SOME SUBSTITUTION AND HETEROCYCLIZATION REACTIONS BASED ON 1,4-DIHYDROPYRIDINES

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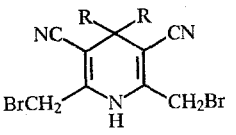
Data on the bromination, chlorination, and bromolactonization of 4-aryl-1,4-dihydropyridines are correlated. The reactions of the products of bromination of 4-aryl-1,4-dihydropyridines with various nucleophilic agents (amines and iodide, azide, and thiocyanate ions) and reactions involving the heterocyclization of the products of substitution of the 2,6-methyl groups of 4-aryl-1,4-dihydropyridines, which lead to condensed furo-, difuro-, pyrrolo-, dipyrrolo-, furopyrrolo-, and furothiazolopyridines and thiazolopyridothiadiazines are examined.

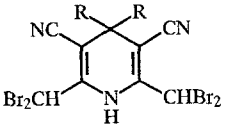
Researchers initially became interested in dihydropyridines (DHP) as analogs and model compounds of the reduced form of the dinucleotide of adenine and nicotinamide, viz, NAD-H, but, beginning in the nineteen seventies, primarily owing to their high cardiovascular activity, 4-aryl-1,4-DHP began to become recognized as an important class of medicinal substances [1-7]. An original preparation of the 1,4-DHP series "foridon" ("riodipin"), which is used for the treatment of hypertonia and ischemic heart disease, was created in the Institute of Organic Synthesis of the Latvian Academy of Sciences [8-11]. In general, thousands of 1,4-DHP have been synthesized throughout the world, but relatively little study has been devoted to their chemical properties; for a long time it has been assumed that the primary reactions that one may speak of relative to 1,4-DHP are exclusively oxidation and reduction. Research on reactions involving cleavage, rearrangement, addition, and substitution of the DHP ring has begun to develop only in recent years. Of greatest interest is the study of processes involving the halogenation of 1,4-DHP and the associated substitution and heterocyclization reactions.

BROMINATION

It has been known for a long time that 4-substituted or 4-unsubstituted 1,4-DHP undergo oxidation when they are treated with bromine [12]. The first example of the successful halogenation of a 1,4-DHP with retention of the ring was the bromination of 2,6-dimethyl-3,5-dicyano-4,4-dialkyl-1,4-DHP, which leads to bromo derivatives I and II [13-15].



I



II

Tetrahydrofuro[3,4-b]pyridines IV were subsequently obtained by the action of pyridinium bromide perbromide on 3,5-dialkoxycarbonyl-4-aryl-1,4-DHP derivatives [16].

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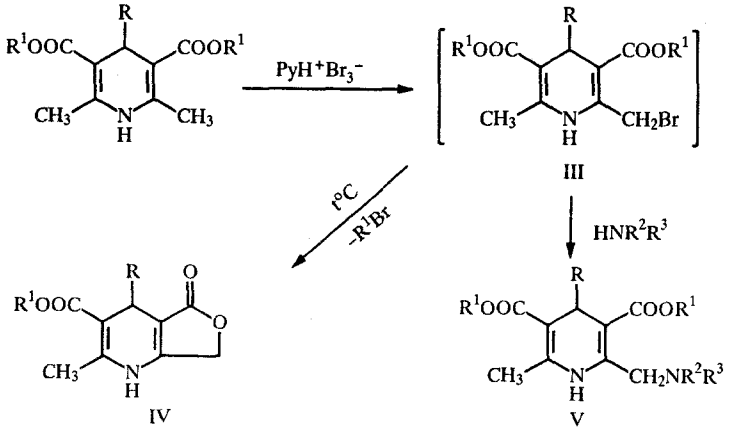
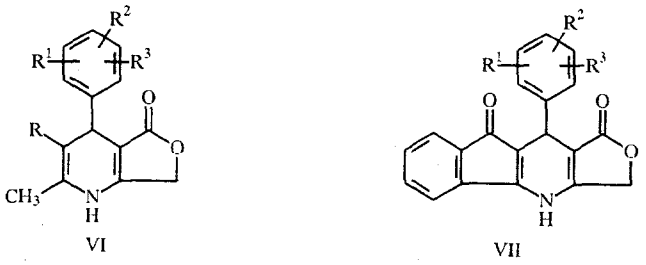
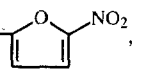


In some studies the relatively unstable intermediate III was either isolated [17-20] or was converted, without isolation, to substitution products V with various primary and secondary amines [21-26].

