# DIELS—ALDER ADDUCTS OF FURAN DERIVATIVES IN THE SYNTHESIS OF CARBO- AND HETEROCYCLIC SYSTEMS (REVIEW)

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The possibilities of the utilization of the Diels—Alder reaction of furan derivatives in the synthesis of polysubstituted alycyclic, aromatic, and heterocyclic compounds are examined.

A great deal of data on the behavior of furan in the Diels—Alder reaction has been accumulated [1-3]. The utilization of furan and its derivatives in the diene synthesis offers rich synthetic possibilities for the preparation of a large number of compounds of various classes. In the present review the synthetic aspects of the application of Diels—Alder adducts of furan derivatives are examined, and the possibility of their use in the synthesis of alicyclic, aromatic, and heterocyclic compounds is demonstrated.

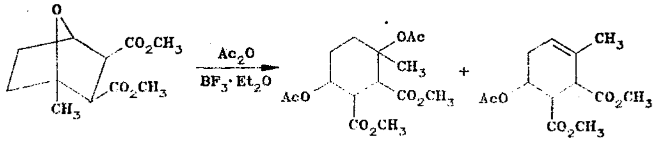
## 1. ADDUCTS OF THE DIENE SYNTHESIS OF FURAN DERIVATIVES WITH ACETYLENIC AND ETHYLENE DIENOPHILES AND THEIR TRANSFORMATIONS

Furan derivatives undergo the diene synthesis with a large number of dienophiles that contain a double or triple carbon—carbon bond to give adducts that are derivatives of 7-oxabicyclo[2.2.1]heptane. The Diels—Alder reaction is the most convenient method for the synthesis of compounds of this class, many of which have valuable biological properties [4-9].

### 1.1. Opening of the Oxygen Bridge in Hydrogenated Diels—Alder Adducts of Furan. Synthesis of Cyclohexane, Cyclohexene, and Cyclohexadiene Derivatives

Adducts of the diene synthesis of furan and their hydrogenated derivatives readily undergo opening of the oxygen bridge in the presence of protic acids or Lewis acids to give cyclohexane and cyclohexane derivatives or aromatic compounds.

In addition to 1,4-disubstituted cyclohexanes, which are formed as a result of a substitution reaction, the formation of elimination products, viz., cyclohexene derivatives, is generally observed in the cleavage of the oxygen bridge in completely hydrogenated adducts of furan. Thus I, which was obtained on the basis of the adduct of 2-methylfuran with maleic anhydride, is converted to a mixture of substitution (II) and elimination (III) products under the influence of boron trifluoride etherate in acetic anhydride [10].



I II III

A detailed study of this reaction in the case of IV showed that rearrangement products X and XI are also formed in addition to substitution (V) and elimination (VI, VII) products. The authors assumed that the reaction proceeds through acyloxonium ion VIII. Migration of a hydride ion, which leads to the formation of carbonium ion IX, explains the development of isomeric 1,5-diacetates X and XI [11].

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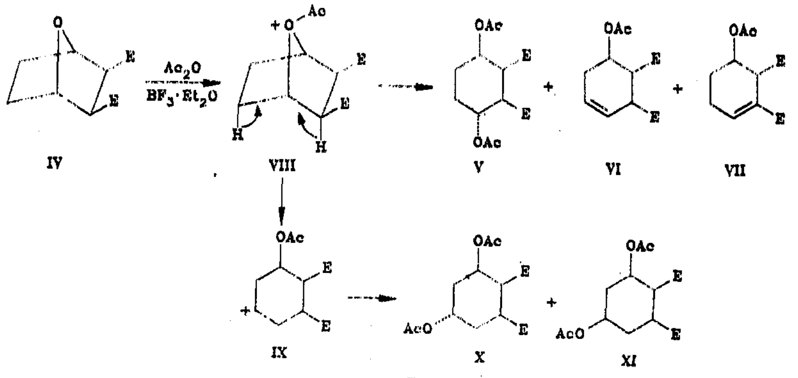
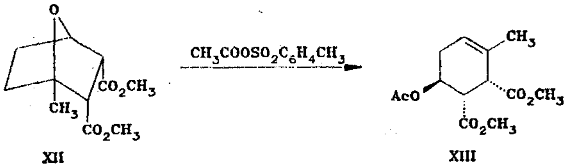


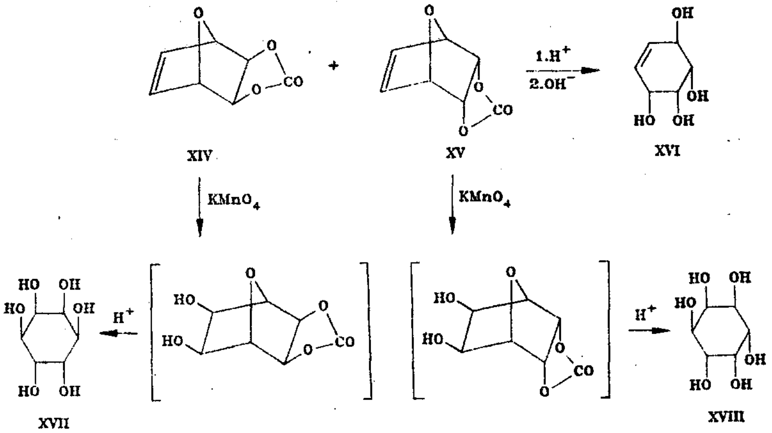
E = $CO_2CH_3$

The utilization of the mixed anhydride of acetic and p-toluenesulfonic acids as the reagent made it possible to obtain exclusively elimination product III from I [12].

The value of the method set forth above consists in the fact that it makes it possible to realize the stereospecific synthesis of cyclohexene derivatives. Thus, starting from the adduct of 2-methylfuran with maleic anhydride, which has an exo configuration, one can obtain I, which is converted to cyclohexene III with a cis orientation of the acetoxy and carbomethoxy groups. On the other hand, by hydrogenation of the adduct of 2-methylfuran with dimethyl acetylenedicarboxylate one can synthesize XII with an endo orientation of the carbomethoxy groups, which is converted under the influence of the mixed anhydride of acetic and p-toluene-sulfonic acids to XIII, in which the acetoxy group is trans-oriented relative to the carbomethoxy groups. Compound XIII was used in the synthesis of an analog of fujenic acid, which is contained in Giberella fujikuroi cultures [12].

