

PYRROLES FROM OXAZOLES

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The review is devoted to the transformation of oxazoles (including those with cationoid and mesoionic structures) into compounds of the pyrrole series.

Keywords: murchonones, oxazoles, oxazolium salts, pyrroles, ylides, recyclization.

The search for new methods for the synthesis of heterocyclic systems is always actual, and the development of new methods for the construction of the pyrrole ring is particularly significant: the pyrrole ring is the basis of life on Earth, as it is the major subunit of the vitally important biomolecules hemoglobin and chlorophyll. Like any other heterocycle, the pyrrole ring can either be assembled from acyclic precursors (one or several synthons) or another heterocycle can be used as its precursor, i.e., the pyrrole can be obtained by recyclization. Transformation of the oxazole ring can provide an interesting and unusual method for the production of pyrrole. Oxazoles are widely used as starting compounds in synthesis (e.g., of pyridines, imidazoles, and other heterocycles), but the diversity of the methods used for the transformation of oxazoles into pyrroles has not yet been duly reflected in the review literature.

Preparation of this review was prompted by the discovery by author and his group of an entirely new family of oxazole transformations to pyrroles in a series of condensed heterocycles containing a bridging nitrogen atom. This transformation, which has already been included in the latest edition of the classic textbook [1] and in a recent monograph on the chemistry of oxazoles [2], deserves comparison with all other examples (both known and exotic) of the oxazole conversion to pyrroles. Such a comparison, with the emphasis on the structural type of the transformation and on comparison of monocycles with condensed systems, is the theme of the present review, which is of interest both to synthesis specialists and to a wide range of teachers and high school students.

It is not necessary to duplicate the known examples of the pyrrole synthesis from oxazoles (presented in the books [2, 3] in the form of detailed tables), and they will only be used in the general schemes in order to complete the picture. In view of classification it is convenient to divide the oxazoles from which pyrroles can be obtained into simple subfamilies – neutral oxazoles (mesoionic and "normal") and oxazolium salts.

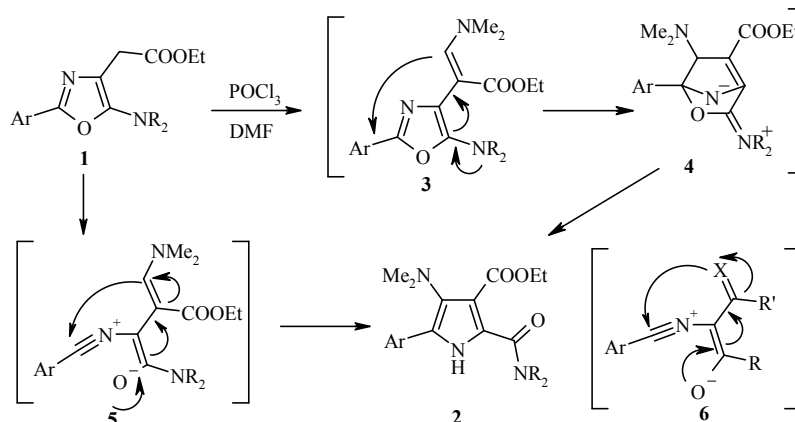
PYRROLES FROM "NORMAL" OXAZOLES WITH NEUTRAL STRUCTURE

During Vilsmeier formylation of the oxazole **1** the pyrrole **2** was isolated [4]. The bicyclic intermediate **4** proposed by the authors is not very plausible. It seems most likely that the oxazole ring of the intermediate

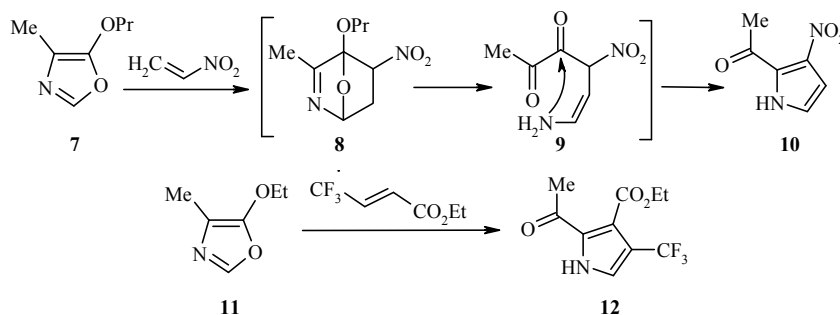
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product **3** is first opened thermally to the acyclic structure **5** and then undergoes cyclization. Exactly in this manner, through the dipolar ylide **6**, oxazoles are usually opened during the Cornforth rearrangement [5].

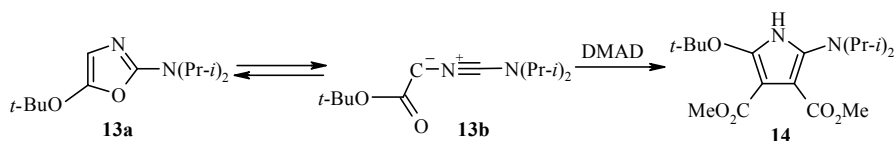


In reactions with dienophiles, oxazoles usually behave as cyclic azadienes forming cyclic adducts, which are easily transformed into pyridines (the Kondrat'eva reaction [6]). In very rare cases, however, the opening of such cyclic adducts can proceed abnormally, leading to pyrroles. Thus, in reaction with nitroethylene the oxazole **7** forms the nitropyrrole **10** instead of the expected nitropyridine [7] probably on account of the simultaneous cleavage of two bonds (C=N and C–O) in the cyclic adduct **8** followed by cyclization of the amino diketone **9**.

