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ARYLCYCLOPROPANES IN THE SYNTHESIS OF NITROGEN- AND OXYGEN-CONTAINING HETEROCYCLES. (REVIEW)

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Methods for the production of nitrogen- and oxygen-containing heterocycles on the basis of the intra- and intermolecular reactions of functionally substituted arylcyclopropanes are discussed.

Keywords: 2-alkoxy-2-methyl(2-propionylphenylamino)indolinones, arylcyclopropanes, 1,4-benzodiazepinones, benzo[c]isoxazoles, 4H-3,1-benzoxazines, butyrolactones, 3,4-dihydroisocoumarins, 3,4-dihydroisoquinolin-1(2H)-ones, dihydroindoles, isoxazoles, isoxazolines, indazoles, indoles, 2-nitrosoacylbenzenes, phthalides, quinolines, intramolecular rearrangements.

Considerable attention has recently been paid to new methods for the synthesis of nitrogen- and oxygen-containing heterocycles, which are as a rule pharmacophoric fragments or natural biologically active organic compounds. In principle the development of new trends in this region of chemistry can result both from the creation of new schemes for the formation of heterocycles and from the synthesis of unique and readily obtainable starting compounds capable of certain paths of transformation into the desired nitrogen- and oxygen-containing heterocycles. In support of the foregoing it was demonstrated, for example, that relatively accessible functionally substituted arylcyclopropanes can be used successfully in the synthesis of widely differing nitrogen- and oxygen-containing heterocycles. Here, the indicated arylcyclopropanes are capable of being transformed into the corresponding heterocyclic compounds either directly or by successive transformations of the products from their initial reactions. The present review is devoted to analysis of the literature on this subject.

1. DIRECT TRANSFORMATION OF SUBSTITUTED ARYLCYCLOPROPANES INTO HETEROCYCLES

Data on the direct transformation of arylcyclopropanes into heterocyclic systems were first obtained in 1976 [1]. During an attempt at the electrophilic nitration of 1,2-diphenylcyclopropane the authors unexpectedly isolated 3,5-diphenylisoxazole (**3**) (R = R^1 = H) and its 4-nitro-substituted analog **4**. Here the geometry had hardly any effect on the direction of the reaction. Further investigations [2, 3] showed that this type of transformation was general in nature.

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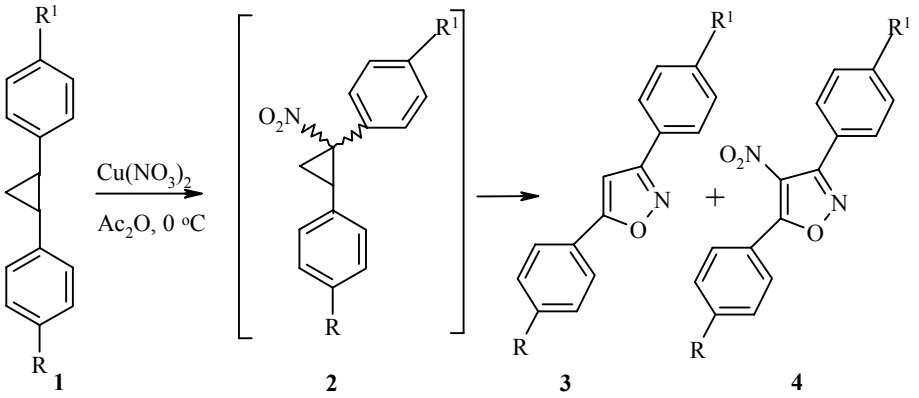


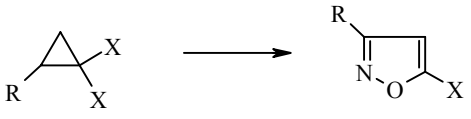
R = R^1 = H, Br, OMe; R = H, R^1 = OMe

During investigation of the reaction mechanism it was confirmed [2, 3] that nitrocyclopropanes of type **2**, formed in the initial stage of the reaction, were responsible for the formation of the isoxazoles **3** and **4**.

It is important to note that the transformation of arylated cyclopropanes by the action of copper nitrate in acetic anhydride is only realized for 1,2-diphenylcyclopropanes. For example, 2-methyl-1-phenylcyclopropane or 2-cyclohexyl-1-phenylcyclopropane is only nitrated in the benzene ring under these conditions [3].

The direct transformation of monoarylated cyclopropanes into isoxazoles likewise by the action of a nitrating agent was only achieved in 1992.

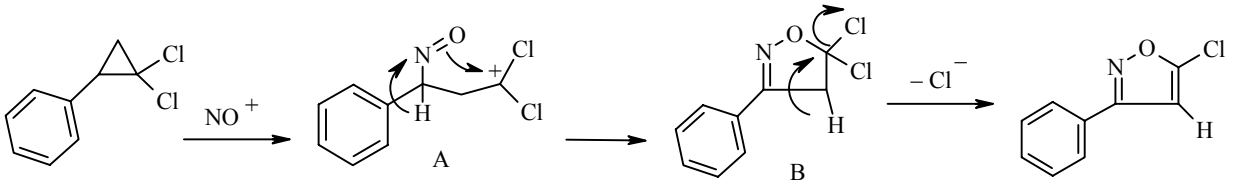
By the action of a nitrating mixture 1,1-dihalo-2-phenylcyclopropanes **5** containing strong electron-accepting substituents (NO_2, CN) in the aromatic ring are transformed into halogen-substituted isoxazoles **6** [4-6]. More recently the same investigators [7] found that the analogous reaction of 2-aryl-1,1-dihalocyclopropanes also gives high yields under the influence of nitrosonium fluoroborate in acetonitrile at 20°C.