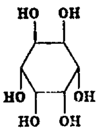


Yur'ev and Zefirov used the diene synthesis of furan with vinylene carbonate in the stereospecific synthesis of cyclitols. A mixture of the endo and exo adducts (XIV, XV) of furan with vinylene carbonate in an acidic medium is converted to condurite (XVI).

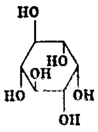


The hydroxylation of the adducts, which proceeds as exo addition, and subsequent opening of the oxygen bridge make it possible to obtain epi-inositol (XVII) and enoinositol (XVIII) [13].

A method based on the epoxidation of the double bond in adducts of furan with vinylene carbonate was later proposed for the synthesis of alloinositol (XIX) and mesinositol (XX) [14].

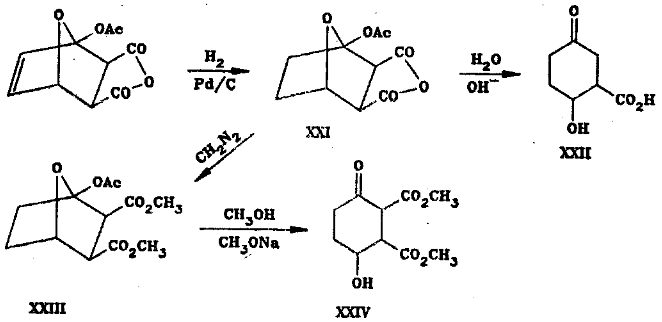


XIX

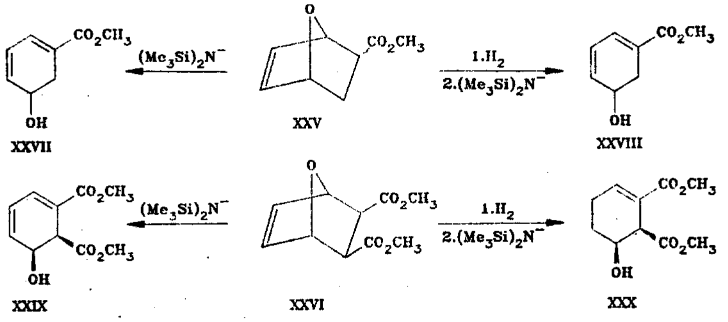


XX

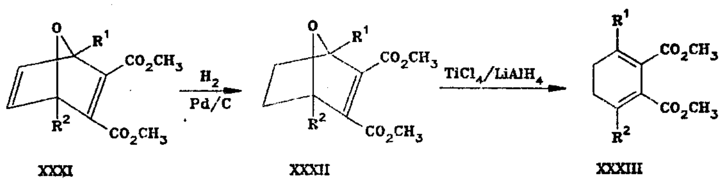
Adducts of the diene synthesis of furan and their hydrogenated derivatives are quite stable in alkaline media, whereas the hydrogenated adduct (XXI) of acetoxyfuran with maleic anhydride, which is a cyclic hemiketal, under the influence of alkalis is converted to keto acid XXII, thereby undergoing partial decarboxylation in addition to hydrolysis [15]. A method that made it possible to avoid decarboxylation was found for the synthesis of a 2,3-disubstituted 4-hydroxycyclohexanone. Methylation of adduct XXI gave dimethyl ester XXIII, which was converted to 2,3-dicarbomethoxy-4-hydroxycyclohexanone (XXIV) by the action of sodium methoxide in alcohol [16].



It was recently shown that adducts (XXV and XXVI) of unsubstituted furan with ethylene dienophiles and their hydrogenated derivatives under the influence of a strong base, viz., lithium bis(trimethylsilyl)amide, are also capable of undergoing opening of the oxygen bridge to give cyclohexadiene derivatives XXVII and XXIX and cyclohexane derivatives XXVIII and XXX, respectively [17].



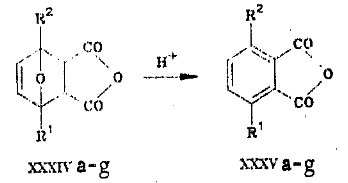
Adducts of furan derivatives with dimethyl acetylenedicarboxylate were also used in the synthesis of cyclohexadiene derivatives. Partial hydrogenation of adducts XXXI with subsequent treatment with $TiCl_4/LiAlH_4$ made it possible to obtain substituted cyclohexadienes XXXIII in high yields [18].



## 1.2. Aromatization of Adducts of the Diene Synthesis of Furan Derivatives with Ethylene and Acetylenic Dienophiles

7-Oxabicyclo[2.2.1]heptene derivatives, which are formed directly via the diene synthesis of furan with ethylene dienophiles or by partial hydrogenation of adducts of furan with acetylenic dienophiles, are converted to aromatic compounds that do not contain hydroxy groups under the influence of acids and sometimes even spontaneously. Under these conditions adducts of furan with acetylenic dienophiles that are 7-oxabicyclo[2.2.1]heptadiene derivatives are converted to phenol derivatives.

1.2.1. Synthesis of Aromatic Compounds That Do Not Contain Hydroxy Groups. The aromatization of adducts of furan derivatives with maleic anhydride has been used for the synthesis of substituted phthalic acids. This transformation was accomplished for the first time by heating the adduct (XXXIVa) of furan with maleic anhydride in acetic acid containing hydrogen bromide [19].