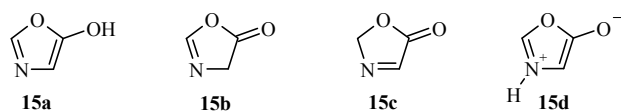


The pyrrole **12** is formed in an entirely similar way from the oxazole **11** [8]. In this case, however, the yield of the pyrrole is low, and the main product is the expected pyridine.

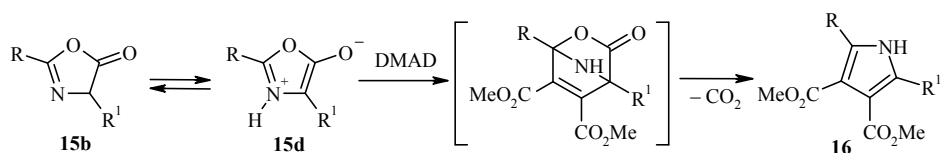
As shown by kinetic investigations [9], the oxazole **13a** is in equilibrium with the open-chain valence isomer – the nitrile ylide **13b**. Under the action of dimethyl acetylenedicarboxylate (DMAD) the 1,3-dipole **13b** readily closes the pyrrole ring **14**.



The 5-hydroxyoxazoles **15a** usually exist in the form of oxazol-5-ones in one of three tautomeric oxo forms, i.e., the "normal" 4*H*-oxazolones **15b**, the 2*H* tautomers (pseudooxazolones) **15c**, and finally the mesoionic 3*H* tautomers (oxazolium-5-olates) **15d**. All three subclasses **15b-d** contain the CO₂ structure motif and can lose this fragment in various ways, often forming pyrroles.

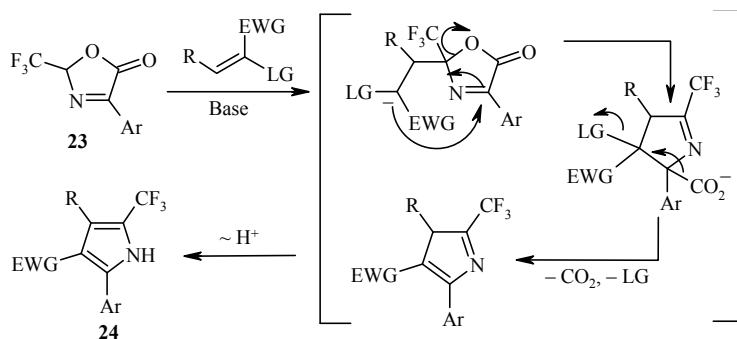
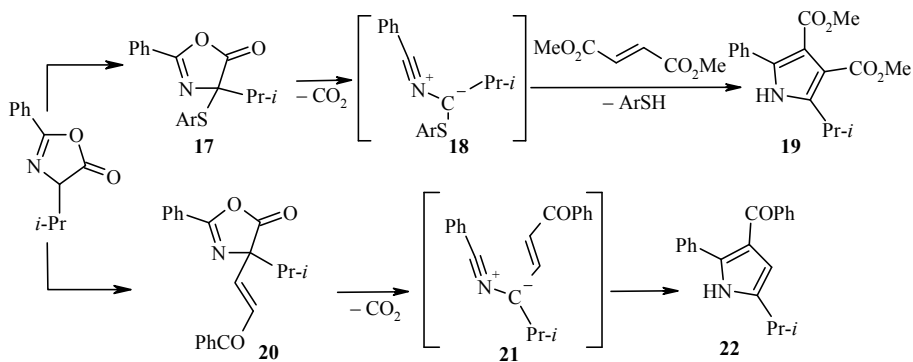


The most usual type of tautomers, oxazol-5(4*H*)-ones **15b**, which are easily obtained by the acylation of α -amino acids, react quickly with DMAD forming the pyrroles **16**. At the same time, as shown in the pioneering works by Huisgen [10], the initial particle in this transformation is most likely not the 4*H*-oxazolone **15b**, but a minor mesoionic component of the tautomeric mixture **15d** having the structure of a 1,3-dipole.