Other selective brominating agents, viz., N-bromosuccinimide (NBS), N-bromoacetamide (NBA), dioxane dibromide (D·Br_2), and 5,5-dibromo-2,2-dimethyl-4,6-dioxo-1,3-dioxane, can also be used for bromolactonization. The best results are obtained when NBS is used. It was found that bromolactonization reactions proceed well when there are various substituents in the 3 and 4 positions of the 1,4-DHP ring and when indeno[1,2-b]pyridine is used as the starting substance [27-29].

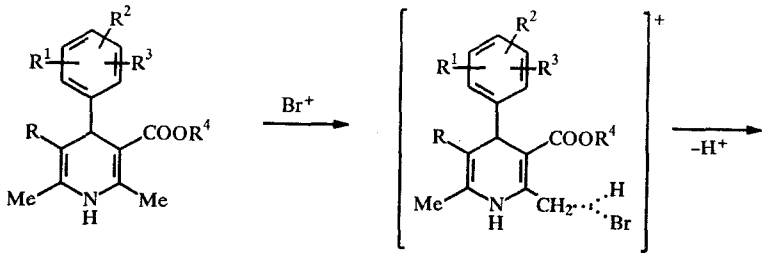


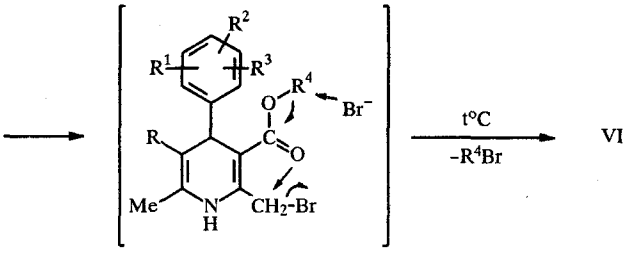
R = COOAlk, CN; R^1,R^2,R^3 = H, Cl, NO_2, OCH_3, $(CH=CH)_n$  , CF_3, $OCHF_2$, $SCHF_2$, SO_2CHF_2

It is generally accepted that allylic bromination, which is bromination of the 2-methyl group of 1,4-DHP, is a radical process [30-32]. But in this case the absence of irradiation and the presence of a radical inhibitor (hydroquinone) do not affect the progress of the reaction. It may therefore be assumed that direct electrophilic attack by the halide cation on the σ bond proceeds through a three-center transition state, as shown in Scheme 1.

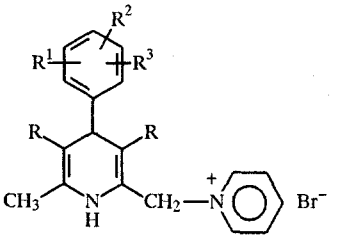
In the second step of bromolactonization splitting out of an alkyl bromide and heterocyclization of the 1,4-DHP to 1,4,5,7-tetrahydrofuro[3,4,5]pyridine VI occur under the influence of the bromide anion. When the reaction is carried out in the presence of pyridine (with pyridinium bromide perbromide or D·Br_2), pyridinium salt VIII can be isolated from the reaction mixture in addition to the lactone. It is evident that