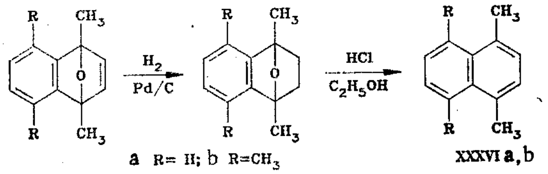


XXXIV, XXXV a,b,d,e $R^1=H$, c,g $R^1=CH_3$, f $R^1=-(CH_2)_n-Ar$ ($n=1,2$); a,f $R^2=H$, b,c $R^2=CH_3$, d $R^2=OCOCH_3$, e,g $R^2=OCH_3$

Anhydrides (XXXVb,c) of 3-methyl- and 3,6-dimethylphthalic acids were synthesized with the use of an aqueous solution of sulfuric acid for aromatization [20, 21]. Aromatization was subsequently carried out by the action of sulfuric acid in sulfolane, which made it possible to raise the yield of the final anhydride [22]. The adduct (XXXIVd) of acetoxyfuran with maleic anhydride was converted to substituted phthalic anhydride XXXVd by heating in acetic anhydride containing sulfuric acid [23]. The atomatization of adduct XXXIVe was used to obtain 3-methoxyphthalic acid anhydride (XXXVe), which is the starting compound for the synthesis of the anthracycline antibiotics daunomycin and adriamycin [24]. This method was used to obtain substituted phthalic anhydrides XXXVf, which were converted to condensed aromatic compounds via intramolecular cyclization [25-27].

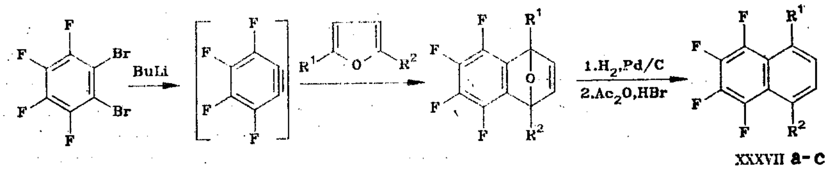
The conversion of adducts of furan with maleic anhydride to aromatic derivatives also occurred in a number of cases even in the absence of acid catalysts. Thus adducts XXXIVg was converted to substituted phthalic anhydride XXXVg during recrystallization from methanol [28], and aromatization occurred in the process of the reaction of alkyl 2-furyl sulfides with maleic anhydride [29].

Adducts of furan with acetylenic dienophiles have also been used in the synthesis of aromatic derivatives that do not contain hydroxy groups. Prior hydrogenation of one double bond in adducts of furan with dehydrobenzene and subsequent aromatization of the hydrogenated adducts made it possible to obtain naphthalene derivatives. This method for the conversion of 1,4-epoxynaphthalene to naphthalene was realized for the first time by Wittig [30]. This method was subsequently used in the synthesis of 1,4-dimethyl- [31] and 1,4,5,8-tetramethylnaphthalene (XXXVIa, b) [32].



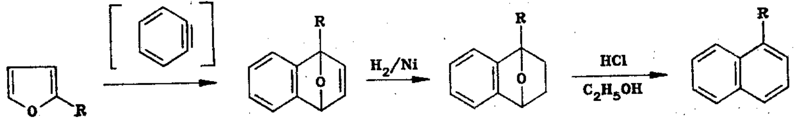
Franck and Leser [33] synthesized 1,4,6,8- and 1,3,6,8-tetra-tert-butylnaphthalene using 2,4-di-tert-butylanthranilic acid as the source of dehydrobenzene and 2,5- and 2,4-di-tert-butylfuran as the furan components.

Tetrafluoronaphthalenes XXXVIIa-c were obtained on the basis of tetrafluorodehydrobenzene and substituted furans [34].

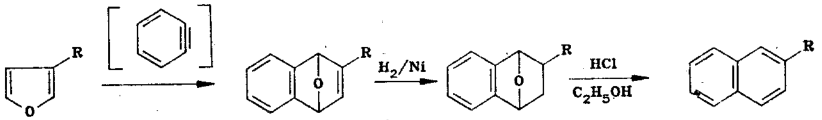


a $R^1=H$, b,c $R^1=Me$; a,b $R^2=Me$, c $R^2=Bu$

Optically active naphthalene derivatives XXXVIII and XXXIX were obtained by means of the diene synthesis on the basis of optically active furan derivatives, viz., (S)-2-(2- and 3-furyl)butanes, and dehydrobenzene [35].



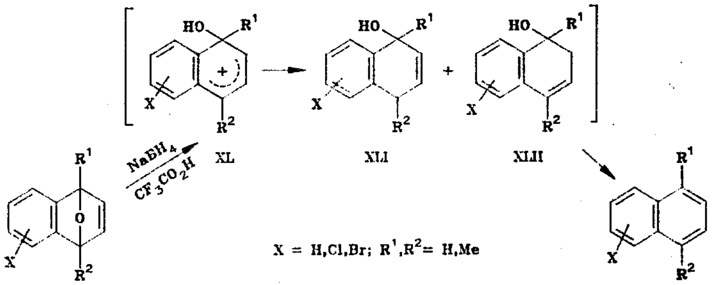
XXXVIII



R = $\overset{*}{C}HCH_3(C_2H_5)$

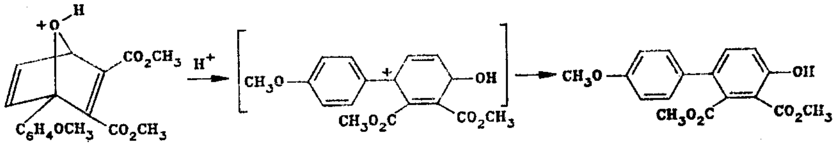
XXXIX

Various reducing agents have been used in recent years for the deoxygenation of adducts of furan with acetylenic dienophiles to aromatic compounds that do not contain hydroxy groups. Thus a method was developed for the reduction of adducts of furan with dehydrobenzene by the action of sodium borohydride in trifluoroacetic acid [36]. It is assumed that the reaction commences with acid-catalyzed opening of the oxygen bridge to give carbonium ion XL, which is reduced to XLI and XLII. This is followed by acid-catalyzed dehydration to the corresponding naphthalene.

