

ACETALS OF AMIDES AND LACTAMS IN THE SYNTHESIS OF HETEROCYCLIC COMPOUNDS

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The review collects the results of investigations on the synthesis of heterocyclic compounds, mainly azaheterocycles, from amide and lactam acetals. The reasons for the high reactivity of these compounds with respect to nucleophilic and electrophilic reagents are considered, as are the principal pathways of heterocyclization of amidine and enamine systems synthesized via amide and lactam acetals.

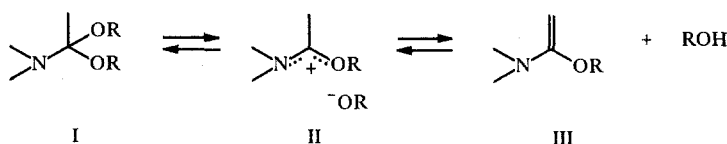
One of the most persistent tendencies of organic chemistry at present is toward the broadening and deepening of investigations into new synthetic routes for various classes of heterocyclic compounds. The development of nontraditional and at the same time preparatively convenient methods assures progress in the various areas of heterocyclic chemistry, among them the search for biologically active compounds and the development of new industrial methods for the synthesis of known medicinal compounds.

In this connection, the broad and ambitious investigations, mainly in the last thirty years, into amide and lactam chemistry based on the preliminary activation of the amide function are very promising. One of the most efficient means of such activation is the transformation of N-substituted amides and lactams into the corresponding acetals – highly reactive compounds whose application has been a prerequisite for the development of original synthetic methods for many organic compounds including heterocycles.

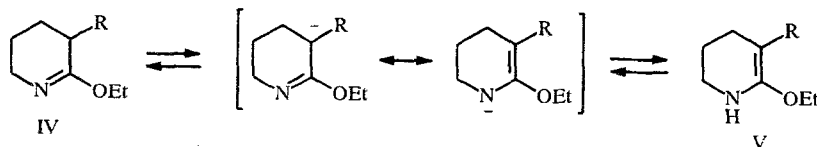
The chemistry of activated amides, among them amide acetals, has been considered in a number of reviews [1-5] and several aspects have been discussed in recent reviews of amidines [6] and enamines [7]. Although these publications were not devoted to the chemistry of heterocycles, to some degree or other they touched upon questions of heterocyclic synthesis.

In the present review the author has made no effort to discuss all of the investigations in which amide acetals were the starting compounds in syntheses leading to heterocyclic end products. The purpose of the review is to survey the main tendencies of the use of amide and lactam acetals in heterocyclic chemistry and outline the most promising directions for their application in this area. For this reason, along with new investigations into the chemistry of amide and lactam acetals, it is important to discuss work performed in previous years that bears strongly on the problems to be considered.

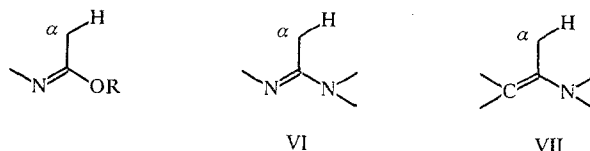
At present it appears generally accepted that in solutions of amide and lactam acetals a ternary equilibrium is established consisting of acetals I, ambidentate cations II and alkoxyanions, and finally α -alkoxyenamines III. It must, however, be noted that if equilibrium I \rightleftharpoons II has been indisputably demonstrated by measurement of the electroconductivity of amide acetal solutions [8-10] and by polarographic study of the diethylacetal of N-methylcaprolactam [10], still the second equilibrium II \rightleftharpoons III is postulated solely based on the investigation of chemical reactions [11-14] and cannot be regarded as certain.



Nonetheless, the existence of the equilibrium $\text{II} \rightleftharpoons \text{III}$ is indicated by an analysis of the data bearing not only on amide acetals but on lactime esters. An imine–enamine tautometry has previously [15-17] been shown, using O-ethylvalerolactime (IV) as an example, to exist in this series of compounds.

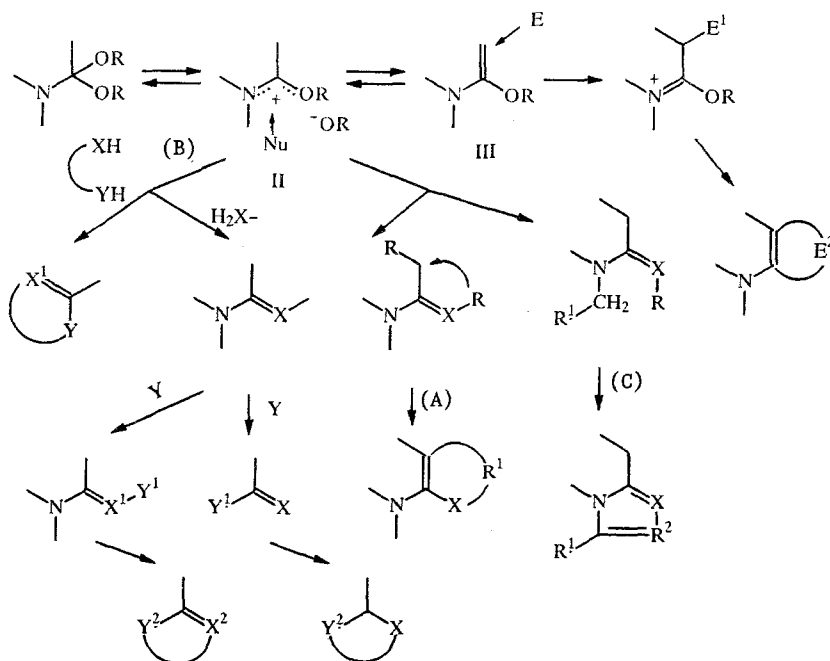


Inasmuch as the equilibrium transformation of lactime esters to α -alkoxyenamines V is accomplished by the transfer of a proton from the $\text{C}_{(3)}$ atom to the nitrogen atom and, as already observed, amide acetals I are in equilibrium with ambidentate cations II, it is reasonable to infer that cation II can lose the 3-H proton with relative ease (to which the presence of the alkoxyanion also contributes) to form the corresponding α -alkoxyenamine III and an alcohol. In actuality the structure of ambidentate cation II is substantially different from that of the lactime (or imino) esters on account of the full positive charge delocalized on the imino ester fragment. Clearly this charge should ease the elimination of the 3-H proton and promote the presence of α -alkoxyenamine III in the equilibrium. This consideration and a comparison of the structures of the lactime esters with the structurally similar amidines VI and enamines VII provide still another indication of the importance to the heterocyclization reaction of the sufficient mobility of the protons in the α positions in these systems as well.