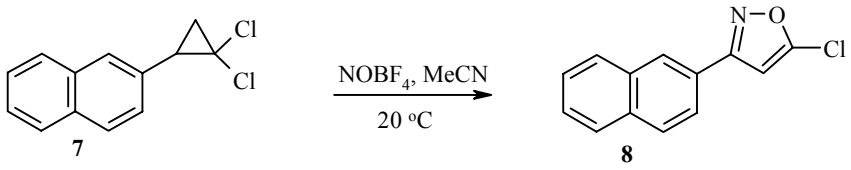
X = Cl, Br; R = Ph, 4-$O_2NC_6H_4$, 3-$O_2NC_6H_4$, 2-$O_2NC_6H_4$, 4-ClC_6H_4, 3-ClC_6H_4, 4-BrC_6H_4, 4-MeC_6H_4, 2-MeC_6H_4, 4-$MeOC_6H_4$, 3-$MeOC_6H_4$

It is important to emphasize that monosubstituted halogenocyclopropanes can also enter into this reaction. However, the yields of the desired substances are substantially lower and do not exceed 45% [7].

It is interesting that this reaction is initiated by the nitrosyl cation, which is a fairly weak electrophile. A deciding role in this transformation in relation to 1,1-dihalogenocyclopropanes that are weakly activated to electrophilic addition is clearly played by the high stability of the ion **A** and by the transformation, irreversible under the adopted conditions, of the intermediates (of type **B**) into the aromatic reaction product.

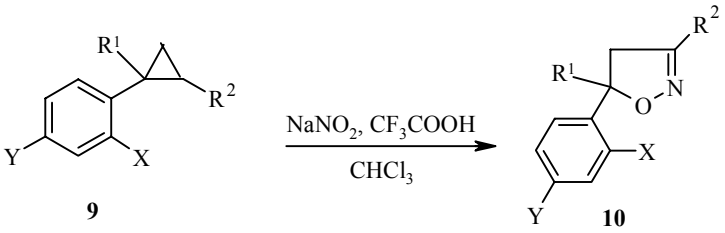


In the case of β-(2,2-dichlorocyclopropyl)naphthalene (**7**) the authors [7] showed that under the given conditions dihalogenocyclopropanes of the naphthalene series can be converted smoothly into the corresponding isoxazoles **8**.



7 **8**

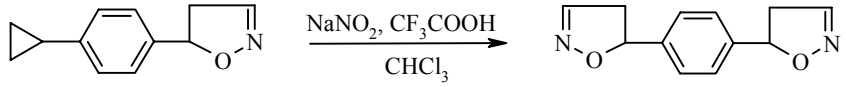
In 1928 [8] Shabarov and coworkers found that under the influence of nitrosyl cation phenyl- and diphenylcyclopropanes **9** are converted with high yields not into isoxazoles but into substituted isoxazolines **10**. Detailed investigation showed that the discovered transformation is general in nature and that arylcyclopropanes containing alkyl, aryl, or alkoxyl groups in the small ring and substituents of almost any kind in the aromatic ring readily form the corresponding isoxazolines [9-11].



9 **10**

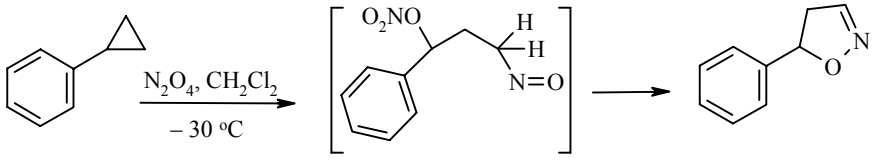
$R^1 = R^2 = H$; X = H; Y = H, Me, Pr-*cyclo*, OMe, NO_2, Br, Cl, I; Y = H, X = H, NO_2, Br, Cl, I;
R^1 = Me, R^2 = X = Y = H; R^2 = Ph, Me, OMe, R^1 = X = Y = H

It should be noted that the second three-carbon ring formed in the reaction of the oxazoline **10** (R = R^2 = X = H, Y = *cyclo*-Pr) is not transformed with the initially employed reagent ratio and is, therefore, only converted into the extremely difficultly obtainable compound **11** after repeated treatment of compound **10**.



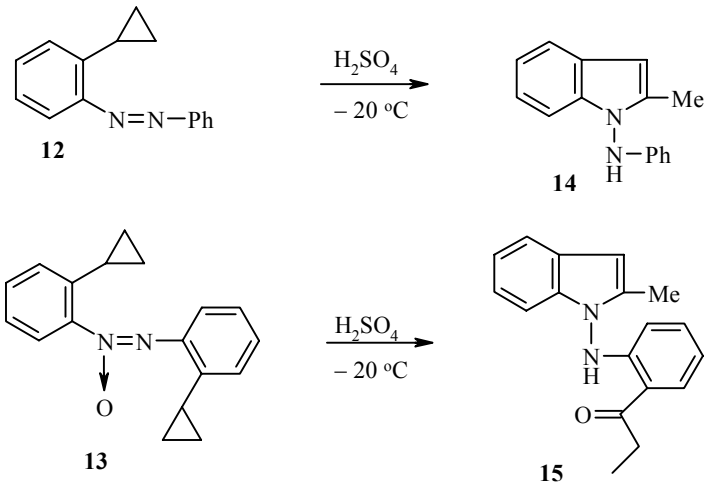
11, 76%

It is interesting that the reaction of substituted arylcyclopropanes with dinitrogen tetroxide in methylene chloride leads to similar results [12, 13]. The most remarkable in this transformation is the fact that dinitrogen tetroxide, which mostly reacts with unsaturated substrates in neutral or weakly polar solvents as a radical reagent, reacts with arylcyclopropanes as nitrosyl nitrate and nitrosyl cation and essentially initiates the transformation of the cyclopropane derivatives into isoxazolines [12, 13].