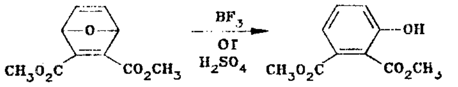


Titanium tetrachloride has been used in conjunction with LiAlH$_4$ for the deoxygenation of adducts of furan with acetylenecarboxylic acid ester [37]. Iron pentacarbonyl, iron, and other reducing agents have also been used as reagents for this transformation [38, 39].

1.2.2. Synthesis of Substituted Phenols and Naphthols. Adducts of furan with acetylenic dienophiles are converted to aromatic derivatives that contain hydroxy groups under the influence of protic acids and Lewis acids. This transformation has been widely used for the synthesis of substituted phenol and naphthols that are difficult to obtain by other methods. The ability to undergo this transformation is determined to a significant degree by the character of the substituents located at the bridgehead of the adduct. It has been shown [40] that the adduct of 2-(p-anisyl)furan with acetylenedicarboxylic acid ester is converted quantitatively to a phthalic acid derivative upon heating in acetic acid. Ayres and Smith [40] linked the ease of this transformation to stabilization of the intermediately formed carbonium ion by the methoxyphenyl substituent.

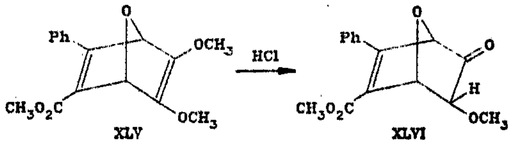


The absence of substituents at the bridgehead significantly increases the stabilities of the adducts with respect to reagents with acidic character. Thus the adduct (XLIII) of unsubstituted furan with dimethyl acetylenedicarboxylate is stable in solution in trifluoroacetic acid in the cold but undergoes retrodiene decomposition upon heating [41]. It was subsequently shown that adduct XLIII is converted to a hydroxyphthalic acid ester under the influence of sulfuric acid or boron trifluoride [42].

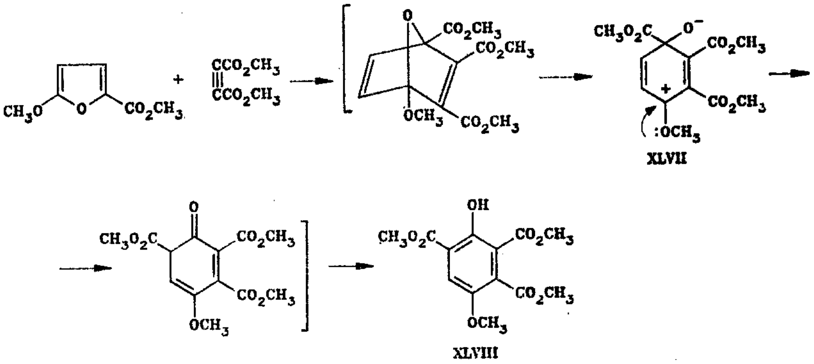


XLIII XLIV

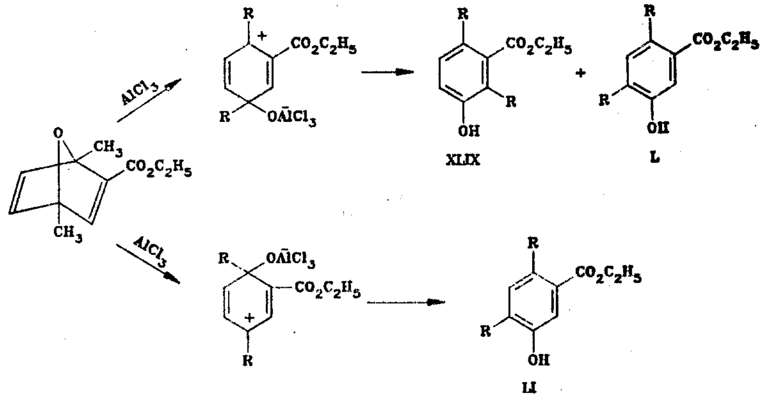
The adduct (XLV) of 3,4-dimethoxyfuran with methyl phenylpropiolate does not undergo aromatization in an acidic medium but is converted to ketone XLVI [43].



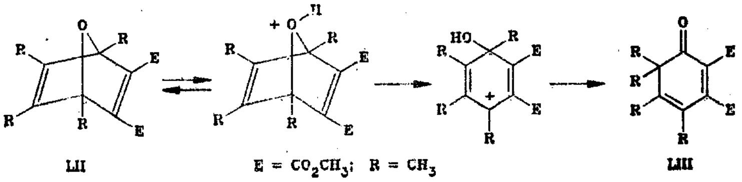
Adducts of 2,5-disubstituted furans with dimethyl acetylenedicarboxylate undergo aromatization accompanied by migration of the substituent to the position adjacent to the hydroxy group [41, 42, 44]. In the case of unsymmetrically substituted furans, in which the formation of two isomers is possible, the direction of the reaction is determined by the stability of the intermediately formed carbonium ion. As a result of stabilization of carbonium ion XLVII by a methoxy group, aromatization occurs during the reaction and leads exclusively to XLVIII [45].



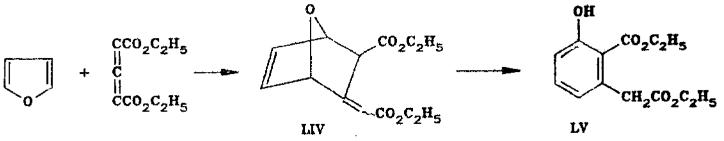
Adducts of 2,5-disubstituted furans with propiolic acid ester are converted to mixtures of isomers XLIX-LI under the influence of aluminum chloride [46].



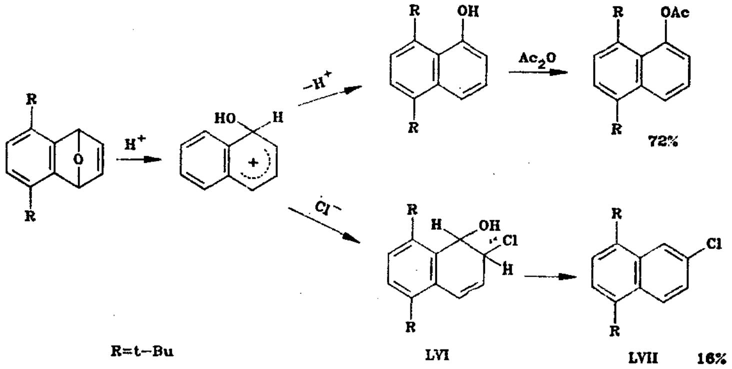
Adducts (LII) of tetrasubstituted furans with acetylenedicarboxylic acid ester are converted to cyclohexadienones LIII under the influence of acids [41].