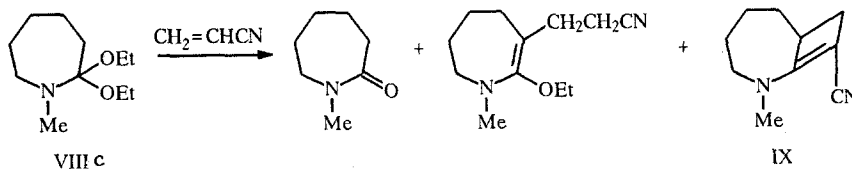
It thus seems reasonable to suppose that in amidines VI and enamines VII – classes of compounds readily obtained via amide and lactam acetals – the conditions may be generated for elimination of α -protons, opening the way in turn to various intramolecular cyclizations at this position of the molecule (pathway A). With respect to another route to heterocyclization, it may be noted that the very structure of amide (but not lactam) acetals bespeaks the possibility of their use as one-carbon fragments (pathway B), which at present is the principal means of application in the heterocyclic synthesis of the acetals of dimethylformamide and dimethyl acetamide [1, 4]. And finally one further possibility for the heterocyclization of amidines obtained from amide and lactam acetals involves the selection of appropriate substituents on the amine and imine nitrogens of amidines and the N and β -C atoms of an enamine system so as to make possible to Torp–Ziegler cyclization with the participation of the activated $\text{N}-\text{CH}_2$ group.

The scheme below summarizes the above-mentioned principles of heterocyclization via amide and lactam acetals.

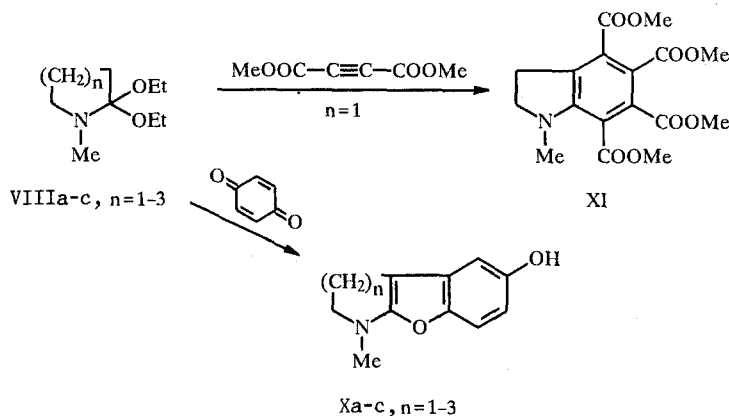


Before considering heterocyclization processes based on the reactions of amide acetals with nucleophilic reagents (due obviously to ambidentate cations II), let us consider the relatively few* investigations dealing with the synthesis of heterocycles via electrophilic attack by α -alkoxyenamines III.

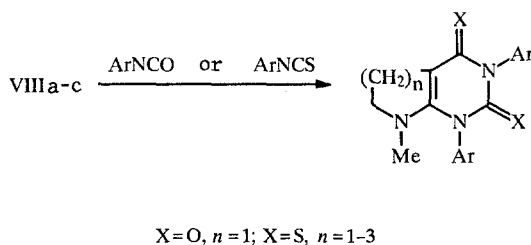
One such involved the reaction of the diethyl acetal of N-methylcaprolactam (VIIIc) with acrylonitrile to give, along with monocyclic compounds, the 2-azabicyclo[5.2.0]nonane derivative IX [18].



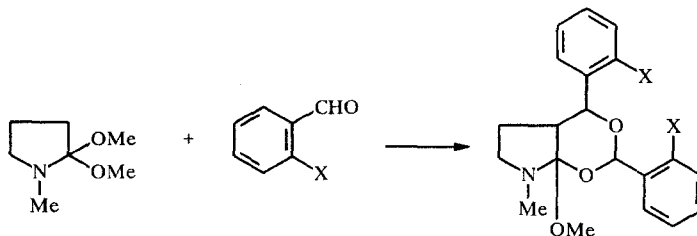
The Nenitzescu reaction of lactam acetals VIIIa-c with benzoquinone gave tricyclic benzofurans X [19], while the cycloaddition of acetylenedicarboxylic ester to the acetal of N-methyl-2-pyrrolidone (VIIIa) led to substituted indoline XI [20].



The acetals of lactams VIIIa-c reacted with aryl isocyanates and aryl isothiocyanates to form azacycloalkanol[2,3-d]pyrimidines (XII) [21].

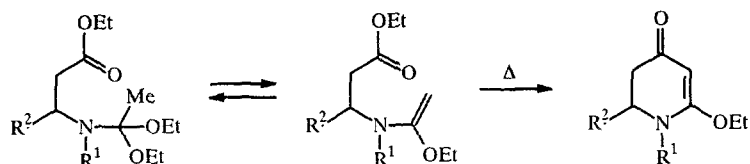


Dioxinopyrrole XIII was obtained on reaction of the acetal of N-methylpyrrolidone (VIIIa) with ortho-substituted aldehydes [22, 23]. It may be supposed that the first step of this process involved electrophilic attack at the 3-position of the lactam acetal:

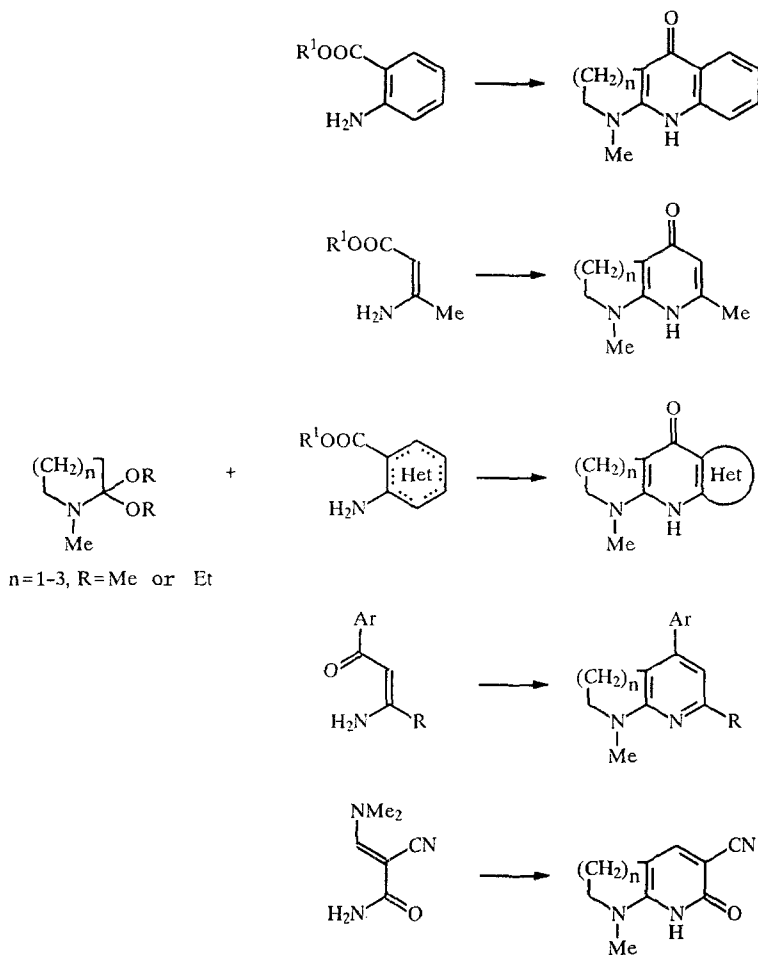


*Reactions with electrophilic reagents are most often undertaken not en route to heterocyclic compounds but rather to obtain α -substituted amides and lactams difficult to reach by other methods [1, 4, 5, 11-14].