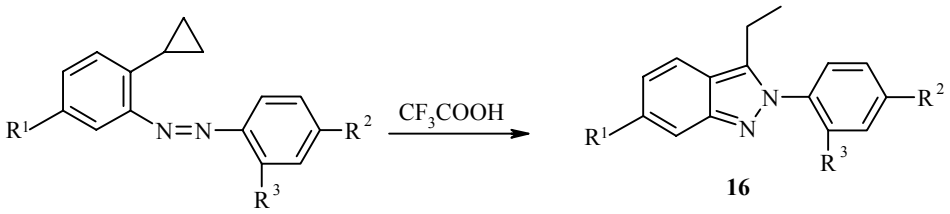


In the reaction of arylcyclopropanes with dinitrogen tetroxide, however, the yields of the targeted substances are somewhat lower. Moreover, this method of synthesis of isoxazolines cannot be used for substrates containing electron-withdrawing groups in the aromatic ring or substrates that readily undergo one-electron oxidation under the influence of the nitrosyl cation.

Up to now one-stage transformations of arylcyclopropanes into nitrogen- and oxygen-containing heterocycles in which the function was inserted into the heterocycle from outside have been considered. At the same time a large number of single-stage transformations in which the essential fragment for the construction of the heterocycle was already present in the initial substrate have been found in a series of arylcyclopropanes. Thus, the acid-catalyzed reactions of *ortho*-cyclopropyl-substituted azobenzenes **12** and azoxybenzenes **13** result in the formation of the respective indoles **14** and **15** [14].

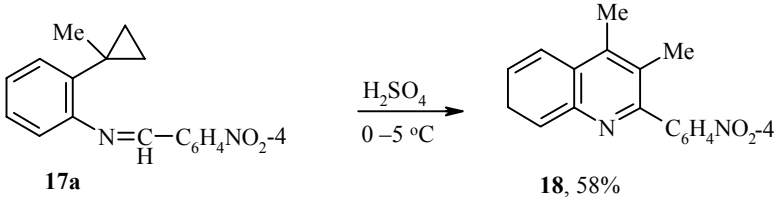


If, however, the reactions of *ortho*-cyclopropyl-substituted azobenzenes of type **12** are conducted under the influence of trifluoroacetic acid, the direction of heterocyclization changes, and the main products (yields 74-95%) are substituted indazoles **16** [15, 16].

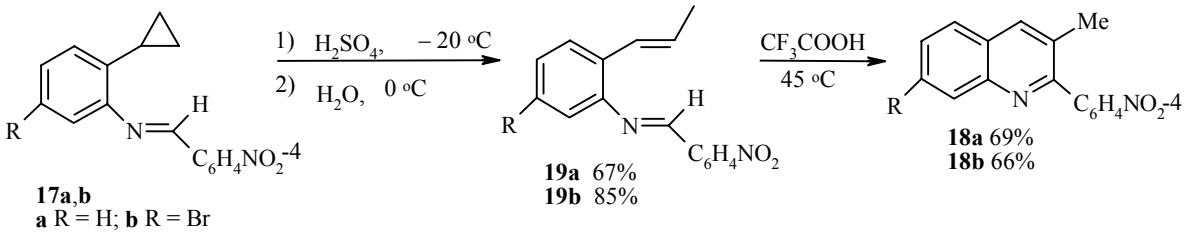


$R^1 = R^2 = H$, $R^3 = H$, Pr-*cyclo*; R^1 = *i*-PrC, Br, $R^2 = R^3 = H$;
$R^1 = R^3 = H$, R^2 = OMe, Cl

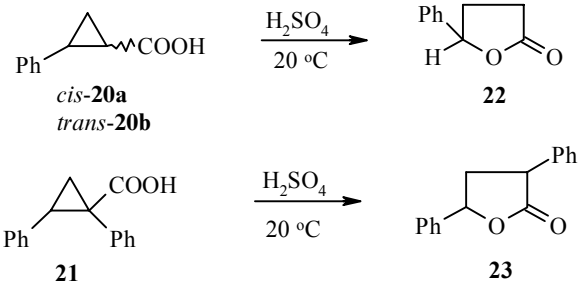
Under the influence of protic acids the isoelectronic analogs of azobenzenes – 2-cyclopropyl-substituted N-benzylideneanilines (of type **17**) – are converted into methyl-substituted quinolines **18**.



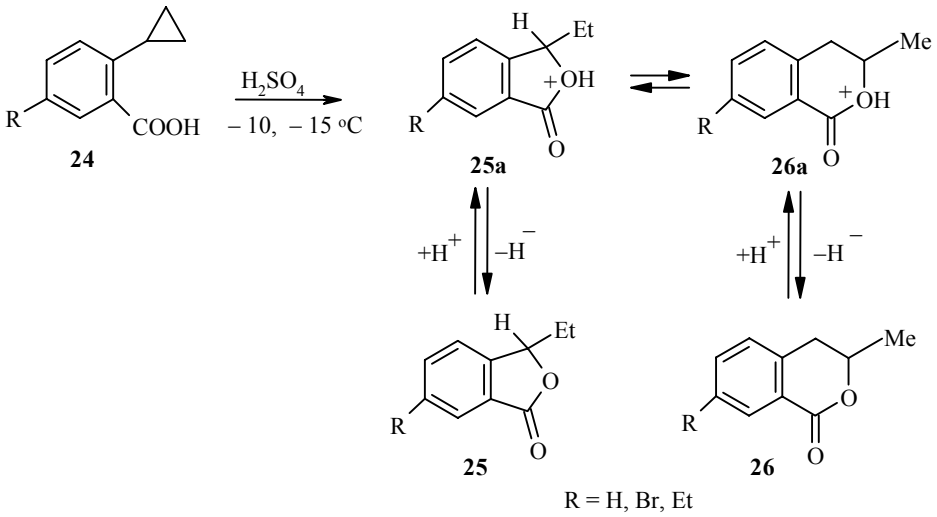
Under the influence of sulfuric acid the 2-(1-methylcyclopropyl)-substituted Schiff base **17** immediately forms the corresponding quinoline **18** [17], whereas the cyclopropane analogs not containing methyl groups in the small ring give satisfactory yields of the targeted substances only if the process is conducted in two separate stages, i.e., isomerization of the cyclopropane derivatives by the action of sulfuric acid to the respective propenyl-containing Schiff bases **19** and cyclization of the latter to the quinolines **18** by heating with polyphosphoric acid (or trifluoroacetic acid) [18].