Scheme 1





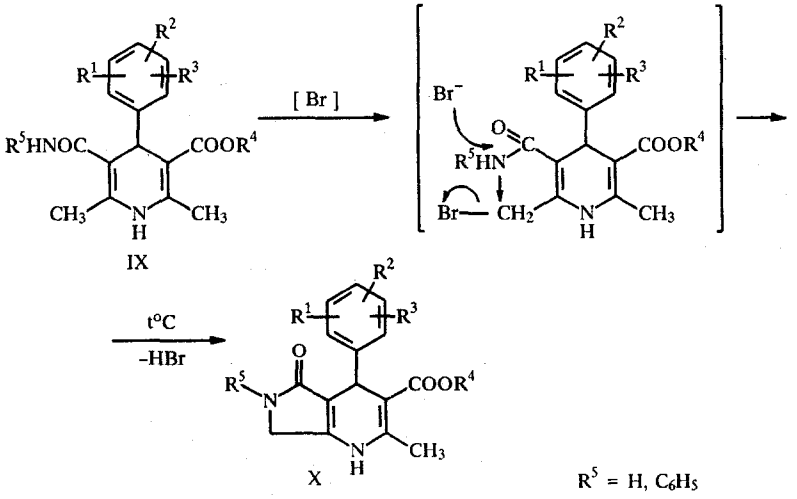
replacement of bromine by the nucleophilic pyridine also occurs simultaneously with bromolactonization [27].



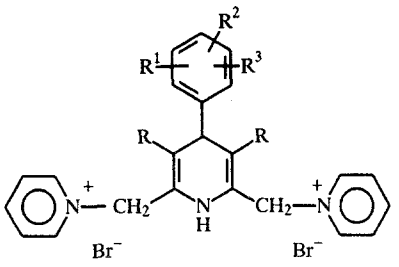
The bromolactonization of unsymmetrical 3,5-dialkoxycarbonyl-1,4-DHP showed that a reaction, during which a mixture of lactones is formed, takes place preferably with splitting out of the less bulky alkyl bromide. It is evident that steric factors play the chief role in this case. This is also indirectly confirmed by the decrease in the yields of furo[3,4-b]pyridines in the bromolactonization of 1,4-DHP with bulky (R^4 = amyl, adamantyl) 3,5-substituents.

Electronic factors evidently play the deciding role in the case of the reaction with another type of unsymmetrical 1,4-DHP, viz., amides IX — the electrophilic bromine attacks the methyl group that is adjacent to the weaker electron acceptor [33].

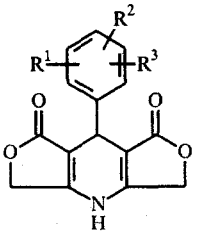
Pyrrolo[3,4-b]pyridines X are formed in the nucleophilic attack of the nitrogen atom containing an unshared electron pair at the bromomethyl group. Up until very recently, the synthesis of pyrrolo[3,4-b]pyridines via this pathway was unknown in the literature, although their preparation by cyclization of 2-aminomethyl-3-alkoxycarbonyl-1,4-DHP was described [19, 21, 34].



When the ratio of the brominating agents is increased, one can obtain either N,N′-[(3,5-dialkoxycarbonyl-4-aryl-1,4-dihydropyridine-2,6-diyl)dimethyl]dipyridinium dibromides XI (as a result of the reaction of the 1,4-DHP with a twofold excess of pyridinium bromide perbromide) or (in the case of bromination with a twofold amount of NBS or NBA) new heterocyclic compounds, viz., 6,8-dioxo-7-aryl-2,3,4,6,7,8-hexahydro(difuro)[3′,4′-b;3,4-e]pyridines XII [27, 35-37].