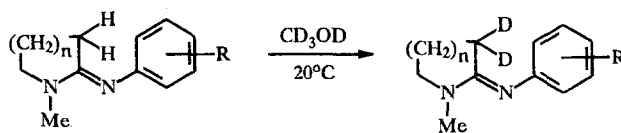
And, finally, we may mention the intramolecular cyclization of diethylacetals of *N*- β -ethoxycarbonyl ethylacetamide, which proceeds because of the presence of the α -alkoxyenamine structure at equilibrium, as shown in the scheme [24]:



An incomparably greater body of investigation has focused on the reactions of amides and lactams with amines and compounds possessing an active methylene unit, leading to the heterocyclization of the amidines and enamines thus obtained. Holding to the classification given above, we dwell first on the synthesis of amidines possessing in a neighboring (to the amidine fragment) position a group capable of electrophilic attack on a methylene unit (or methyl group) in the amidine meso position.

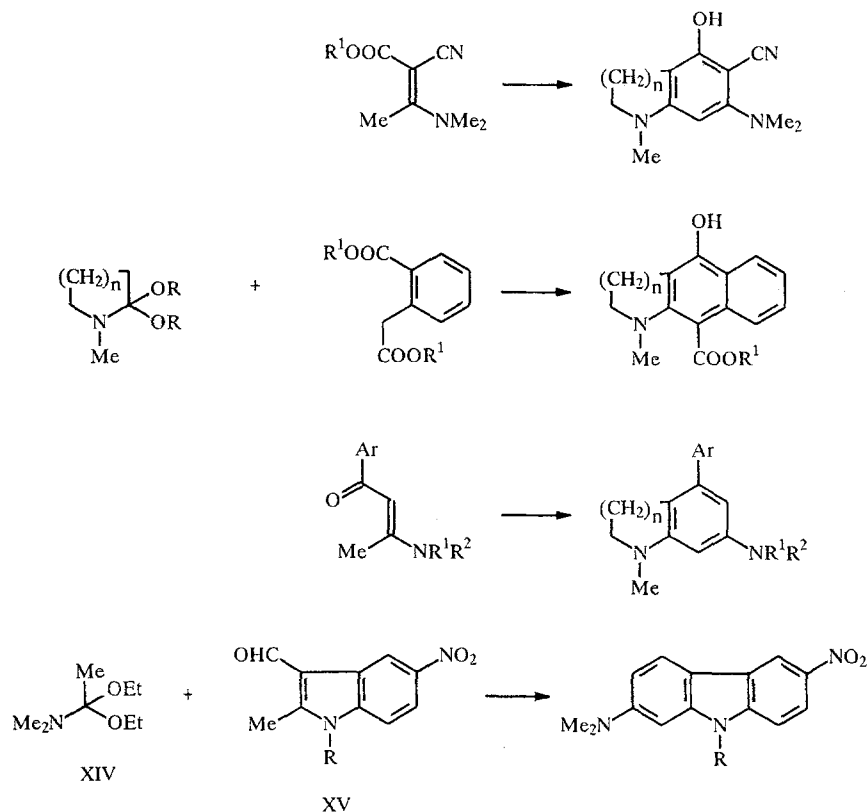


Among the reagents used have been anthranilic acid derivatives [25-27], β -aminocrotonic ester [28], the esters of ortho-aminoacids of the pyridine, pyrimidine, isoquinoline, and pyridine series [29-31], β -aminovinyl ketones [32], and enaminoamides [33]. It should be noted that although the schemes show examples involving lactam acetals, in most cases the diethyl acetal of dimethylacetamide (XIV) was also employed in the same studies and furnished the corresponding heteromono- and bicyclic systems. In many cases the intermediate amidines were isolated and characterized. In a special investigation [34] it was shown that hydrogen atoms of the CH_2 group in position 3 of the ring were subject to deuterium exchange under mild conditions, supporting the above proposition concerning the ready mobility of these protons. We may also point out that the rapidity of deuterium exchange is strongly dependent on the size of the ring and decreases in the order $n=6 > n=7 > n=5$, in good agreement with the ease of the cyclizations shown in the scheme.

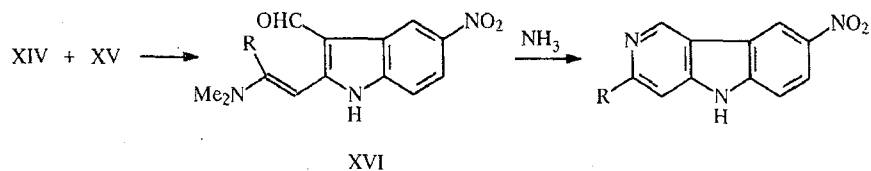


The influence of the size of the saturated rings on similar processes and the interpretation of the experimental results are discussed in detail in review [35].

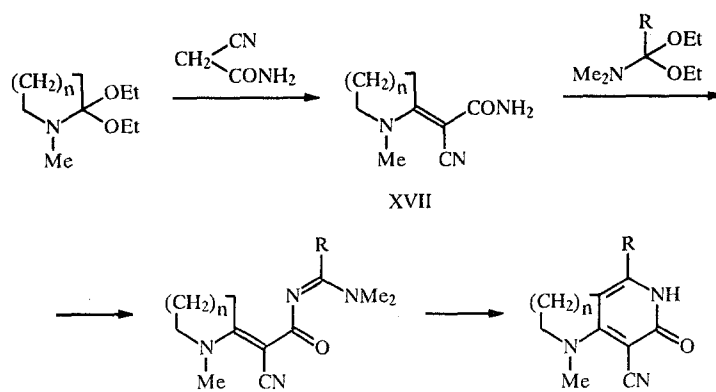
By essentially the same principle but with an initial condensation at an active methylene or methyl group, many condensed systems such as derivatives of indole, benzindole, quinoline, benzazepine, etc. have been synthesized. As the scheme illustrates, cyclization in these cases leads to the formation of a newly annealed benzene ring. As examples are shown the reactions of lactam acetals (although in many cases acetal XIV has also been employed; these studies are given in the references) with tertiary enaminoesters [36], ortho-substituted esters of phenylacetic acid [5], tertiary enamino ketones [37], and the diethylacetal of N,N-dimethylacetamide (XIV) with derivatives of 2-methyl-3-formylindole XV [38].