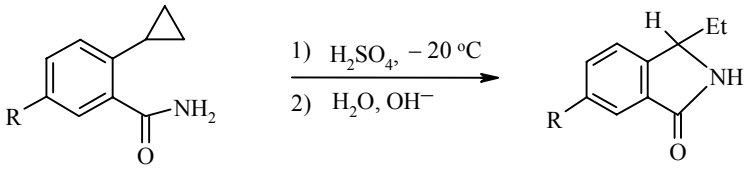
The mono- and diaryl-substituted cyclopropanecarboxylic acids **20** or **21** can be converted with high yields in a single stage into the corresponding butyrolactones **22** and **23** [19].



If, however, the carboxyl group is at the *ortho* position in the benzene ring of the substituted arylcyclopropanes **24**, the acid-catalyzed reactions of the latter can give either substituted phthalides **25** or 3,4-dihydroisocoumarins **26**, depending on the reaction time [20]. The compounds **25** and **26** obtained in the pure form can isomerize through the protonated forms **25a** and **26a**.



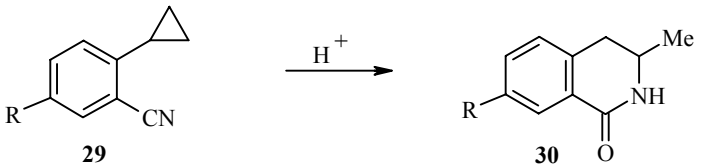
Under the same conditions the amides of *ortho*-cyclopropylbenzoic acids **27** isomerize with high yields to the 3-ethylphthalimidines **28** [21].



27a–c **28a–c**

27, **28** **a** R = H, **b** R = Br, **c** R = Et

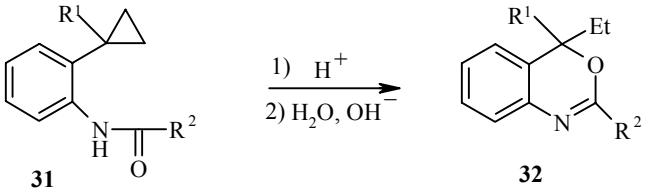
In contrast to the amides the corresponding nitriles of 2-cyclopropylbenzoic acids **29** are converted by the action of the same protic acid into 3-methyl-3,4-dihydroisoquinolin-1(2H)-ones **30** with yields of 70-82% [22].



29 **30**

R = H, Br, Et

Quite recently a new rearrangement was discovered in the series of functionally substituted arylcyclopropanes. By this rearrangement it is possible to transform N-acyl-2-cyclopropylanilines **31** in a single stage into difficultly obtainable 4H-3,1-benzoxazines **32** with yields of 74-91% [23].

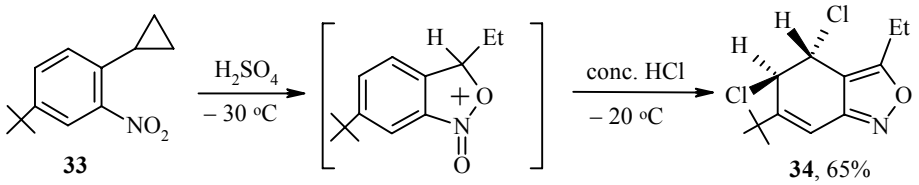


31 **32**

R^1 = H, R^2 = Me, Ph, 2-ClC_6H_4, 4-ClC_6H_4, 2-BrC_6H_4, 4-BrC_6H_4, 3-$MeOC_6H_4$, 4-$MeOC_6H_4$, 4-$O_2NC_6H_4$; R^1 = Me, R^2 = Me, Ph, 2-ClC_6H_4, 4-ClC_6H_4, 2-BrC_6H_4, 4-BrC_6H_4

The acid-catalyzed single-stage transformations of functionally substituted arylcyclopropanes examined above [14-23] are essentially the result of intramolecular interaction, arising from the three-carbon ring of a carbenium ion with *ortho* substituents exhibiting nucleophilic characteristics. The heterocyclic intermediates formed here as a rule do not undergo more profound transformations under the reaction conditions, and the nitrogen- or oxygen-containing heterocycles corresponding to them are isolated when the acidic solutions are neutralized.

However, more profound transformation of the intermediately formed heterocyclic intermediates is possible in principle, and the transformations of functionally substituted arylcyclopropanes, taking place as single-event processes, can lead to heterocycles with more complex structure [24].



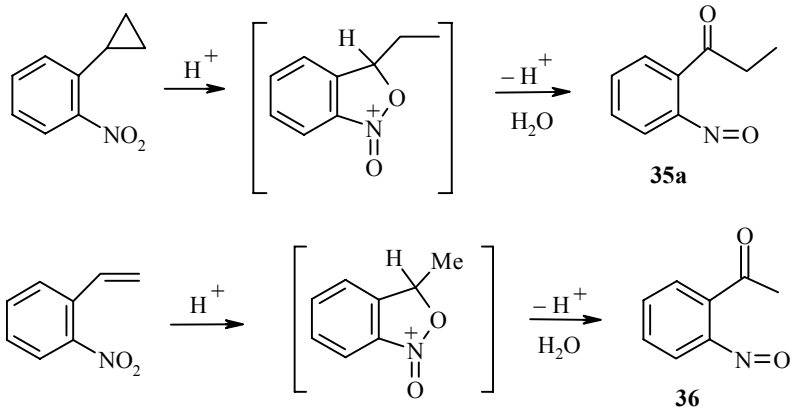
It is clear that trans-6-*tert*-butyl-4,5-dihydro-4,5-dichloro-3-ethyl-2,1-benzoxazole (**34**) is formed during of a series of successive stages, beginning with the formation of a heterocyclic ion from the initial 4-*tert*-butyl-2-nitrophenylcyclopropane (**33**) by the action of sulfuric acid.