XI



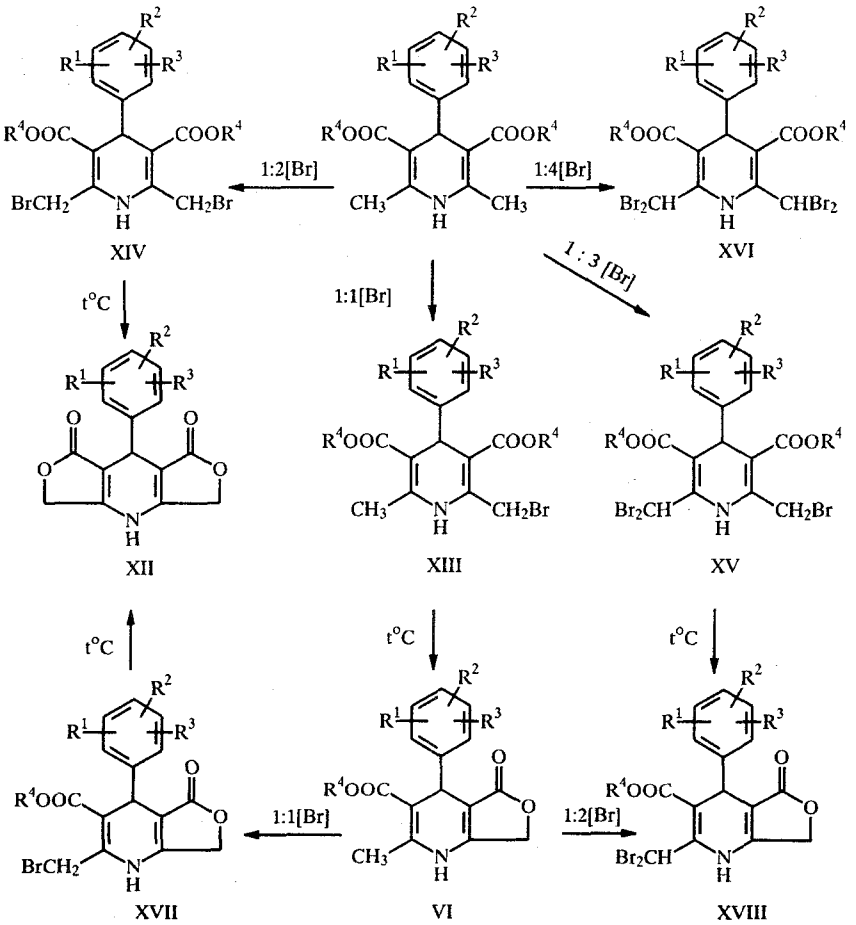
XII

A characteristic property of lactones and dilactones as compared with 1,4-DHP is an increase in their stabilities. It was established by means of a rotating disk electrode with a ring (RDER) that on passing from the 1,4-DHP to furo[3,4-b]pyridine the half-wave potential of the electrolytic oxidation $E_{1/2}$ increases by ≈ 0.2 V, while difuro[3′,4′-b;3,4-e]pyridines are, as a rule, oxidized with greater difficulty by, on average, 0.3 V than the corresponding 1,4-DHP. It is evident that this is associated with the fact that the presence of a lactone ring in the molecule determines a trans orientation of the carbonyl group and a coplanar (or close-to-coplanar) structure of the aminovinyl carbonyl system, and, as a consequence, the electron-acceptor effect of this group is expressed considerably more markedly as compared with the analogous ester group [27, 38].

Signals of protons of a lactone ring, viz., singlets at 4.56-4.79 ppm, are characteristic for furo and difuro compounds VI and XII.

An increase in the amount of NBS from 1 to 4 moles makes it possible to successively introduce bromine atoms into the 2,6-methyl groups of 1,4-DHP (Scheme 2). The formation of products XIII, XIV, XV, or XVII, which contain bromomethyl groups, is possible only at no higher than room temperature and only in the case of a brief reaction time. If these derivatives are subjected to higher temperatures, elimination of a molecule of an alkyl bromide and lactonization to the corresponding furopyridine occur [39-42].