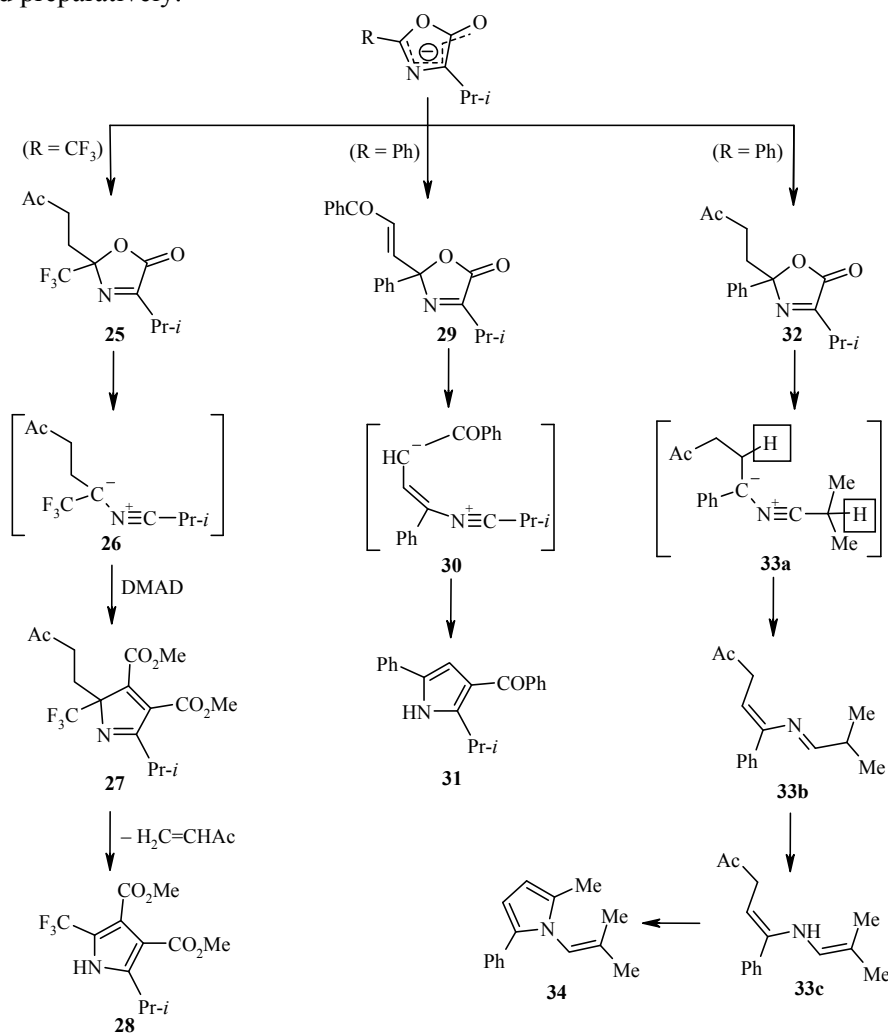
The reactions of the mesoionic oxazoles will be discussed below, and we will therefore emphasize the examples where the "normal" (covalent) tautomers of the oxazolones **15b,c** were converted into pyrroles without participation of the dipolar forms **15d**. It is not difficult to obtain such oxo forms by replacing both protons of the CH_2 groups in the oxazolones with other radicals.

As it turned out, if the oxazol-5-one is fixed rigidly in the 4*H* form the elimination of a CO_2 molecule leads to dipolar nitrile-ylide intermediates, which readily undergo cyclization to pyrroles under the action of dipolarophiles. Thus, the 4*H*-oxazolone **17** is capable of reacting with alkene through a stage of the 1,3-dipole **18** formation to give the pyrrole **19** [11]. In another example from the same authors, the adduct **20** (obtained from oxazolone and benzoylacetylene), which has the same type of oxazolone 4*H*-form as compound **17**, underwent intramolecular transformation to the pyrrole **22** through a stage of the 1,5-dipole **21** formation, which is vinylogous to the ylide **18**.

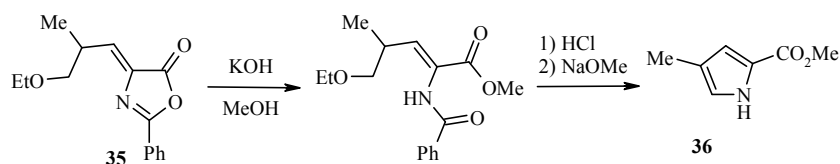


It is possible to obtain various pyrroles just as effectively in the case of the isomeric pseudooxazolones **15c**, which have a rigidly fixed *2H* form. One method is the reaction of 5(*2H*)-oxazolones such as compound **23** with alkenes having electron-withdrawing (EWG) and leaving (LG) groups which leads to the formation of the pyrroles **24** according to the mechanism shown above proposed by the authors in [3, 12].

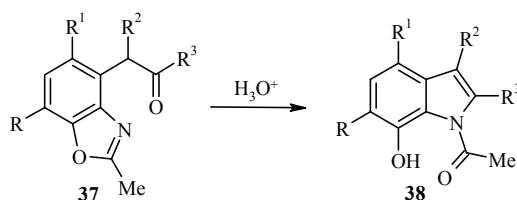
A series of interesting experiments in the already cited work [11] makes it possible to obtain a more precise idea about the mechanism of the transformation of *2H*-forms of oxazolones into pyrroles. Under the action of DMAD, 5(*2H*)-oxazolone **25** forms the pyrrolenine **27**, which undergoes aromatization to the pyrrole **28** by elimination of a methyl vinyl ketone residue. In this case, the authors suggested that the 1,3-dipole **26** formed intermediately from the oxazolone takes part in cycloaddition. The intramolecular transformation of *2H*-oxazolone **29** to the pyrrole **31** can be explained just as easily by the initial formation of the 1,3-dipole **30** from the oxazolone by loss of CO₂. It is interesting that the *2H*-oxazolone **32**, being the structural analog of the molecule **25**, undergoes transformation during thermolysis into a pyrrole **34** by a completely different structural type. To explain this fact the authors admitted the possibility that the 1,3-dipole **33a** is transformed into the azadiene **33b** and the enamine **33c** (which enters into cyclocondensation); and a series of analogs of structures **33b,c** were isolated preparatively.



Two more examples of transformation of oxazoles into pyrroles, differing in structural type from all the preceding ones and involving hydrolytic cleavage of the oxazole ring, have been described in the literature. In the first of them, hydrolysis of the substituted oxazolone **35** into an acylamino acid leads to oxidative cyclization to the pyrrole **36**.