2. SYNTHESIS OF NITROGEN- AND OXYGEN-CONTAINING HETEROCYCLES FROM ARYLCYCLOPROPANES AS A SERIES OF CONSECUTIVE INDEPENDENT REACTIONS

It is known that methods for the synthesis of nitrogen- and oxygen-containing heterocycles condensed with an aromatic ring are largely based on the use of functionally *ortho*-substituted benzenes and their analogs.

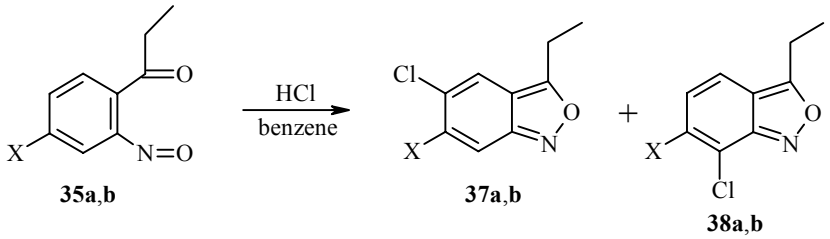
In particular, it was shown for individual examples that nitrogen- and oxygen-containing heterocycles of the most varied types can be synthesized from *ortho*-nitroacylbenzenes or *ortho*-nitrosoacylbenzenes. However, the synthetic possibilities of the *ortho*-substituted benzenes were restricted significantly on account of complications arising in their preparation. In actual fact, it is not a simple task to synthesize an *ortho*-substituted benzene containing two neighboring electron-withdrawing groups (a nitro group or nitroso group and an acyl fragment).

In 1969 it was found that *ortho*-nitrophenylcyclopropane or *ortho*-nitrostyrene is capable of being converted almost quantitatively by concentrated acids into the corresponding *ortho*-nitrosoacylbenzenes **35** and **36** [25].



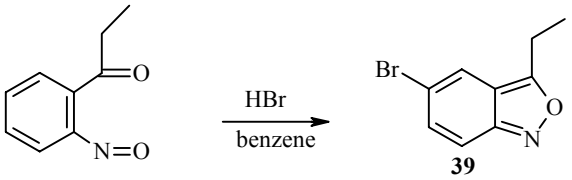
The comprehensively studied [26-34] intramolecular oxidation–reduction reaction opened up access to substrates that promised broad synthetic prospects for the synthesis of the corresponding nitrogen- and oxygen-containing heterocycles.

The first corroboration was obtained in 1973 [35, 36]. It was shown that the respective *ortho*-nitrosopropiophenones formed from 2-nitrophenylcyclopropanes are converted by the action of gaseous hydrogen chloride in benzene into 5- and 7-chloro-substituted 3-ethylbenzo[*c*]isoxazoles **37** and **38** with good yields.



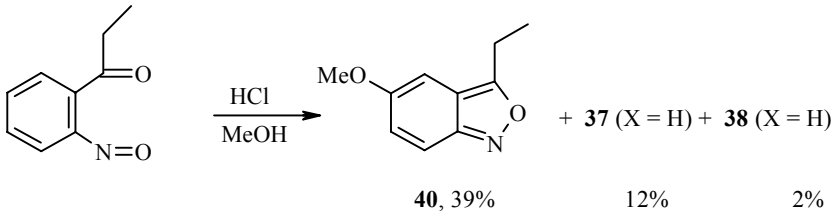
35, **37**, **38** **a** X = H, **b** X = Br

If, however, the reaction of the nitroso ketones is carried out in benzene under the influence of hydrogen bromide, a bromine atom is inserted into the benzo[*c*]isoxazole molecule [37].

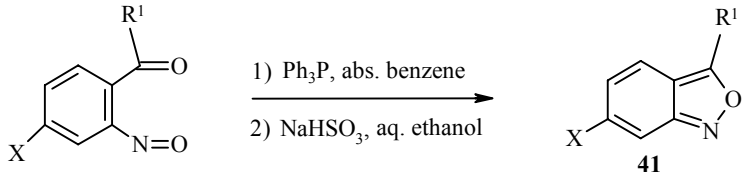


It is important to note that under the influence of gaseous hydrogen bromide the reaction takes place even without the formation of the isomeric 7-bromo derivative but is not nevertheless regioselective (a series of parallel processes is observed) as a consequence of intermolecular oxidation–reduction reactions.

It is interesting that if the acid-catalyzed reaction under the influence of gaseous hydrogen chloride is conducted in a nucleophilic solvent a fragment of the solvent can even be inserted into the targeted benzo[*c*]isoxazole [37].

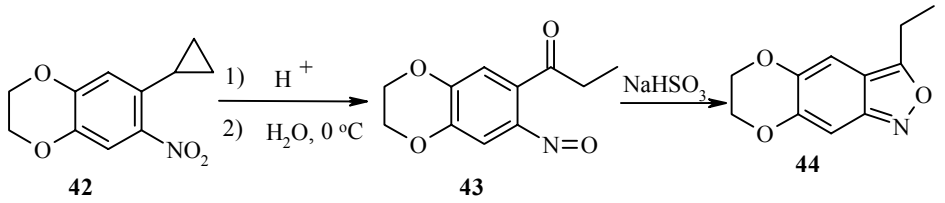


The synthesis of benzo[*c*]isoxazoles **41** from *ortho*-nitrosoacylbenzenes not accompanied by insertion of the nucleophilic group into the final reaction product can be realized by the action of deoxidizing reagents – triphenylphosphine in dry benzene or alcohol (yields 61-90%) [38] or sodium hydrosulfite in aqueous ethanol (yields 73-95%) [39, 40].