Scheme 2

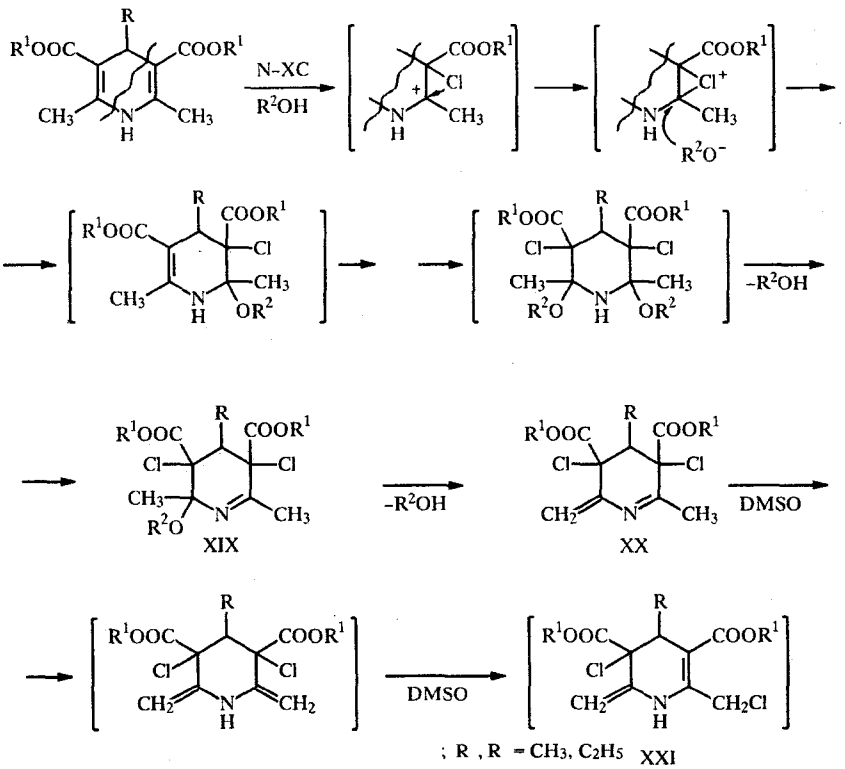


In the case of dibromomethyl groups (XVI and XVIII) heating the reaction mixture does not give rise to heterocyclization. It is evident that rapid replacement of two hydrogen atoms in each 2,6-methyl group by halogen occurs when the amount of brominating agent is increased. Subsequent splitting out of an alkyl bromide does not occur, as demonstrated by Stuart—Briegleb models, because of steric hindrance. It is evident, for the same reason, that replacement of all three hydrogen atoms of the methyl group by bromine is impossible. In contrast to the N-unsubstituted compounds, N-substituted 1,4-DHP react with NBS to give stable 2,6-bis(bromomethyl)-1,4-DHP of the XIV type, which do not undergo cyclization to furopyridine derivatives even when they are heated for many hours [43]. In these compounds steric fixation of the substituents in the 2 and 3 positions apparently hinders the formation of a lactone ring. The PMR spectral data and Stuart—Briegleb models show that for N-substituted 1,4-DHP of this series rotation of the bromomethyl groups in the 2 and 6 positions is impossible, and drawing together of the reaction centers for heterocyclization is therefore unlikely.

CHLORINATION

Prior to our research, reactions involving the chlorination of 1,4-DHP were virtually unknown. We used chlorine gas and a milder reagent, viz., N-chlorosuccinimide (NCS), as the chlorinating agents.

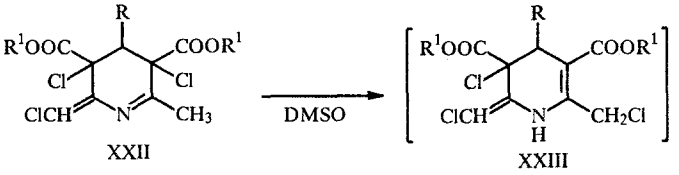
2,6-Dimethyl-3,5-dichloro-6-methoxy-3,4,5,6-tetrahydropyridines XIX ($R^2 = CH_3$) were isolated in good yields from the reaction mixtures in the reaction of a twofold excess of NCS with 2,6-dimethyl-3,5-dialkoxycarbonyl-4-aryl-1,4-DHP in methanol [44]. It might be assumed that the chlorination of the 1,4-DHP proceeds through the formation of carbonium and chloronium ions with the subsequent addition of methoxy groups of the solvent (Scheme 3). The resulting tetrahydropyridine splits out a molecule of alcohol and forms XIX. The analogous addition of ethoxy groups ($R^2 = C_2H_5$) occurs when the reaction is carried out in ethanol.



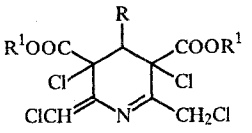
Tetrahydropyridines XIX are extremely unstable. The presence of three products, viz., 6-alkoxytetrahydropyridine XIX, XX with an exocyclic double bond (this substance could be isolated in some cases by fractional crystallization), and rearrangement product XXI, was detected in the NMR spectra (DMSO). The formation of rearrangement product XXI can be conceived of as being a two-step process. First, proton migration to the nitrogen atom occurs as a result of imine—enamine tautomerism; second, in the course of allylic rearrangement chlorine migrates to the side group (XXI). The literature contains

virtually no other data regarding the migration of chlorine (except for a [1,2]-rearrangement) and rearrangements in series of hydrogenated pyridines.