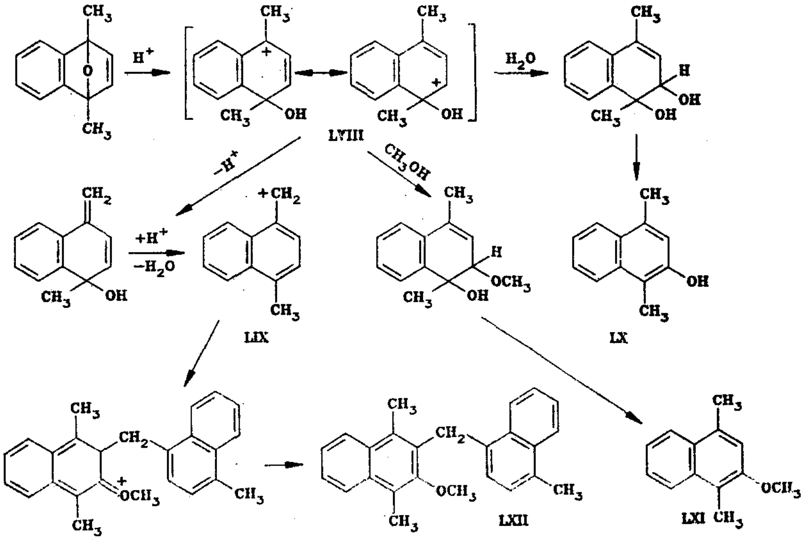
Dicarbethoxyallene, which is also capable of acting as a dienophile, reacts with furan to give adduct LIV, which is converted to arylacetic acid ester LV in the presence of acids [47].



Adducts of furan derivatives with dehydrobenzene undergo aromatization in the absence of substituents at the endoxo bridgehead. 1,4-Dihydro-1,4-endoxonaphthalene undergoes quantitative conversion to α-naphthol upon heating in a methanol solution of hydrogen chloride [30]. Under these conditions 1,4-endoxonaphthalene with tert-butyl groups in the 5 and 8 positions is converted to a mixture of 1- and 2-substituted naphthalenes [48].

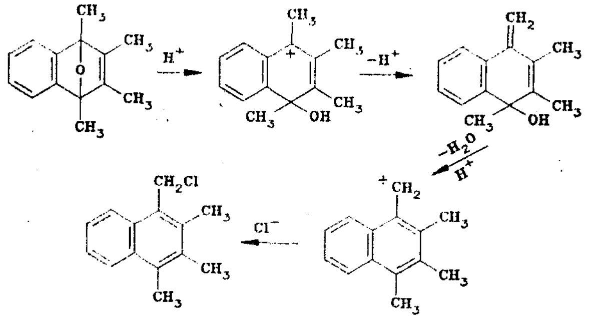


The authors explain the observed effect by assuming that aromatization of the adduct leads to shortening of the $C_{(1)}-C_{(9)}$ bond and drawing together of the hydroxy and tert-butyl groups, thereby increasing the energy of activation of elimination of a proton. This increases the contribution of the competitive addition of a nucleophile, leading to dihydronaphthalene LVI, which is converted to naphthalene LVII by splitting out water.

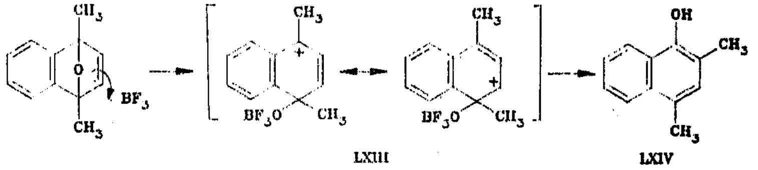


When there is a substituent in the 1 and 4 positions of 1,4-endoxonaphthalene, the reaction proceeds ambiguously. The adduct of 2,5-dimethylfuran with dehydrobenzene is converted to a mixture of LX-LXII under the influence of a methanol solution of hydrogen chloride.

Compounds LX and LXI are formed as a result of the addition of a molecule of water or alcohol to carbonium ion LVIII. The authors explain the formation of LXII by the intermediate formation of cation LIX, which acts as an electrophile in the electrophilic substitution of 1-methoxynaphthalene LXI. The scheme for the formation of LXII presented above is confirmed by the fact that tetramethyl-1,4-endoxonaphthalene is converted to 1-chloromethyl-2,3,4-trimethylnaphthalene under these conditions [49].



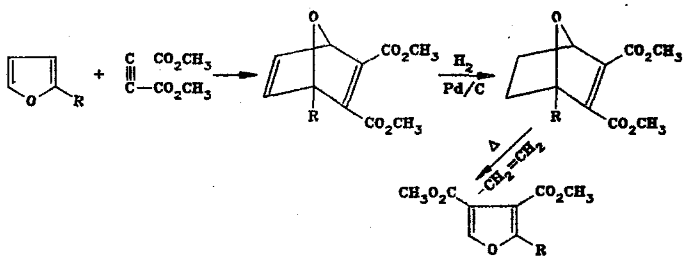
In the absence of a nucleophile, opening of the oxygen bridge to give carbonium ion LXIII is accompanied by migration of a methyl group and leads to the formation of 1-naphthol LXIV [50].



Polysubstituted aromatic compounds obtained by aromatization of the adducts of the diene synthesis of furan derivatives have been used as the starting compounds in the synthesis of a number of condensed aromatic and heterocyclic systems [51-54].