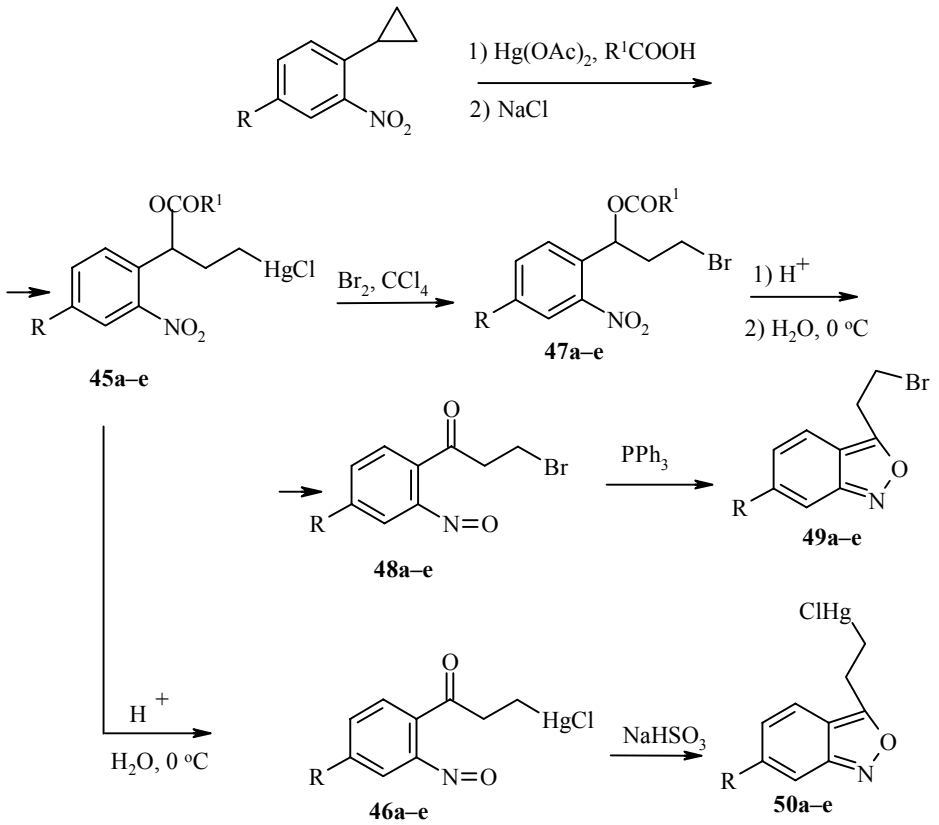
R^1 = Me, X = H; R^1 = Et, X = H, Alk, Pr-*cyclo*, Br, COMe, NO_2, 4-$O_2NC_6H_4$

Benzo[*c*]isoxazole **44** of the 1,4-benzodioxane series was obtained according to a similar scheme [34].



42 **43** **44**

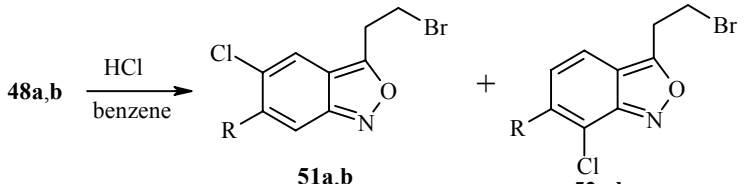
Benzo[*c*]isoxazoles **49** and **50**, functionalized not only in the benzene ring but also in the side chain, can be synthesized [33, 41] by a series of solvomercuration reactions in the arylcyclopropanes, transformation of the obtained adducts **45** into the corresponding *ortho*-substituted nitrosoacylbenzenes **46**, and deoxidation of the latter according to the familiar path [40].



45a–e **47a–e** **48a–e** **49a–e** **46a–e** **50a–e**

45–50 a R = *i*-Pr, **b** R = *t*-Bu, **c** R = Br, **d** R = COMe, **e** R = NO_2;
45, **47 a**, **b** R^1 = Me, **c**–**e** R^1 = H

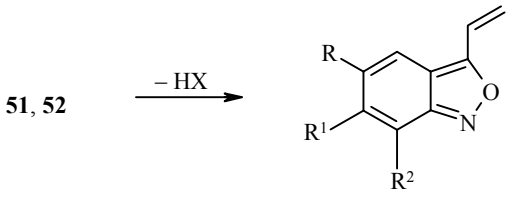
If, however, nitroso compounds of type **48** are brought into reaction with hydrogen chloride in dry benzene, as in the case of nitrosopropiophenones **35** [35, 36], the corresponding chlorine-substituted benzo[*c*]isoxazoles **51** and **52** are formed [42].



51a,**b** **52a**,**b**

a R = H, **b** R = Br

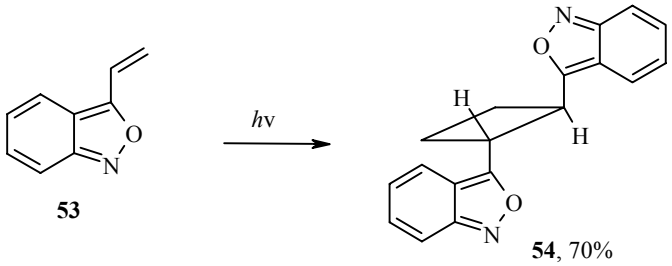
Here, as found, benzo[*c*]isoxazoles of type **51** and **52**, containing a β-haloethyl substituent at position 3, readily eliminate a molecule of HHal when their solutions are passed through a layer of aluminum oxide and form the difficultly obtainable 3-vinylbenzo[*c*]isoxazoles **53** with almost quantitative yields [43].



