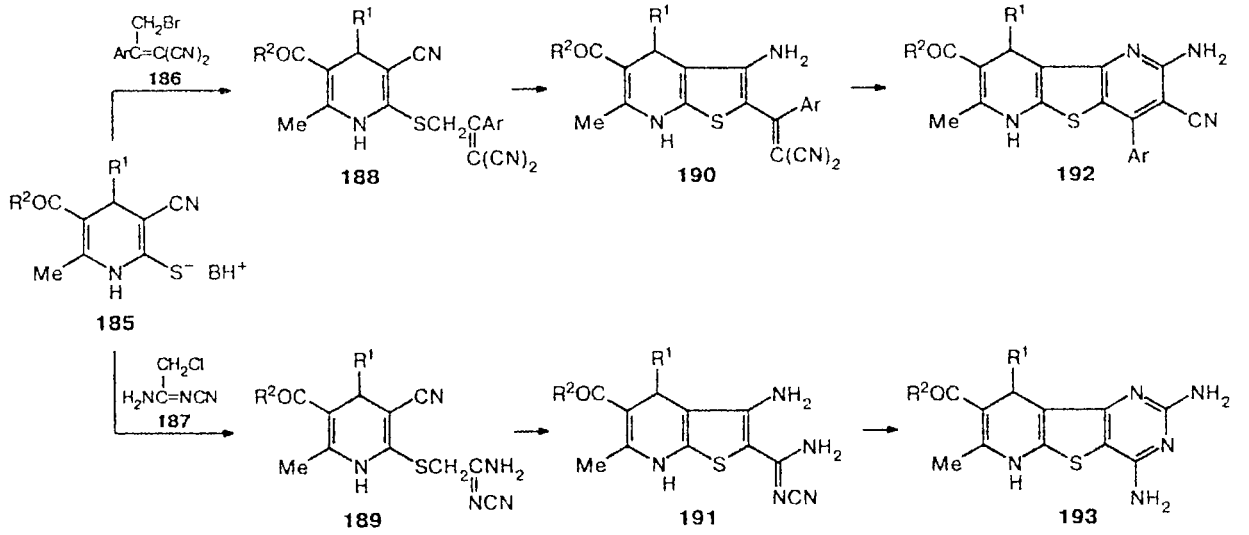


160 184

X = S, Se

In addition, this approach has also been used successfully in the synthesis of the first representatives of tricyclic systems containing a hydrogenated pyridine ring. Thus regioselective alkylation of 3-cyano-1,4-dihydropyridine-2-thiolates (**185**) with 2-bromo-1-(4-bromophenyl)ethylidenemalononitrile (**186**) or *N*-cyanochloroacetamidine (**187**) in ethanol gives rise to compounds **188** and **189**, which undergo cascade heterocyclization under the reaction conditions giving, first, thienopyridines **190** and **191** and then 6,9-dihydropyrido[2′,3′:4,5]thieno[2,3-*b*]pyridines (**192**) or 6,9-dihydropyrido[3′,2′:4,5]thieno[3,2-*d*]pyrimidines (**193**).[106]

Scheme 59



185 188 189 190 191 192 193

Conclusion

The analysis of the current state of the chemistry of partially hydrogenated pyridinechalcogenones presented here makes it possible to conclude that the development of this field of organic chemistry is highly promising, because not only regio- and stereoselective methods for their synthesis have been developed over the short period of time (the first systematic studies along this line were carried out in the middle 1980s), but also the structure and reactivity of these compounds have been studied. This made it possible to develop convenient methods for the preparation of various dihydro- and tetrahydropyridinechalcogenone derivatives and functionally substituted annelated heterocycles, containing a partially hydrogenated pyridine ring, based on them. The good prospects for the use of many of these compounds in the synthesis of new medical preparations are beyond doubt.

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