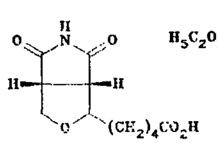
## 1.3. Retrodiene Decomposition of Diels—Alder Adducts of Furan Derivatives

The ability of adducts of the diene synthesis to undergo retrodiene decomposition is widely used in synthetic organic chemistry. An important aspect of this sort of utilization of Diels—Alder adducts in the furan series is the preparation of 3,4-disubstituted furans from them. The difficulty involved in the introduction of substituents into the β position of the furan ring makes this method one of the most convenient methods for the synthesis of furan derivatives that contain substituents in the β position. The partial hydrogenation of adducts of furan with acetylenedicarboxylic acid ester, which proceeds selectively at the unsubstituted double bond, and subsequent thermal decomposition of the hydrogenated adducts make it possible to obtain furan derivatives with ester groups in the β position [55-57].

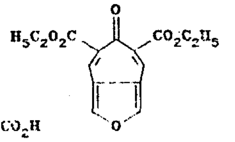


R=H, CH3, Ar

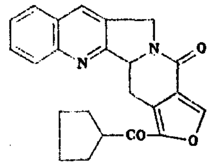
β,β-Dicarbomethoxyfurans have been used in the synthesis of a large number of condensed heterocyclic compounds containing a furan ring, including biotin analog LXV [58], furotropone LXVI [59], and LXVII, which has served as an intermediate in the synthesis of the alkaloid camptothecin [60]. It proved to be possible to synthesize a number of condensed heterocyclic systems, viz., cycloheptafuropyridone LXVIII, furopyridazinone LXIX, naphthofuropyridone LXX, furobenzothiepine LXXI, etc., by varying the substituents in the α position of the furan ring and by using retrodiene decomposition for the introduction of ester groups into the β position [61-63].



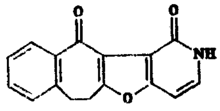
LXV



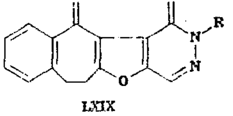
LXVI



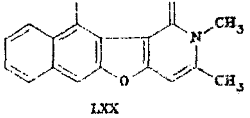
LXVII



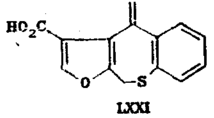
LXVIII



LXIX

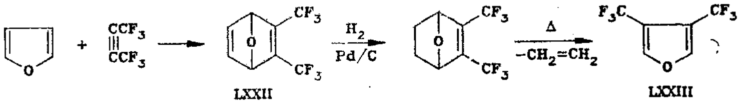


LXX

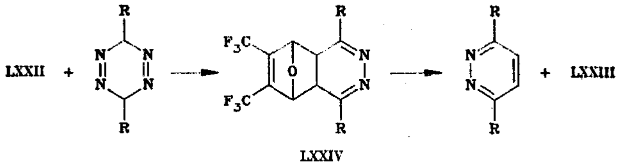


LXXI

The utilization of hexafluoro-2-butyne as the dienophile made it possible to obtain a β-(trifluoromethyl) derivative (LXXIII) of furan [64].



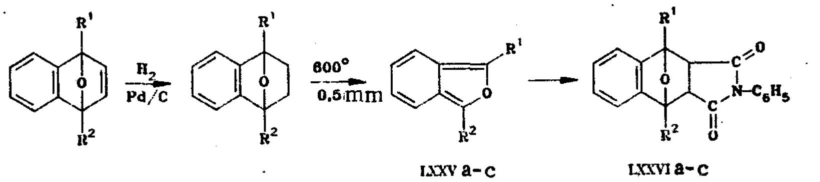
A new method for the transformation of adduct LXXII under milder conditions was later proposed. The adduct was treated with 3,6-di(2-pyridyl)-s-tetrazine. The intermediately formed LXXIV decomposed at room temperature to give β-substituted furan LXXIII [65].



R= 2-pyridyl

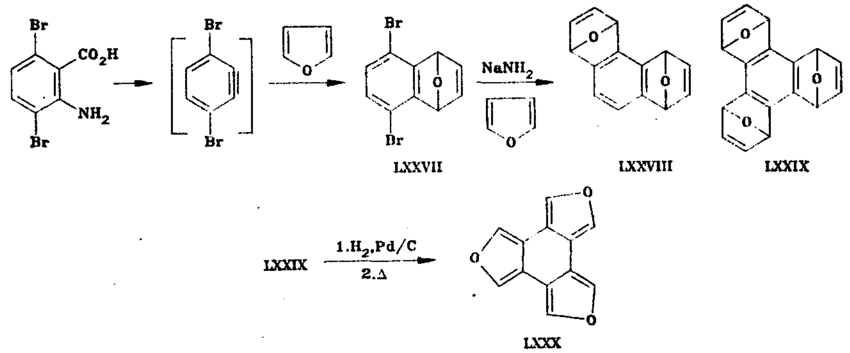
The bromination of the adduct of furan with maleic anhydride with subsequent decomposition made it possible to obtain 3-bromofuran [66, 67].

In addition to the preparation of β-substituted furans, other examples of the utilization of the retrodiene decomposition of adducts of furan with various dienophiles have been described in the literature. Thus the decomposition of the hydrogenated adduct of furan with dehydrobenzene made it possible to synthesize unstable isobenzofuran LXXV, which was then converted to Diels—Alder adduct LXXVI [68, 69].

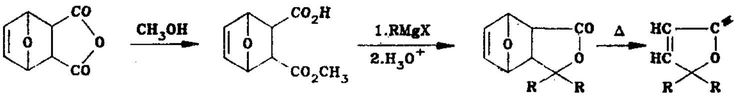


a $R^1=CH_3$, $R^2=H$; b $R^1=R^2=CH_3$; c $R^1=CH_2C_6H_5$; $R^2=H$

Retrodiene decomposition was used in the synthesis of triphenylene analog LXXX. The adduct (LXXVII) of furan with dibromodehydrobenzene was converted to a mixture of adducts LXXVIII and LXXIX under the influence of sodium amide in the presence of furan. Hydrogenation and subsequent retrodiene decomposition of adduct LXXIX lead to triphenylene analog LXXX [70].