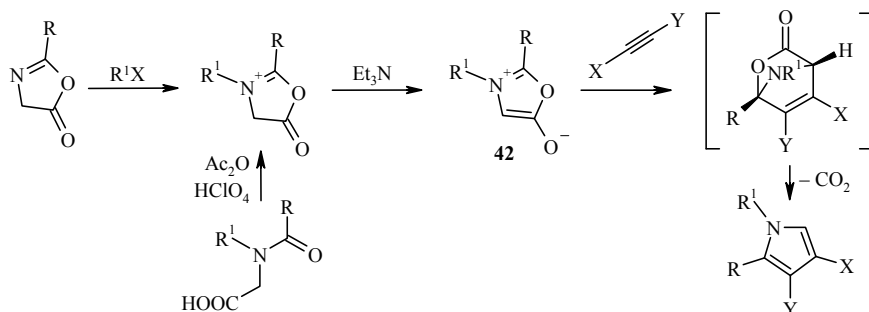
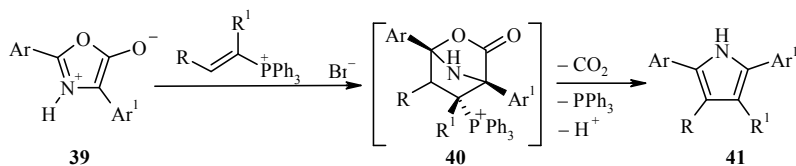
In the second example hydrolysis of the oxazole ring in the series of benzoxazoles **37** leads to closure of the pyrrole ring of indoles **38**.



As seen, examples of the production of pyrroles from "normal" oxazoles (not having mesoionic or cationoid character) are encountered infrequently. An exception is oxazol-5-ones, in which any of the three possible tautomeric types is a prospective precursor of pyrrole compounds.

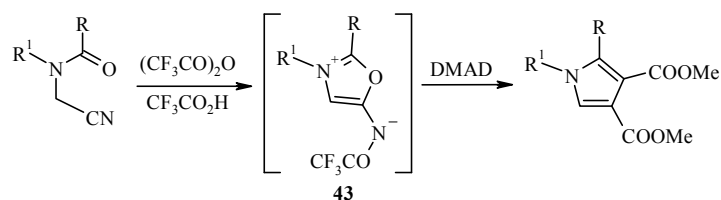
PYRROLES FROM MESOIONIC OXAZOLES

A reaction, in which a microimpurity of the tautomer **15d** predetermines the high yield of the pyrrole **16** from the oxazolone **15b**, clearly illustrates how promising the mesoionic oxazoles are as precursors of pyrroles. There are few other examples of the conversion of "normal" oxazolones into pyrroles with the participation of mesoionic tautomers **15d**, and the most significant is the synthesis of pyrroles **41** from oxazolones **39** by the action of vinyltriphenylphosphonium salts [13]. The regioselectivity of this process is interesting and indicates the preferable formation of a cyclic adduct **40** in which the phosphorus atom and the oxygen atom of the C=O group are as close together as possible in space.

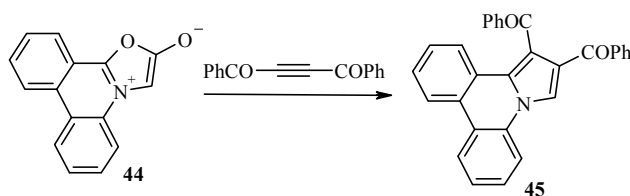


If the dipolar form **15d** is fixed by inserting an alkyl group at the nitrogen atom, a class of well known mesoionic oxazolium-5-olates or munchnones **42** is generated. Munchnones are unstable and are easily dimerized, and it is convenient to generate them *in situ* by *N*-alkylation of oxazol-5-ones or by cyclization of α -amino acids having an *N*-acylated alkylamino group. The large charge gradient in munchnones (at the ends of the CNC 1,3-dipole) promotes the readily occurrence of 1,3-dipolar cycloaddition reactions, while the possibility of elimination of CO₂ from cyclic adduct generates a multitude of possibilities for conversion of munchnones into other heterocycles. Using alkynes (and alkenes with leaving groups) permits to obtain various pyrroles specifically from mesoionic oxazoles.

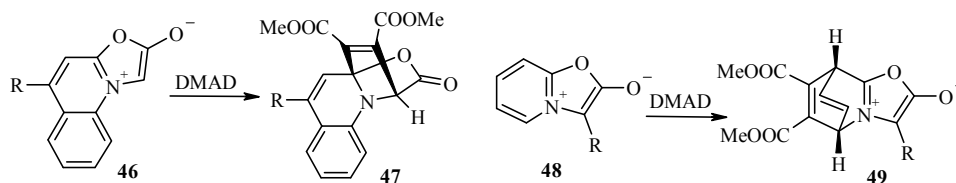
This aspect of the oxazole chemistry is represented extremely widely in the classic series of books [2, 3, 14] and in earlier reviews. Replacement of the exocyclic oxygen atom in munchnones by a nitrogen atom (i.e., the transition to oxazolium-5-imidates **43**) as before makes it possible to obtain compounds of the pyrrole series successfully from mesoionic oxazoles [15].



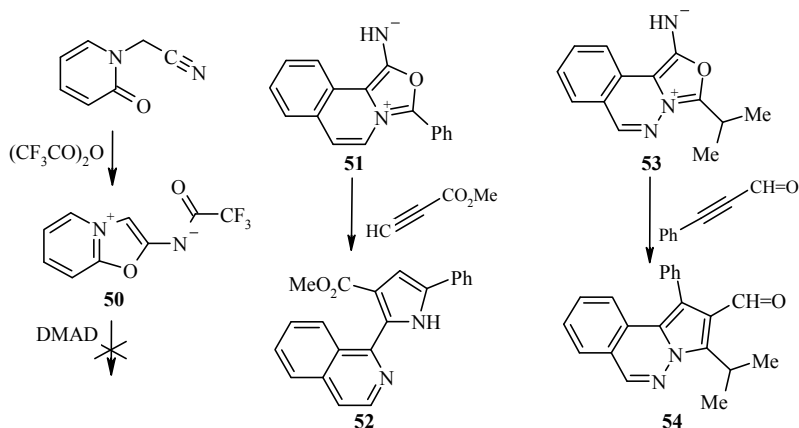
Coming from monocyclic munchnones and imidates to condensed systems, the direction of the reactions with dipolarophiles becomes less well-defined. The tetracyclic munchnone **44** behaves like monocycles and is capable of transforming the oxazole ring into a pyrrole ring by the action of an acceptor alkyne [16] with the formation of compound **45**.