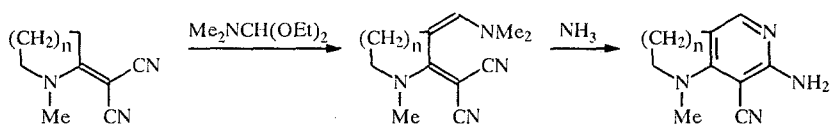
It is evident that in these cases substituted enamines are formed as the intermediates and further cyclize by the indicated pathways. This circumstance can be exploited for other heterocyclization reactions, in particular in the last case [38], intermediates of the XVI type were starting compounds for a novel synthesis of γ -carbolines.



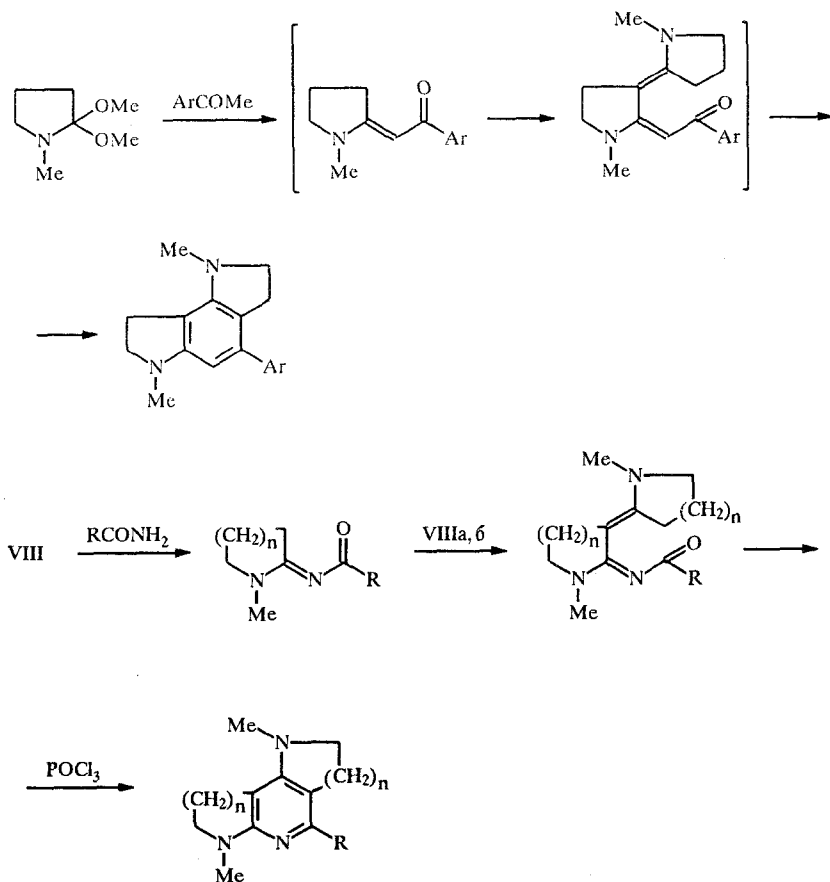
The intermediate enamines may have no appropriate substituents for further cyclization and such functional groups may be introduced into the molecule at a subsequent stage. Thus the reaction of enaminoamides XVII with amide acetals proceeds at the amide NH_2 group and generates the conditions for closing of a new pyridine ring, the amidine fragment being the substituent that generates the conditions for heterocyclization. This method was used in a series of syntheses of 2-pyridones and condensed 2-pyridones [39-42].



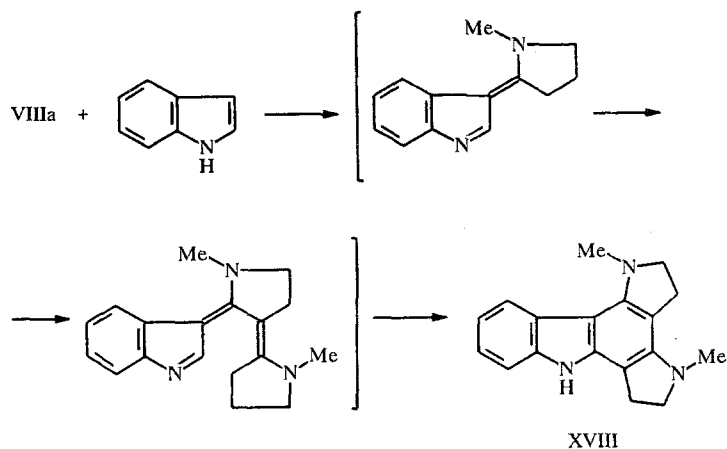
A new substituent can also be generated in such a way that heterocyclization proceeds at the β function of the enamine and not at the 3-position of the ring or the meso- CH_3 group of the amidine fragment [42].



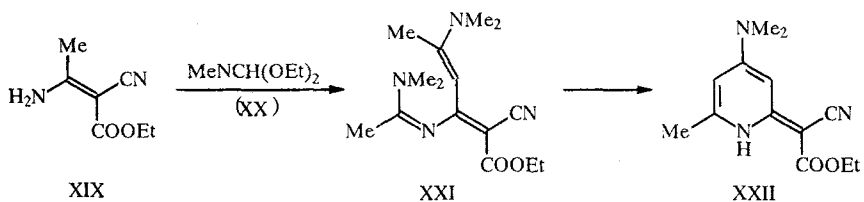
Closely related to these studies are heterocyclizations based on cyclic enaminoketones [43] and derivatives of acyl amidines [44].



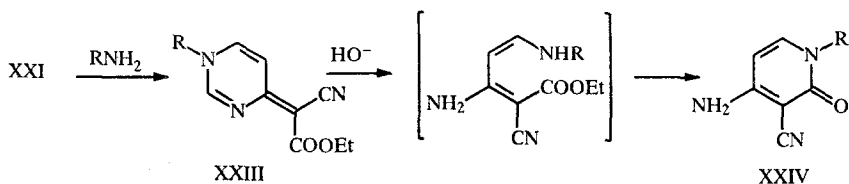
In principle the one-step synthesis of dipyrrolo-carbazole XVIII [45] is of the same type.