53

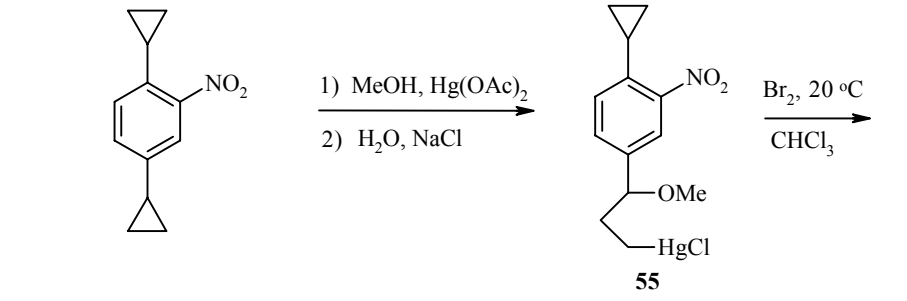
X = Br, Cl; R = R^1 = H, R^2 = H, Cl; X = Br; R = R^2 = H, R^1 = Br; R = H, R^1 = Br, R^2 = Cl; R = Cl, R^1 = R^2 = H; R = Cl, R^1 = Br, R^2 = H, X = Cl; R = R^1 = R^2 = H

In turn 3-vinylbenzo[*c*]isoxazoles **53** readily enter into [2+2] cycloaddition and form here cyclobutanes containing benzo[*c*]isoxazole fragments in the *trans* position [44].

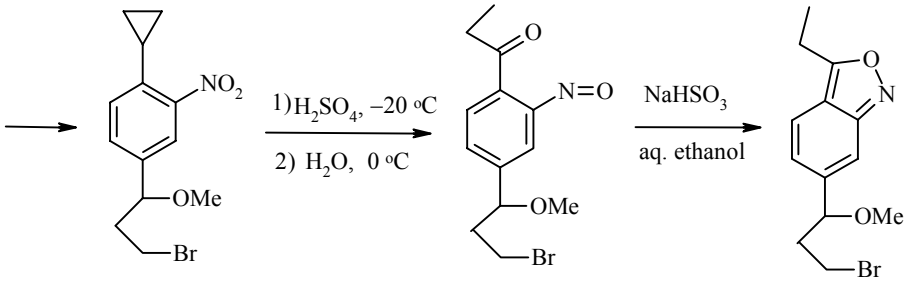


53 **54**, 70%

In acid-catalyzed with two cyclopropane fragments in the molecule the ability to open differs, and this is an interesting feature in the behavior of nitro-substituted phenylcyclopropanes. It makes it possible, for example, to synthesize polyfunctionally substituted benzo[*c*]isoxazoles according to the following scheme [45].

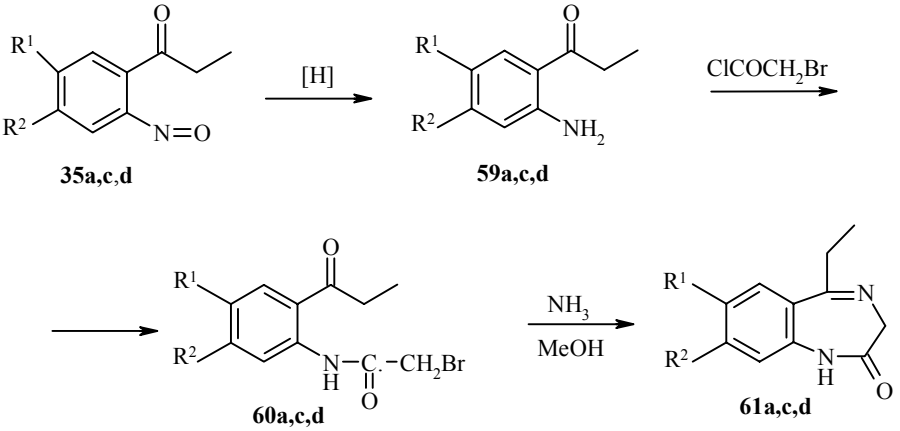


55



56 **57** **58**

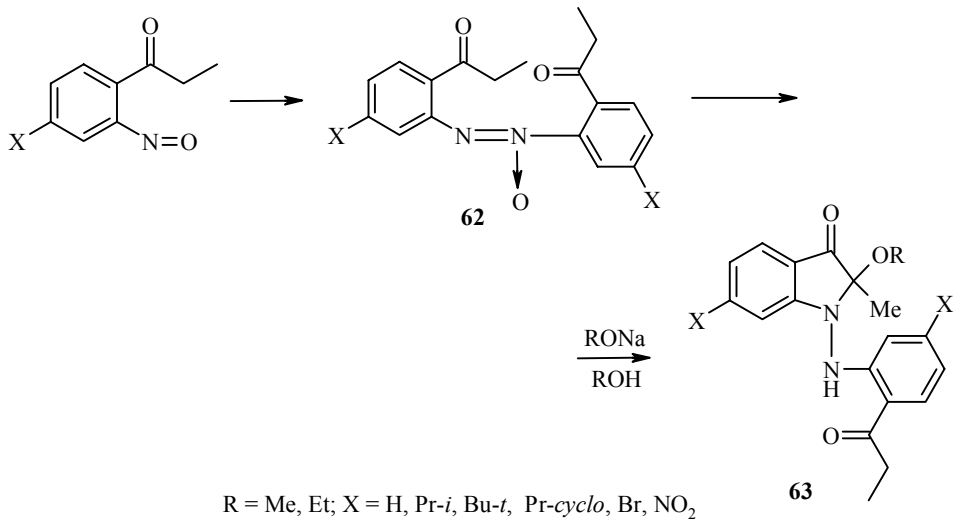
Nitrosoacylbenzenes, which are easily obtained by rearrangement of the corresponding *ortho*-substituted cyclopropylbenzenes, can also be used successfully for the production of 1,4-benzodiazepinones, the synthesis of which has until now been a crucial problem [46, 47].