At the same time for the tricyclic munchnone **46**, the reaction with the alkyne can be stopped at the stage of the cyclic adduct **47** [17], while the bicyclic munchnones **48** on the contrary lose the ability for cycloaddition in the five-membered fragment completely. For the reaction of munchnones **48** in the case if R = H with DMAD rapid dimerization occurs, with R = Ac no signs of reaction are observed, and only with R = Ar is the cyclic adduct **49** was formed with participation of the six-membered ring [18, 19].

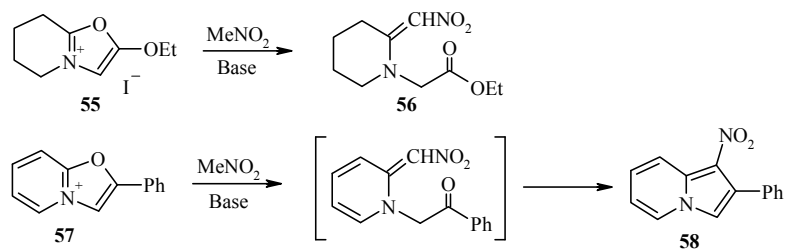


The bicyclic oxazolium-2-imidates **50** are passive in reaction with DMAD and very slowly give trace quantities of tarry products [19, 20]. For a different type of annelation of the munchnone imine and azine, e.g., in the case of the tricycles **51** [21] or **53** [22], the oxazole fragment can be transformed into pyrrole. However, the mechanisms of the processes and the types of products differ greatly, and the structure of the pyrroles **52** and **54** is determined by the nature of the annelated ring.

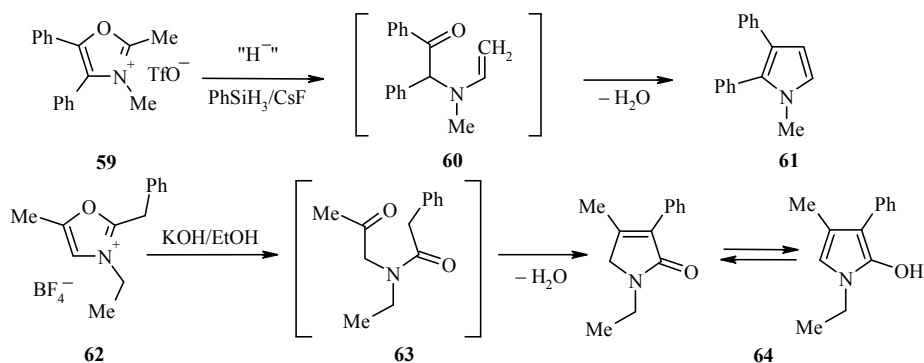


PYRROLES FROM OXAZOLIUM SALTS

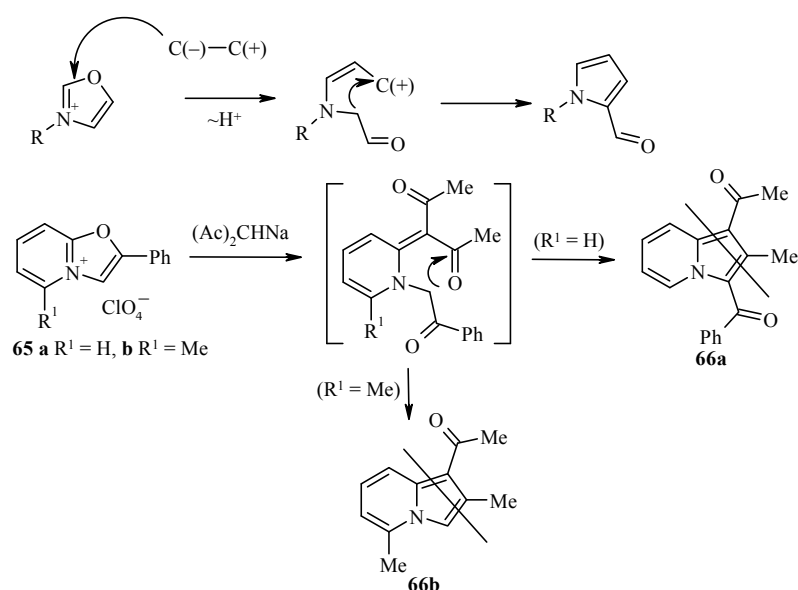
There are many different examples of the pyrrole formation from oxazolium salts. The electrophilic oxazolium cation reacts vigorously with a large number of nucleophiles, including the anions of CH acids, with ring opening, while the obtained intermediates can close a new pyrrole ring at the next stage. Two illustrative examples are presented in the scheme below – opening of the oxazolium cation **55** by the action of the nitromethane anion [23] with the formation of the nitroenamine **56**, the cyclization of which to pyrrole is somewhat hindered. In the second example the bicyclic oxazolium cation **57** is not only opened by the action of the same anion, but transforms the oxazole ring into a pyrrole ring, giving the indolizine **58** [24].