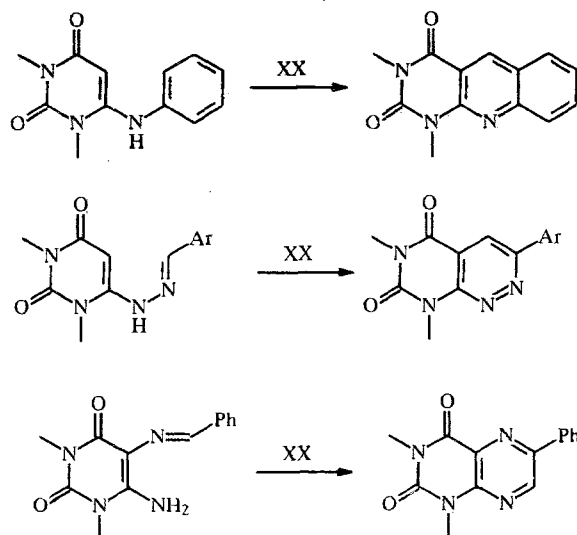
And finally we may point at a study concerning the reactions of primary enamines XIX with the diethylacetal of dimethylformamide (XX). Here the reaction proceeded at the α -methyl as well as at the primary amino groups and the resulting dienaminoamidine XXI cyclized to pyridine derivative XXII. The reaction proceeds unambiguously by attack on the α -methyl substituent by the amidine fragment [46].



It should be noted that compound XIX was also used as an intermediate in a pyrimidine synthesis, the methylenepyrimidine XXIII thus synthesized being readily opened by alkaline reagents with subsequent recyclization to 2-pyridine XXIV, possessing a primary amino group at the 4-position [46].

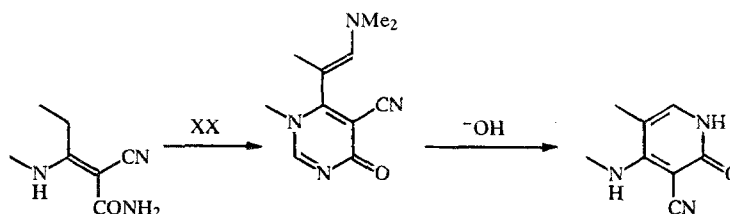


As is evident from the foregoing material, the substituents employed for further cyclization are frequently the amidine $-\text{N}=\text{CR}-\text{NMe}_2$ or the enamine $-\text{CH}=\text{CR}-\text{NMe}_2$ groups, which form on reaction of amide acetals with amines or active methylene components. These compounds can sometimes be isolated from the reaction mixture and subjected to further reactions such as transamination or reaction with CH-acids, which in many cases are accompanied by heterocyclization. These processes are rather hard to classify and it must be kept in mind that the final result of participation in them of amide acetals is the introduction into the forming heterocycle of one or several one-carbon fragments. To this species of reaction (pathway B above) belong the reactions of amide acetals with various bifunctional reagents such as derivatives of *o*-phenylenediamine [47], *o*-aminoanthranilamide [47], 2-amino alcohols [48], and *o*-nitrosoamines [49]. Cyclizations to condensed pyridine [50], pyridazine [51], and pyrazine [52] derivatives with the aid of the acetal of DMFA are shown in the scheme:

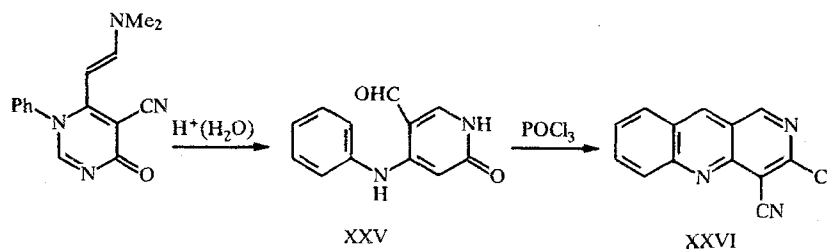


A long series of cyclizations of this type is discussed in detail in review [4].

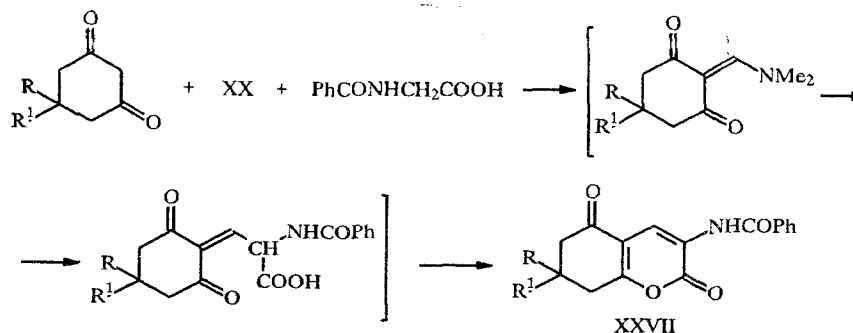
Secondary enaminoamides, under the action of amide acetals, cyclize to 4-pyrimidinone derivatives. It should be noted that at the same time acetal XX condenses also at the methylene unit in the 3-position of the ring (or the α -enamine methyl group) [42, 53-60]. This fulfilled the prerequisites for a new pyrimidine-pyridine recyclization proceeding in alkaline medium to form 3,4-disubstituted 2-pyridones.



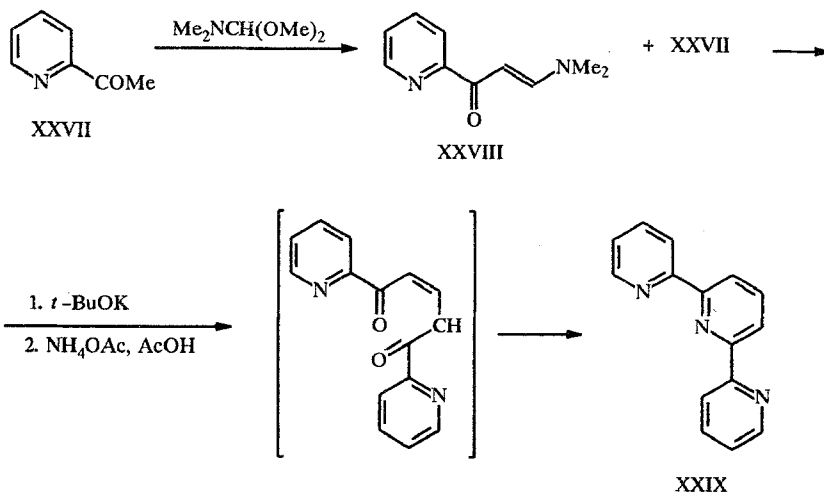
Using the example of 1-phenyl-4-oxo-5-cyano-6-dimethylaminomethylene-1,4-dihydropyrimidine, recyclization in acid medium was studied as well. It proved to proceed unusually, forming 3-cyano-4-anilino-5-formyl-2-pyridone (XXV), which was converted to benzonaphthiridine XXVI [60] under the action of phosphorus oxychloride.