In addition to the addition of chlorine to the C=C bonds of the heteroring, chlorination of the 2,6-methyl substituents (to give XXII and XXIV) was also observed in the reaction of 1,4-DHP with a fourfold (sixfold) amount of NCS [44]. Judging from the NMR spectra, pyridine XXII in DMSO undergoes an analogous rearrangement as in the preceding case (to give XXIII). In the case of tetrahydropyridine XXIV no changes whatsoever occurred after several days.



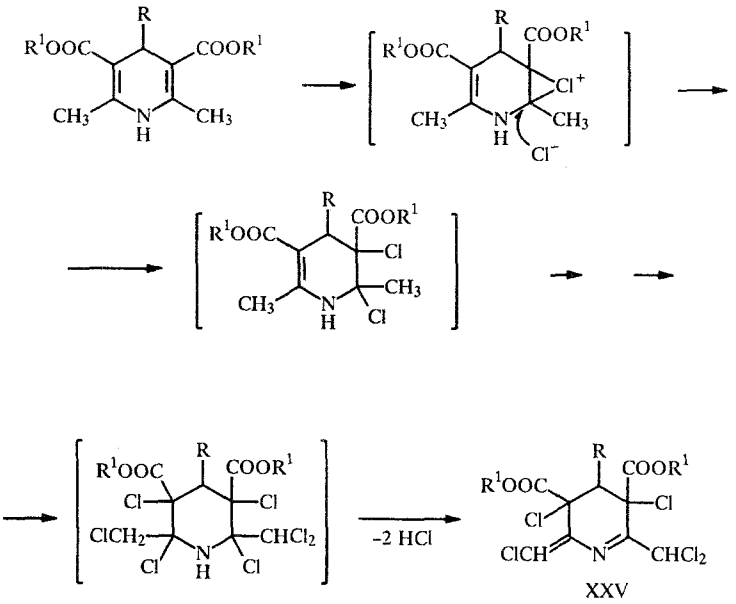
XXII XXIII



XXIV

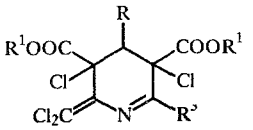
A higher degree of chlorination can be achieved only when chlorine gas is used. 2-Dichloromethyl-3,5-dimethoxycarbonyl-3,5-dichloro-4-aryl-6-chloromethylene-3,4,5,6-tetrahydropyridine (XXV) was obtained when an approximately sixfold excess of chlorine was used in the reaction with the 1,4-DHP [47]. It is evident that the addition of chloride anions rather than alkoxy groups occurs here. Since, according to the data in [48], the halogen atoms attached to the $C_{(2)}$ and $C_{(6)}$ atoms are distinguished by a high tendency to undergo splitting out, two molecules of hydrogen chloride are liberated, and an exocyclic double bond is formed as a result.

Scheme 4



XXV

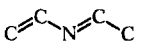
Tetrahydropyridine XXVI, which differs from analog XXV with respect to the presence of a dichloromethylene group in the 6 position, was isolated in good yield when the amount of chlorine passed into the mixture was increased to 12 moles.



XXVI R^3 = $CHCl_2$, XXVII R^3 = CCl_3

If the amount of chlorine passed into the mixture is increased to an even greater extent, the product is 3,4,5,6-tetrahydropyridine XXVIII, in which all of the hydrogen atoms in the 2-methyl and 6-methylene groups are replaced by the halogen.