The transformation of the derivative **57** to **58** is somewhat reminiscent of the transformation of pyrylium (or pyridinium) salts into nitrobenzenes (see our early review [25]) on the one hand and of the Yur'ev reaction (replacement of the oxygen atom in oxazoles or furans by a different heteroatom) on the other. At the same time the one-carbon nucleophilic fragment that converts the oxazole into the pyrrole does not necessarily have to be in the external reagent. This is illustrated by two transformations differing in mechanism, but of the same structural type, where a one-carbon unit with potential C-nucleophilicity, i.e., the alkyl group (the α -methyl group in the salt **59** or the α -benzyl group in **62**), is connected directly at position 2 of the oxazolium cations. During hydride opening (for the salt **59** [26]) or alkaline opening (for the salt **62** [27]) of the cations the C-nucleophilic character of these groups is retained in the open forms **60** and **63** (or their tautomers) and leads to cyclocondensation with the formation of pyrroles **61** and **64**. In structural type the two recyclizations are extremely reminiscent of the Dimroth rearrangement, and in the inclusion of the *exo*-carbon radical in the new ring they are reminiscent of its modifications (the synthesis of phenols from 2-alkylpyrylium salts or of anilines from 2-picolinium salts by the Kost–Sagitullin–Gromov method).

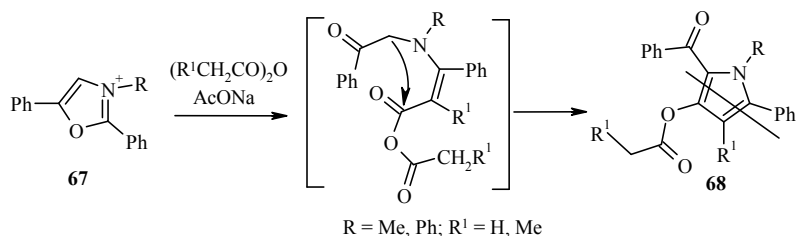


If the *C*-nucleophilic and *C*-electrophilic centers in the molecule of a CH acid capable of bringing about opening of the oxazolium salt are adjacent, closure of the new pyrrole ring can take place in a different way.

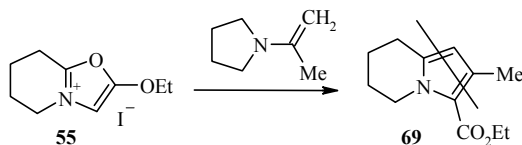


Thus, during the reaction of the bicyclic oxazolium salts **65a,b** with the acetylacetone anion [28, 29] two carbon atoms of the reagent (indicated in the scheme above by a solid line) enter the composition of the new pyrrole ring **66**. In the case of the salt **65b** deacylation of the pyrrole ring of the indolizine **66b** takes place as a side reaction.

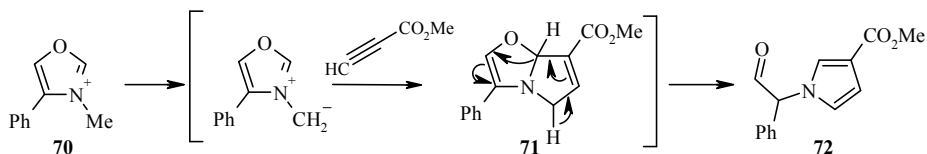
It is interesting that no such transformation under the action of β -diketones is known for monocyclic oxazolium salts (it may simply be that nobody has tried to realize it), although there is a direct analog. In this case the *C*-nucleophile for the salts **67** is an extremely unusual substrate, i.e., the anion of acetic (or propionic) anhydride. This is a bifunctional reagent of the $\text{C}(+)-\text{C}(-)$ type and inserts two carbon units into the new pyrrole ring **68** [30].



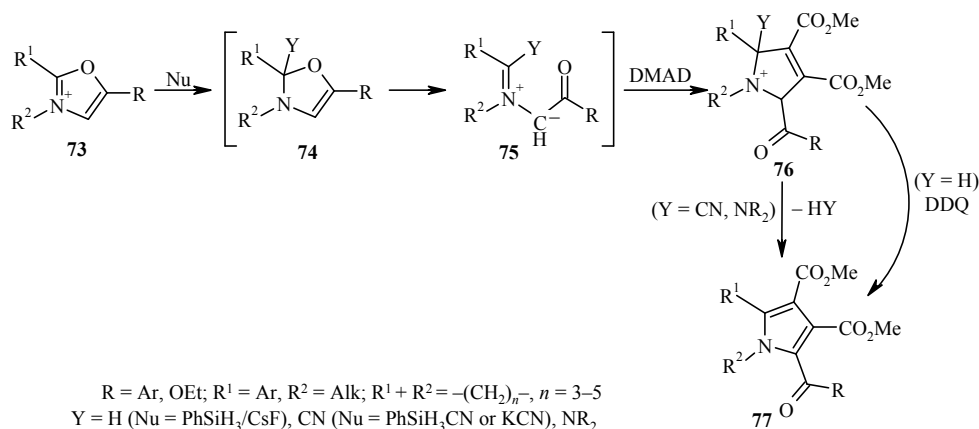
A completely analogous type of transformation to that described above was also observed in the reaction of the oxazolium salt **55** with acetone enamine [23], prepared *in situ*. As in previous cases, two carbon atoms of the reagent, nucleophilic and electrophilic, appear in the final structure of the pyrrole **69**.