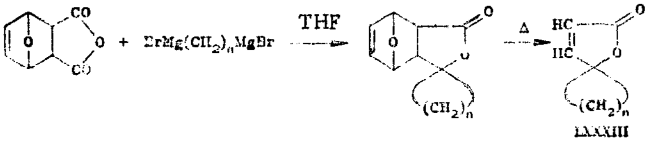


The retrodiene decomposition of LXXXI, obtained from the adduct of furan with maleic anhydride, made it possible to obtain 4-substituted 2-butenolide LXXXII [71, 72].

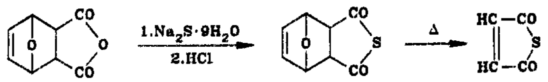


R= alkyl, alkyl, aryl

Spirolactone LXXXIII was obtained by the reaction of the alkylenedimagnesium dibromide with the adduct of furan with maleic anhydride and subsequent retrodiene decomposition [73,

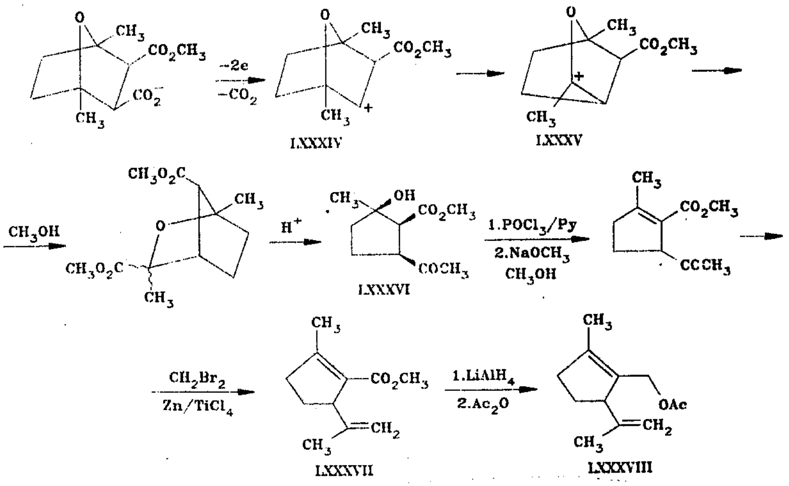


The adduct of furan with maleic anhydride was used in the synthesis of thiomaleic anhydride [75].

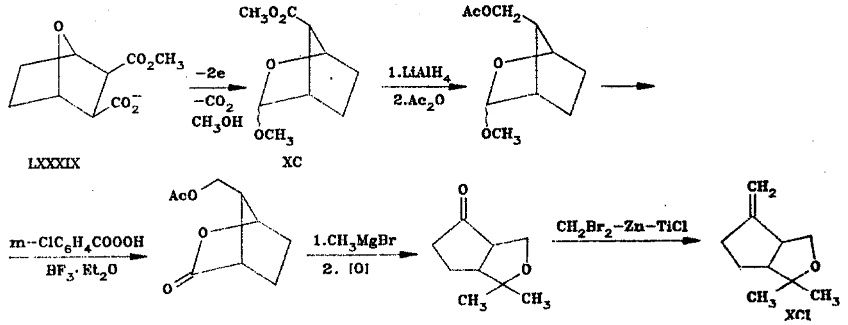


## 1.4. Wagner–Meerwein Rearrangement. Synthesis of Cyclopentane Derivatives.

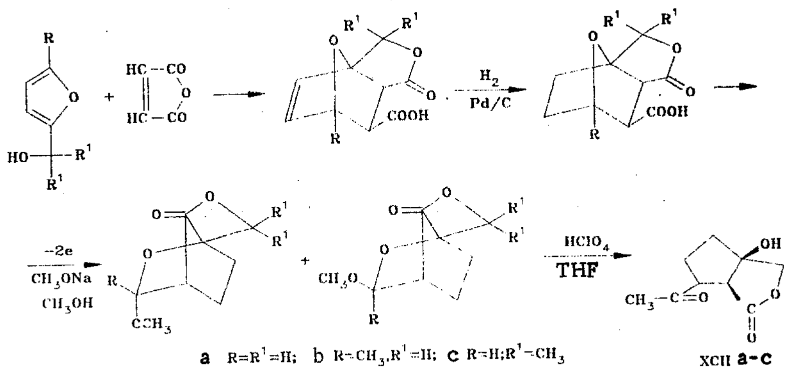
The Wagner–Meerwein rearrangement in the 7-oxobicyclo[2.2.1]heptane series makes it possible to synthesize cyclopentane derivatives that are difficult to obtain [76, 77]. Thus a stereoselective method was developed for the synthesis of 1,2,3-trisubstituted cyclopentanes; this method was based on the Wagner–Meerwein rearrangement of 7-oxobicyclo[2.2.1]hept-2-yl cation LXXXIV, obtained by electrolysis of a 3-carbomethoxy-7-oxobicyclo[2.2.1]heptane-2-carboxylic acid salt. As a result of the rearrangement, cation LXXXIV was converted to cation LXXXV. The resulting 1,2,3-trisubstituted cyclopentane LXXXVI was converted to 3-isopropenyl-1-methylcyclopentenes LXXXVII and LXXXVIII, which are starting compounds in the synthesis of terpenes [78, 79].



Compound XC, which was obtained by the Wagner–Meerwein rearrangement under the conditions of the electrolysis of the LXXXIX anion, was used in the synthesis of ether XCI, which has been isolated from Japanese hops [80].