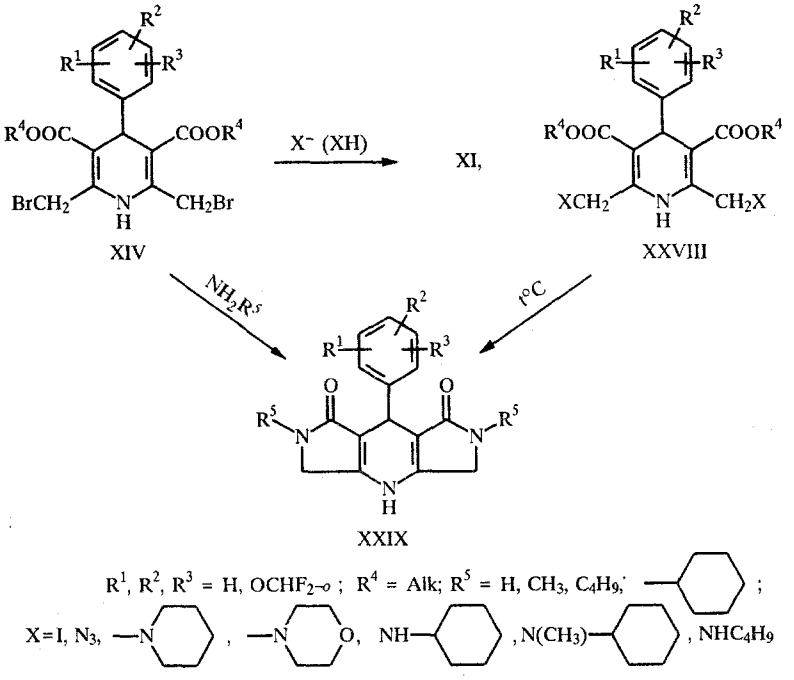
The presence in the ^{13}C NMR spectra of XX, XXII, XXIV, XXV, XXVI, and XXVIII at very weak field (157-162 ppm) of one signal and signals at 136-138 ppm is in agreement with the presence of a C—N bond and an exocyclic double bond in the molecule, which, simultaneously with the absence of a signal from an NH proton in the PMR spectrum, makes it possible to assume the presence of the fragment depicted below in the molecules:



The $C_{(3)}$ and $C_{(5)}$ atoms absorb at 60-65 ppm, which indicates the presence of two chlorine atoms in these positions of the heteroring. The change in the signals from the protons of the 2,6-methyl (methylene) groups indicates graphically the successive chlorination of precisely the latter. The protons of the CH_2Cl group give a signal in the form of a singlet at 4.54-4.56 pm, the introduction of a second chlorine atom into this group shifts the singlet to weak field to 6.41-6.77 ppm, and the signal from the CHCl group is located at 7.11-7.41 ppm.

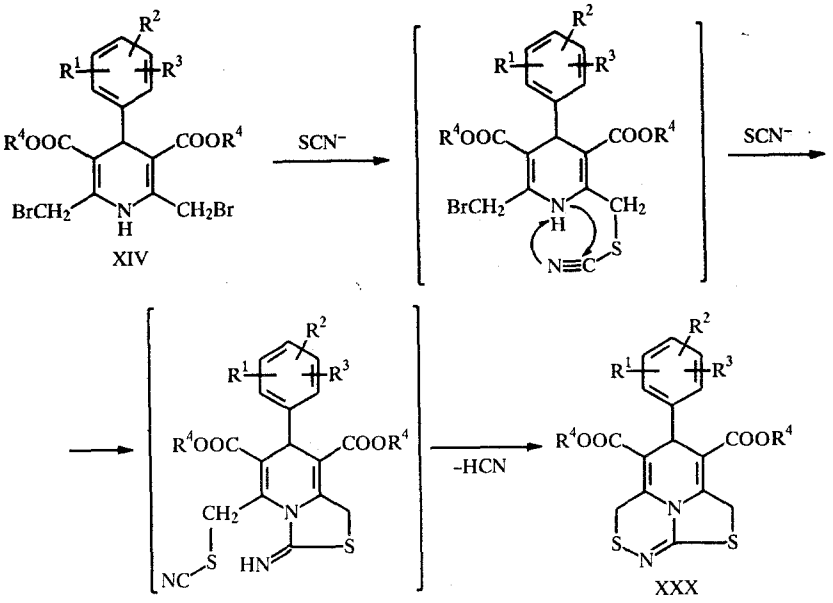
NUCLEOPHILIC SUBSTITUTION AND HETEROCYCLIZATION OF BROMINE-CONTAINING 1,4-DIHYDROPYRIDINES

Additional possibilities for the chemical modification of 1,4-DHP consist in nucleophilic-substitution reactions of 2,6-bis(bromomethyl)-1,4-DHP XIV and 2-bromomethyl-1,4,5,7-tetrahydrofuro[3,4-b]pyridine XVII with a number of nucleophilic reagents: primary and secondary amines and iodide, azide, and thiocyanate ions [41, 43].