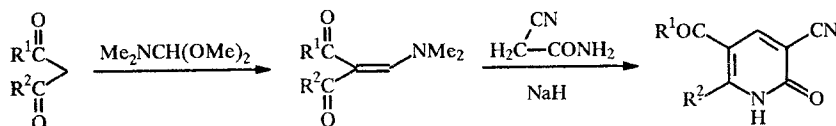
In recent years a number of reports have been published of the synthesis of various heterocycles based on enamines synthesized by condensation of the acetal of DMFA (XX) at the active CH_2 group. Thus a three-component reaction of derivatives of cyclohexane-1,3-dione, acetal XX, and N-benzoylglycine gave in 40-50% yield coumarin derivatives XXVII [61]. The reaction evidently proceeds by the following scheme:



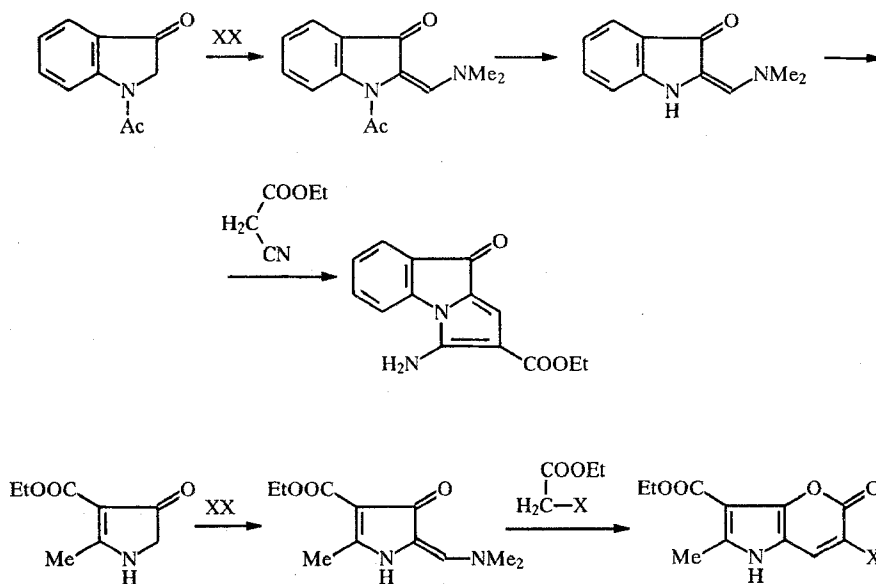
Enaminoketone XXVIII, obtained from 2-acetylpyridine, on reaction with the latter with further transamination by ammonia accompanied by cyclization is converted to 2,2':6',2''-terpyridine (XXIX) [62].



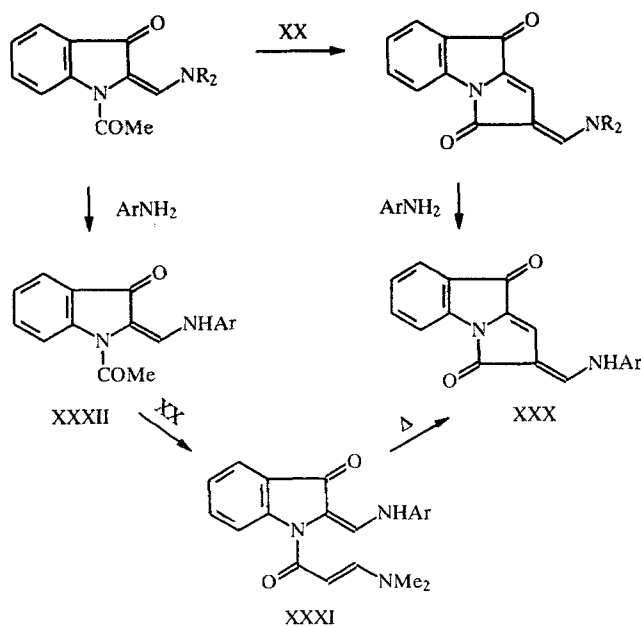
The reaction of enaminodiketones with cyanoacetamide gave 3-cyano-2-pyridones [63].



Enaminoketones – derivatives of indoxyl and 3-pyrrolin-2-one – were the starting compounds in a synthesis of pyrrolo[1,2-a]indoles and pyrrolo[3,2-b]pyranes in which the starting acetal XX in both cases was the source of the methine fragment [64, 65].

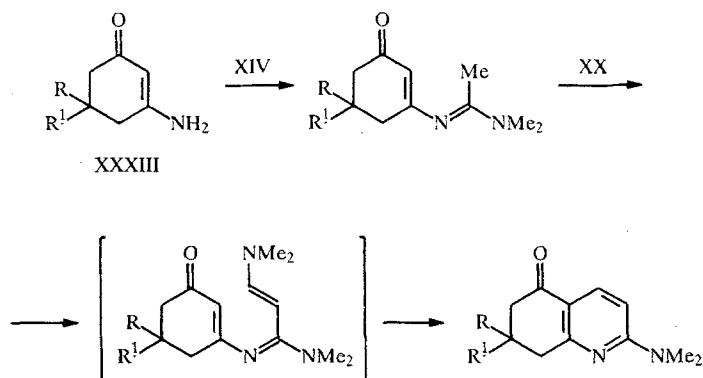


A novel reaction of amide acetals, forming a pyrrolo[1,2-a]indole XXX with another structure was recently discovered in an investigation of the reaction of 1-acetyl-2-dialkylaminomethylene indoxyl with acetal XX. As the scheme shows, acetal XX initially condenses at the methyl group of the N-acetyl fragment and then an unusual cyclization follows, involving attack at the β -position of the enaminoketone by the α -position of the other enaminocarbonyl fragment [66].

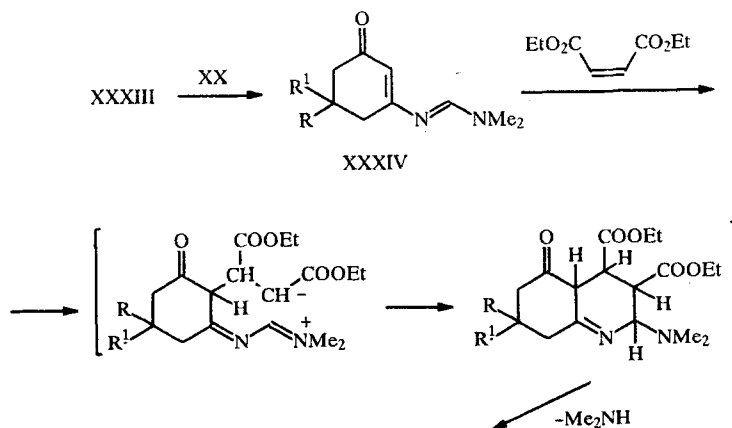


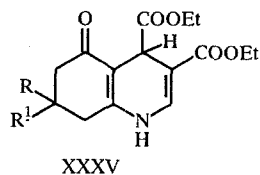
Just such a reaction pathway (if not an alternative one involving the initial cyclization of the pyrrole ring) is indicated by the isolation of the key intermediate XXXI on reaction of acetal XX with 1-acetyl-2-arylaminomethylene indoxyl (XXXII).

Still another unusual reaction based on successive use, first of the acetal of dimethylacetamide (XIV) and then of the acetal of DMFA (XX), in which the latter appears as the one-carbon component, is based on the use of the primary cyclic enaminoketones XXXIII [67].