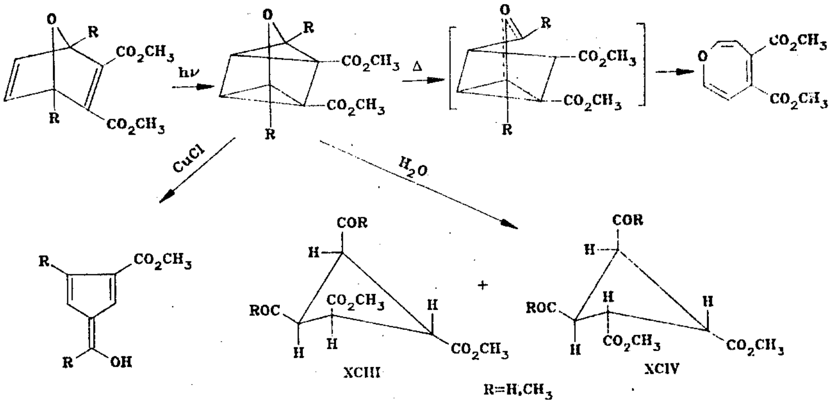


Japanese chemists have synthesized a number of other 3-oxabicyclo[3.3.0]octane derivatives XCII also using a method based on the Wagner–Meerwein rearrangement [81].



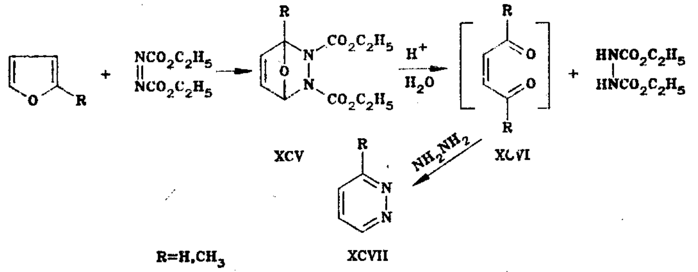
### 1.5. Photochemical Transformations of the Diels—Alder Adducts of Furan Derivatives

Adducts of the diene synthesis of furan with acetylenic dienophiles are capable of undergoing photochemical isomerization to 3-oxaquadricylanes [41, 82-86]. The latter, in turn, are used in the synthesis of oxepines [82], isomeric cyclobutanes XCIII and SCIV [87], and fulvenes [88-91].

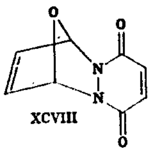


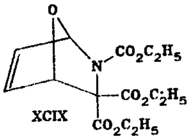
## 2. ADDUCTS OF THE DIENE SYNTHESIS OF FURAN DERIVATIVES WITH HETERODIENOPHILES

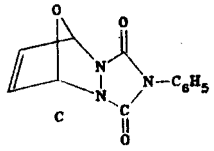
The investigation of the reaction of furan with heterodienophiles was begun relatively recently, and a limited number of studies have been devoted to this question. Furan derivatives react with diethyl azodicarboxylate to give adducts XCV, which are readily hydrolyzed in an acidic medium to give diethyl hydrazodicarboxylate and dicarbonyl compounds XCVI, which are converted to pyridazines XCVII upon treatment with hydrazine [92-94].



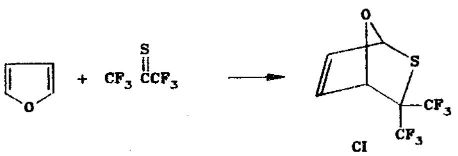
The reaction of unsubstituted furan with diazaquinone [95], diethyl ethoxycarbonyliminomalonate [96], and N-phenyltriazolinedione [97] to give adducts XCVIII-C has been described.


XCVIII

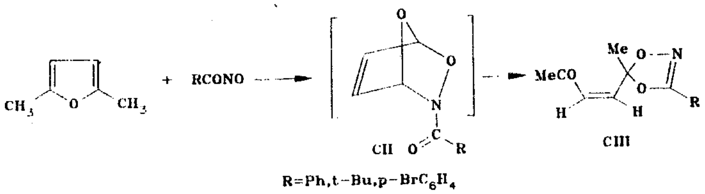

XCIX


C

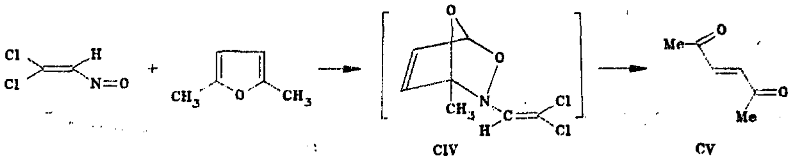
Hexafluorothioacetone is also capable of acting as a dienophile; it reacts with furan to give addcut CI [98].



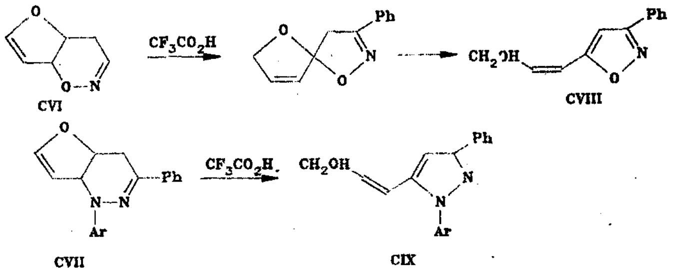
The reaction of furan derivatives with nitrosocarbonyl compounds, which leads to substituted 1,4,2-dioxazoles CIII, also possibly proceeds through Diels—Alder adduct CII [99].



The diene synthesis of a number of dienes, including furan, with nitroso and aza olefins that are capable of acting as $2\pi$ and $4\pi$ components of the Diels–Alder reaction has been investigated [100]. It has been previously shown that β,β-dichloronitrosoethylene reacts with 2,5-dimethylfuran to give hexenedione CV. The authors assume that β,β-dichloronitrosoethylene acts as a dienophile in this reaction to give adduct CIV, which is then converted to CV [101].



As a rule, nitroso and aza olefins act as $4\pi$ components in the Diels–Alder reaction; the reaction proceeds readily if the nitroso and aza olefins contain electron-acceptor substituents. Furan derivatives react as $2\pi$ components with nitroso and aza olefins to give furooxazines CVI and furopyridazines CVII, respectively [100], which undergo rearrangement to isoxazoles CVIII and pyrazoles CIX under the influence of trifluoroacetic acid [102].



Thus the heterodiene synthesis with the participation of furan derivatives offers the possibility of the preparation of complex bridged heterocyclic systems, as well as difficult-to-obtain condensed systems, that contain a furan ring.

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