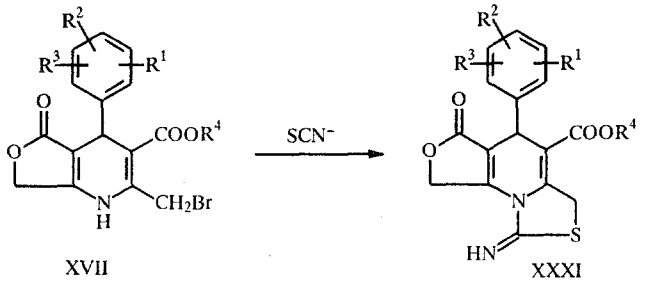


N,N′-[2,6-(3,5-Dialkoxycarbonyl-4-aryl-1,4-dihydropyridine-2,6-diyl)dimethyl]dipyridinium dibromides XI, which were previously isolated as side products in the bromolactonization with pyridinium bromide perbromide, are formed in reactions with pyridine. When ammonia or methylamine is used as the nucleophilic agent in this reaction, the products of replacement of bromine undergo cyclization so readily that only dipyrrolo[3′,4′-b;3,4-e]pyridines XXIX can be isolated even when the process is carried out at temperatures below 0°C [41]. Evidently because of steric hindrance, prolonged refluxing is required for the formation of the analogous lactams XXIX when more bulky amines are used.

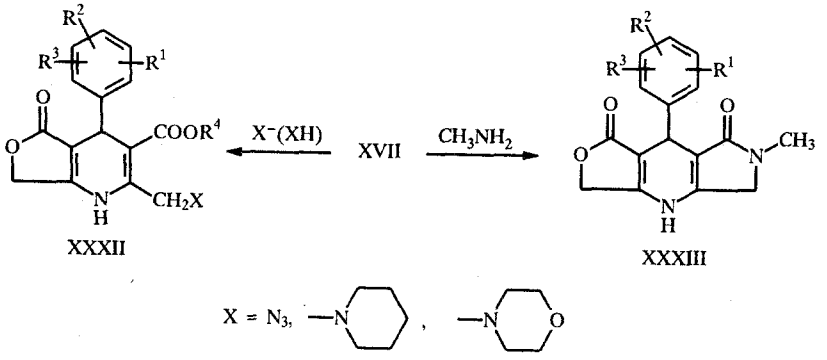
It is known that potassium thiocyanate reacts with N-substituted 2,6-bis(bromomethyl)-1,4-DHP to give nucleophilic-substitution products [41], but, judging from the IR, PMR, and ^{13}C NMR spectral data, in the analogous reaction with N-unsubstituted compounds subsequent intramolecular cyclization to give a 1,3-thiazole ring occurred after replacement of the bromine atom by a thiocyanato group. Splitting out of a molecule of hydrogen cyanide to give a six-membered ring, as a result of which a new heterocyclic system, viz., [1′,3′]-thiazolo[3′,4′:1,2]pyrido[6,1-d][1,2,4]thiadiazine XXX, was obtained, occurred when a second thiocyanato group was included in the reaction [49, 50].



For additional confirmation of the cyclization scheme, a similar synthesis with 2-bromomethyl-1,4,5,7-tetrahydrofuro[3,4-c]pyridine XVII was carried out. As one should have been expected, another previously undescribed heterosystem, viz., furo[3′,4′-e][1,3]thiazolo[3,4-a]pyridine XXXI, was formed [50].



Substitution products XXXII can be synthesized from 2-bromomethylfuropyridine XVII in reactions with sodium azide and secondary amines, but the formation of a lactam ring to give a new heterocyclic system, viz., furo[3′,4′-b]pyrrolo[3,4-e]pyridine XXXIII, occurs in the case of methylamine even with cooling of the reaction mixture and a minimal reaction time [50, 51].



Two signals of protons of methylene groups (at 4.81 ppm for the lactone ring, and at 3.98 ppm for the lactam ring) are characteristic in the PMR spectra of polycyclic 1,4-DHP XXXIII.

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