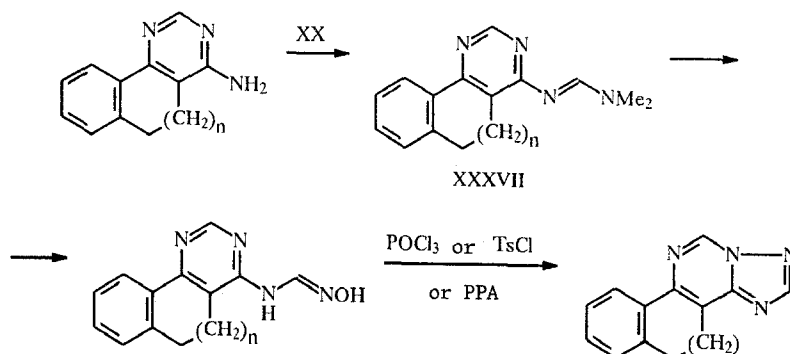
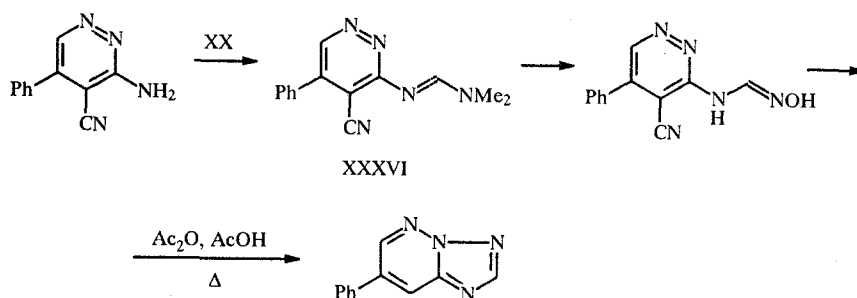


Interestingly, if acetal XX is introduced into the reaction with compound XXXIII, the enaminoketone XXXIV that forms may be used as the diene component in a reaction with maleic ester to give good yields of derivatives of dihydropyridine XXXV [67].



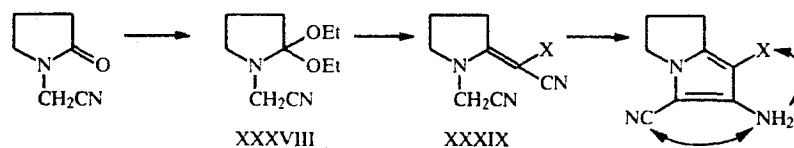


And to conclude this section of the review we may mention recent reports in which the intermediate compounds in cyclizations (where amide acetals take part also as one-carbon components) were amidines. These amidines (XXXVI, XXXVII) were transaminated with hydroxylamine with subsequent cyclization to triazole derivatives [68, 69].

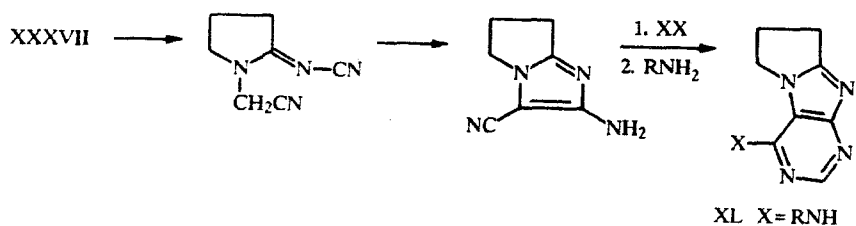


Without dwelling on other heterocyclizations of a similar type we may point out that amide acetals have also been used as the one-carbon fragment in the syntheses of isoquinoliniones [70], pyridopyrimidines [71], pyridine derivatives [72, 73], pentaazapyrene [74], and pyridine and isoquinoline [75].

With respect to pathway C above, we may first consider the reactions of the diethylacetal of N-cyanomethyl-2-pyrrolidone (XXXVIII) [76] with compounds possessing an active methylene unit. In this case enamines XXXIX are formed, having a functional substituent on the nitrogen atom. The introduction of enamines of this type into a Torp–Ziegler reaction makes possible the synthesis of pyrrolizine derivatives that are convenient starting compounds for the synthesis of heterocyclic systems such as condensed pyrrolo[3,2d] and [4,3-d]pyrimidines [77-81] (to this end the acetal of DMFA was used to supply the carbon fragment).