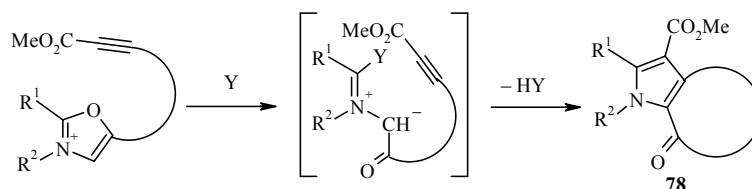
In all the cases examined above, the *N*-substituent in the oxazolium salts did not take part in the formation of the new pyrrole ring, passing unchanged into the radical at the nitrogen atom of the final pyrrole. At the same time it is well known that the *N*-CH₃ (or *N*-CH₂R) group in the salts of any heterocycles potentially possesses both CH acidity and *C*-nucleophilicity due to the possibility of the *N*-ylide formation from the salt. Heterocyclic *N*-ylides are excellent 1,3-dipoles, while in reactions with dipolarophiles they are convenient precursors of pyrroles. This is confirmed in example of the oxazolium *N*-ylide obtained from the salt **70**, to which it is possible to add a pyrrole ring. In the obtained cyclic adduct **71** the oxazole ring is easily opened, and the final transformation [31] corresponds to conversion of the oxazole **70** to the pyrrole **72**.



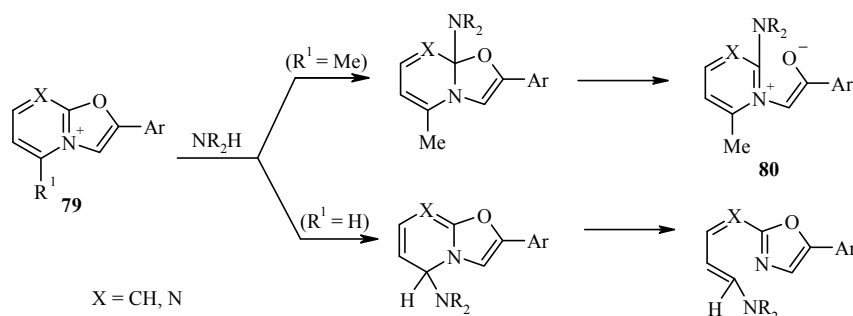
Examples of the synthesis of pyrroles by generation of ylides from oxazoles and their salts are not limited to those described above. Earlier we mentioned that various types of ylides are often formed during the thermolysis of neutral oxazoles. Oxazolium salts and their adducts *2H*-oxazolines are even more closely related to the ylides. As was shown by two research groups (see [26, 32] and [23, 33]), oxazolium salts **73** with free position 4 form with certain nucleophiles (*Y* = H, CN, NR₂) unstable oxazoline adducts **74** that readily open the ring with the formation of the azomethine-ylides **75**. The obtained ylides react readily with dipolarophiles, giving the pyrroline cyclic adducts **76**, which are aromatized spontaneously (or by enforced oxidation) to the pyrroles **77**. The close parallels between the chemistry of such azomethine-ylides **75** (with leaving group *Y*) and oxazolium salts, including an interesting aspect of their synthetic equivalence, were discussed in details in our recent review [34].



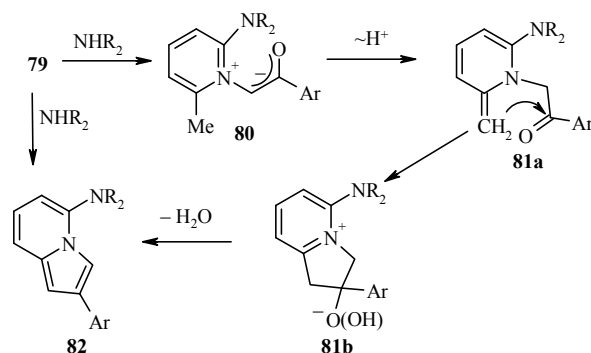
The methodology of direct oxazolium salt conversion into pyrroles through ylides was used effectively in the intramolecular cycloaddition for the synthesis of condensed pyrroles **78** and of certain natural compounds of the indole series [35-37].



Condensed oxazolium salts **79** in which the pyridine (or other azine) ring is annelated at a C=N bond are capable of being analogously converted into the betaine-ylides **80** by the action of secondary amines. In this case, however, the oxazoloazinium salts **79** exhibit ambident properties and are capable of opening both the oxazolium and the azinium rings (see the review [38]).



Only in cases where the azine fragment of the salts **79** contains a methyl (alkyl) group R^1 (sterically preventing attack in the six-membered ring) adjacent to the nitrogen atom does regioselective opening of the oxazole ring to the azinium *N*-ylide **80** occur. Due to the high CH acidity of the α -methyl groups in the pyri(mi)dinium salts the obtained ylides do not have any transformation pathways apart from tautomerism to the enamines **81a**. The enamines **81a** in turn are considered classical intermediates in the Chichibabin synthesis of indolizines and rapidly undergo cyclization to the pyrroline hydrates **81b**, which are aromatized to the 5-substituted indolizines **82**.

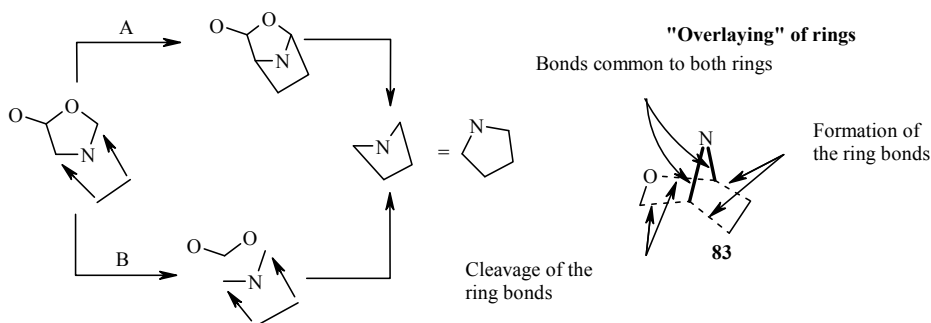


As a result a completely new type of the oxazole ring conversion into a pyrrole ring, leading to the formation of 5-aminoindolizines difficult to obtain in any other way, is realized. Pyrrolopyrimidines can be obtained in a similar way.