35a,c,d **59a,c,d** **60a,c,d** **61a,c,d**

a $R^1 = R^2 = H$; **c** $R^1 = R^2 = –OCH_2CH_2O–$; **d** $R^1 = Cl$, $R^2 = H$

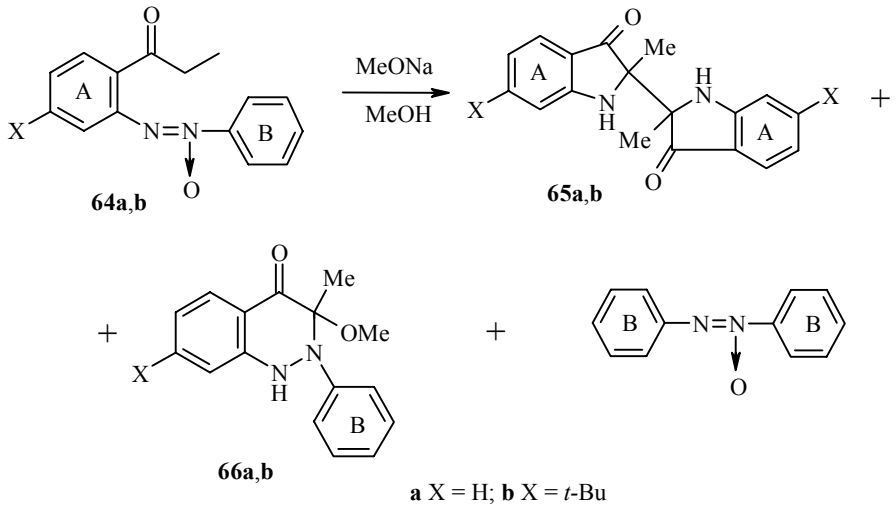
ortho-Nitrosopropiophenones **35** as products from the acid-catalyzed rearrangements of *ortho*-nitrophenylcyclopropanes can be used in the synthesis of heterocycles and, from the latter, the difficultly obtainable 2,2'-dipropionylazoxybenzenes **62**. The latter are converted with high yields into 2-alkoxy-2-methyl-N-(2-propionylphenylamino)indolinones **63** by the action of catalytic amounts of sodium alcoholates in the respective alcohols [48, 49].



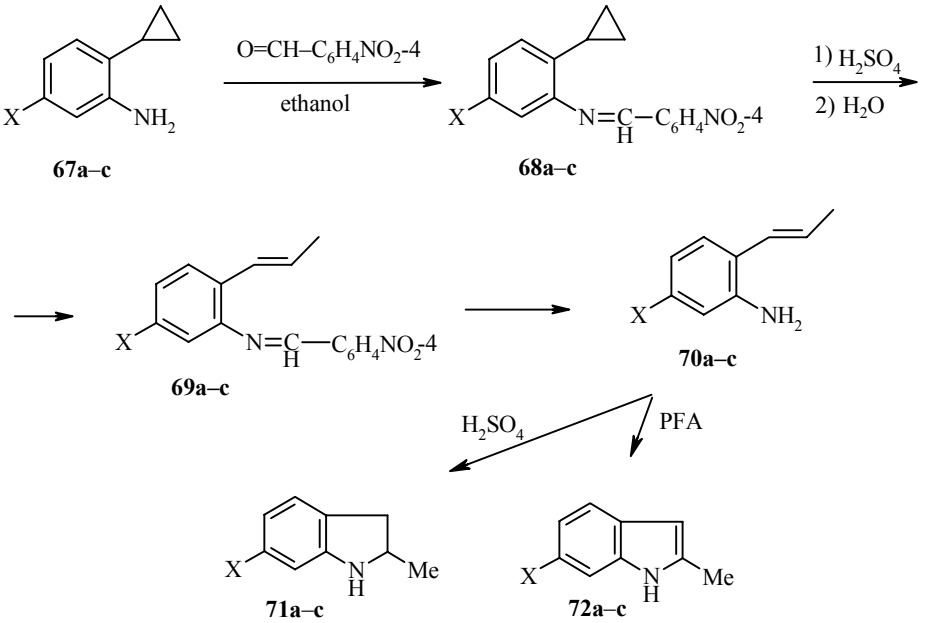
62 **63**

R = Me, Et; X = H, Pr-*i*, Bu-*t*, Pr-*cyclo*, Br, NO_2

An interesting feature of the discovered heterocyclization of azoxybenzenes is the fact that under the influence of catalytic amounts of alcoholates it can only be realized in the case of 2,2'-dipropionyl-substituted starting compounds of the **62** type. Monopropionylazoxybenzenes only enter into the reaction in the presence of equimolar amounts of sodium alcoholates, and the reaction takes place by a different path [50].



As well as *ortho*-nitrosoacylbenzenes *ortho*-alkenylanilines **67**, which are also easily obtained from the corresponding *ortho*-cyclopropyl-substituted benzenes, have been used in the synthesis of nitrogen-containing heterocycles [51]. Since it was not possible to realize the direct acid-catalyzed transformation of 2-cyclopropylanilines **67** into the targeted heterocyclic compounds, a series of consecutive transformations of compounds **67** and the products of their initial reactions were carried out. As a result the 2-methyl-substituted 2,3-dihydroindoles **71** [52] and indoles **72** were obtained with yields of 44-69% [52, 53].



In conclusion, we note that the unique ability of the three-carbon ring in phenylcyclopropanes to direct the nitro group during electrophilic nitration preferentially at the *ortho* position to the small ring opens up substantial prospects for the synthesis of functionally *ortho*-substituted cyclopropyl-containing benzenes. The transformations of the latter under the influence of various reagents will undoubtedly open up new possibilities for the production of both nitrogen- and oxygen-containing heterocycles and heterocycles containing other heteroatoms.

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