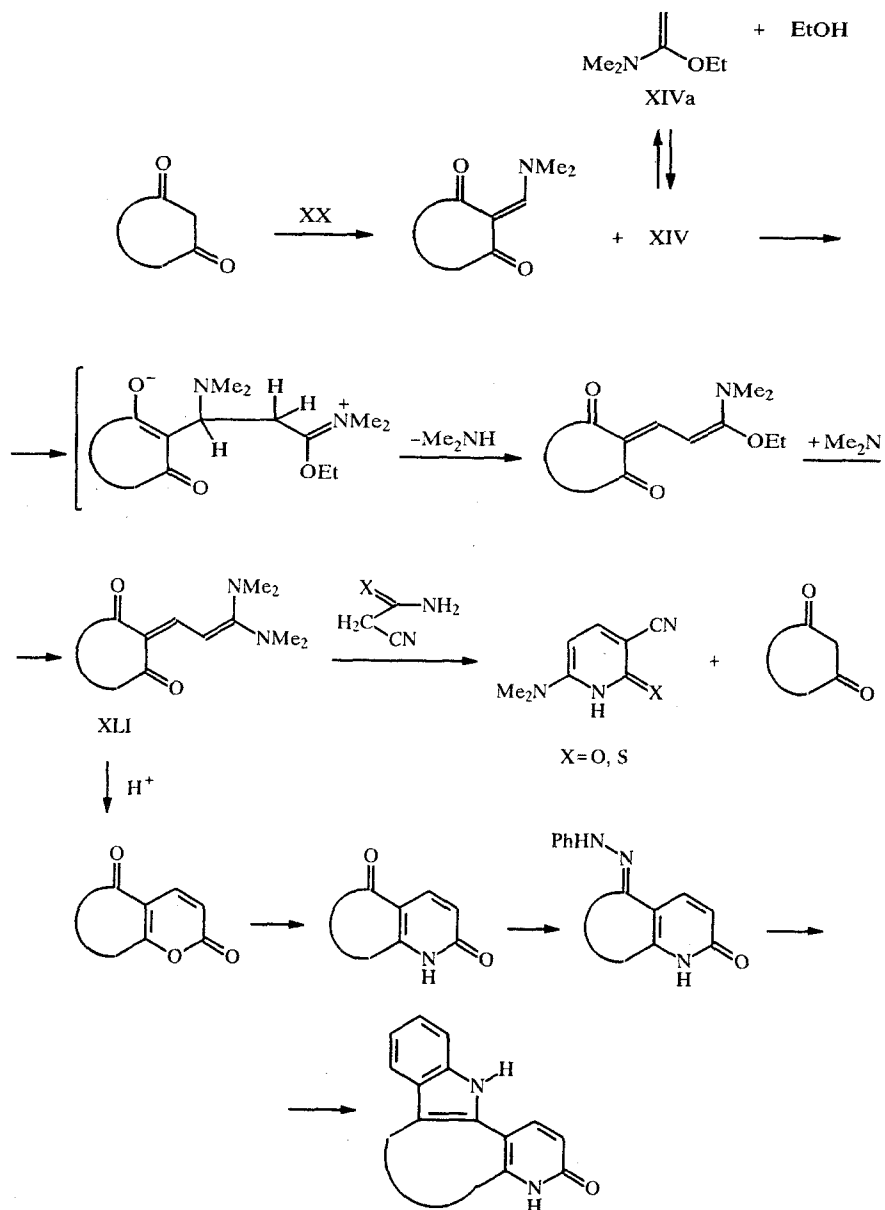


An analogous approach using acetal XXXVIII and cyanoamide enabled the synthesis of condensed imidazoles and from them polymethylenepurines XL, systems difficult to reach by other methods [82]:



In concluding the survey of reports on the synthesis of various heterocyclic compounds from amide and lactam acetals, we should dwell on a number of studies that do not fit into the classification proposed.

These are principally investigations based on unusual reactions of enaminediketones with the diethylacetal of dimethylacetamide (XIV).* The latter acts here as α -alkoxyenamine XIVa and this new reaction leads to dienediaminodiketones XLI, very promising synthons for many kinds of heterocyclic synthesis [83-88]. Some of the approaches taken to the synthesis of oxygen- and nitrogen-containing heterocycles to date are summarized in the scheme:†

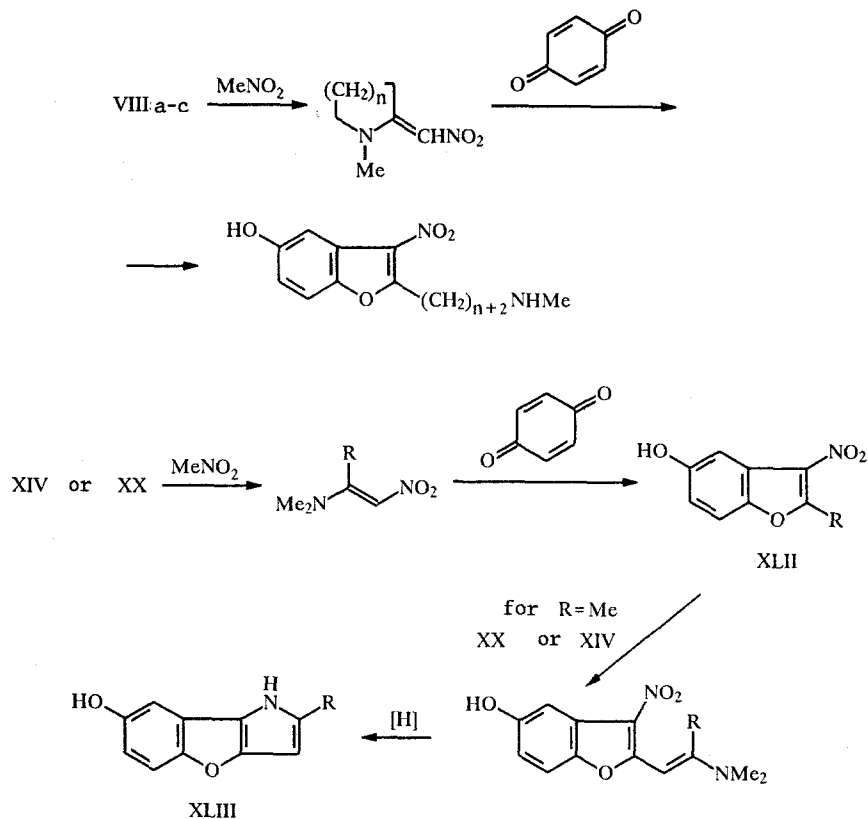


*The acetals of lactams VIIIa-c also react analogously.

†An arc in the scheme indicates that either cyclic or acyclic diketones may be used.

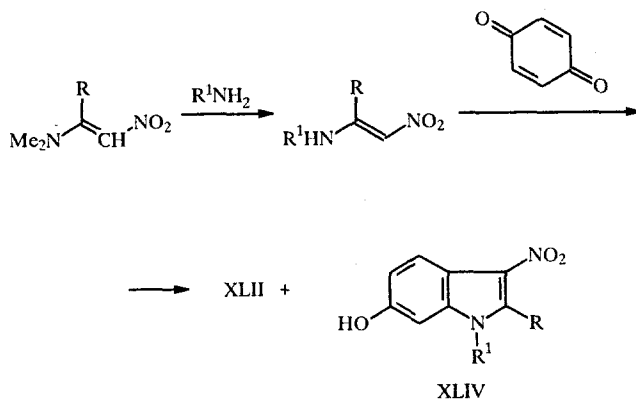
And finally, it is deserving of attention that the high reactivity of amide and lactam acetals offers an approach to the synthesis of the most diverse synthons and thereby opens new perspectives on various routes of heterocyclic synthesis. This refers in particular to the Nenitzescu synthesis of benzofuran and indole derivatives, where the use of amide acetals to obtain β -functionally substituted enamines has enabled the investigation of the condensation of these compounds with benzoquinone and its derivatives [89-92].

In particular, β -nitroenamines obtained on reaction of amide and lactam acetals with nitromethane condense smoothly with quinone in acetic acid in the presence of acetic anhydride to form 5-hydroxybenzofurans; for cyclic enamines the opening of the saturated azaheterocyclic fragment in the process is observed.



5-Hydroxybenzofuran XLII was also the starting compound in a synthesis of pyrrolo[3,2-b]benzofurans XLIII: here acetals XIV and XX acted in their usual capacity as sources of the one-carbon fragment.

Quite interestingly, when secondary nitroenamines (obtained from tertiary transamination) are introduced into a reaction with quinone, the reaction



proceeds along two pathways to the formation of benzofurans XLII and to indole cyclization. Here, however, not the expected 5-hydroxyindoles, but 6-hydroxyindole derivatives XLIV were isolated.

A discussion of the questions involved in the reasons for one or another pathway of the Nenitzescu reaction is beyond the scope of the present review; we here wished only to point out novel, nontraditional possibilities for the synthesis of heterocyclic compounds based on the use of amide and lactam acetals.

In summary, it may be concluded that the high reactivity of amide and lactam acetals in conjunction with their preparative accessibility offers great promise for the use of these compounds in heterocyclic synthesis and furnishes new approaches to the most diverse heterocycles of both synthetic and practical interest.

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