Such a transformation proceeds successfully under the action of secondary amines [38-43] or alcoholates [42, 44] and is accelerated by microwave irradiation [43]. The functions in the pyridine fragment vary extremely widely, and they include acceptor type substituents ($A = \text{CN}, \text{CONH}_2, \text{CO}_2\text{Et}, \text{NO}_2$), alkyl groups and/or alicycles where the number of additional methylene units n varies from 1 to 4 [42-48]. The preparative aspects of this reaction were described in the review [38], and in this context it is important to note the uniqueness of the structural type of this oxazoles to pyrroles conversion, which does not have analogs in the chemistry of monocyclic oxazolium salts. In fact, as discussed above, it is not difficult to obtain ylides (e.g., compound **75**) from monocyclic oxazolium salts, but in the analogous ylides **80** from oxazoloazinium salts there is a completely new structural motif, i.e., the α -methyl group of the azinium ylide, which acts as C-nucleophile during closure of the pyrrole ring.

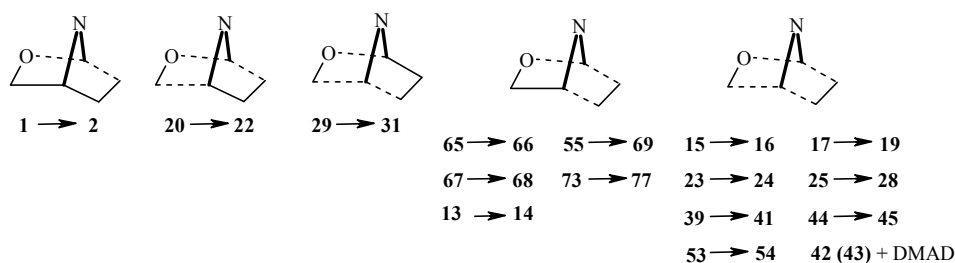
CLASSIFICATION OF THE TRANSFORMATIONS OF OXAZOLES TO PYRROLES

For the purpose of classifying the recyclization of one heterocycle to another a convenient structural language of recyclization graphs (RG) was developed [49-51]. Briefly we recall that to construct the RG the ring of the starting heterocycle is placed over the ring of the final structure, and the superfluous atoms and bonds (not contained in the rings) and also all the multiple bonds of the rings are removed. In the obtained RG (usually having only two rings) the breaking and forming bonds are marked by dotted lines (edges) while the bonds passing from one ring to another are marked by bold lines. The RG does not carry information about the sequence of cleavage and formation of the bonds (i.e., about the mechanism of the process), but makes it possible to compare the recyclizations with each other according to the degree of structural relation.



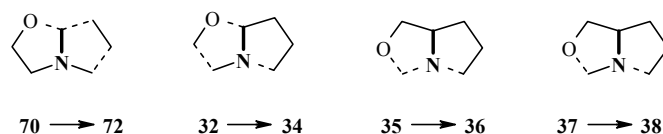
Simple analysis shows that the processes of CO_2 elimination from the tautomeric oxazolones (including the mesoionic oxazolones) by the action of acetylenic dipolarophiles are described by the same type of RG **83**, irrespective of whether the process takes place through a cyclic adduct (path A) or through the generation of an ylide (path B). The bold bridge of graph **83** shows clearly that it is the three-atom $\text{C}(2)\text{-N-C}(4)$ fragment of the oxazole that changes into the three-atom $\text{C}(2)\text{-N-C}(5)$ fragment of the new pyrrole ring in both processes of types A and B.

In a number of the examples under discussion this structural type (the passage of the CNC fragment from the oxazole to the pyrrole) is preserved, but the RG has a different number of dotted lines since certain

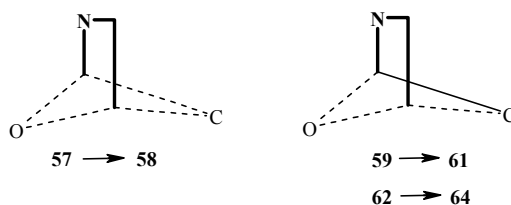


additional bonds are initially present and/or are not cleaved in the course of the reaction. The scheme above shows the corresponding RGs and the pyrrole–oxazole pairs (with the numbers of the compounds) for the reactions described above that have a clearly defined structural relationship.

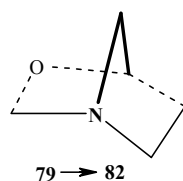
The second group of recyclizations is more scattered and includes processes in which the *two-atom* CN fragment of oxazole (marked by a bold line in the RG) moves into the pyrrole ring. The graphs clearly show that this CN fragment can be different in different processes: in the starting oxazole it is either the C(2)–N or the C(4)–N fragment.



The third group of recyclizations involves transfer of the *four-atom* CNCC fragment of the oxazole into the pyrrole ring. The one-carbon center that complements this fragment up to pyrrole is, for example, nitromethane or a C-nucleophilic alkyl group already added to the oxazole. As already mentioned, these transformations are closest in structural type to the Yur'ev reaction and Dimroth rearrangement.



The three classes described above actually exhaust all the diversity of the currently known transformations of oxazoles to pyrroles. The last structural subclass is our discovered transformation of oxazoloazinium salts to condensed pyrroles and has the following recyclization graph:



Obviously, in this case the three-atom oxazole fragment NCC is transferred to the pyrrole ring, and this distinguishes this structural subclass from the general group, where the oxazole gives up a rather different fragment CNC to the pyrrole ring.

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