SYNTHESIS OF HETEROCYCLIC COMPOUNDS ON THE BASIS OF CYCLOHEXANE-1,3-DIONES (REVIEW)

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and I. A. Strakova

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The literature data for the period 1973-1985 on the synthesis, on the basis of cyclohexane-1,3-diones and their derivatives, of heterocyclic compounds that contain a 3-oxocyclohexene structural fragment or are formed as a result of a change in the cyclohexane ring are examined.

In developing our previous review [1] we have correlated data on the synthesis of heterocycles on the basis of cyclohexane-1,3-diones for the period 1973-1985; data on the formation of heterocycles as a result of opening of the cyclohexane skeleton of the molecule are also presented. The material in the review is systematized with respect to classes of heterocycles; spirocycles, to which a separate section is devoted, constitute an exception.

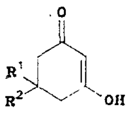
BENZOFURANS AND DIBENZOFURANS

Known methods (see [1]) for the synthesis of benzofurans from cyclohexane-1,3-diones 1 and α-halo carbonyl compounds were used in [2-5]. The action of nucleophilic agents on 2-(chloroacetyl)cyclohexane-1,3-diones leads to 3,4-dioxo-2,3,4,5,6,7-hexahydrobenzofurans [6]. New examples of the synthesis of benzofuran derivatives from diketones 1 and nitro olefins have been published [7-9]. Benzofuran 2 was obtained from dimedone and trichloronitroethylene [10].

The reaction of 2,2-dibromocyclohexane-1,3-diones with olefins and acetylenes in the presence of powdered copper leads to benzofuran derivatives [11-13]. 2,3-Dihydro-4-hydroxybenzofurans were obtained by heating 2-allyl-2-chlorocyclohexane-1,3-diones in DMF [14].

Dibenzofuran derivatives 3 are formed as a result of the anode electrochemical oxidation of mixtures of diketones I and pyrocatechol [15, 16]. Dibenzofuran hydroxy derivatives 4 were obtained from diketones 1 or their enamines and quinones [17, 18]. The condensation of 4-arylcyclohexanones with cyclohexanediones 1 in the presence of p-toluenesulfonic acid leads to dibenzofurans 5 (n = 4) [19].

The thermolysis of aminobicyclo[n.1.0]alkanes with dimedone gives cycloalkano[b]benzofurans 5b [20].



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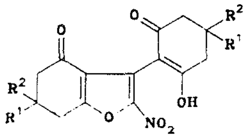

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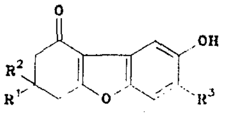
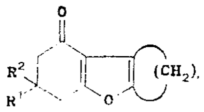


3 R^3=OH; 4 R^3=H



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INDOLES AND CARBAZOLES

The cyclization of the corresponding enamines has been most widely used for the synthesis of indoles and carbazoles on the basis of cyclohexanediones. The N-substituting group should, of course, contain a function that is suitable for reaction in the 2 (or 4) position of the cyclohexane ring. In the case of N-(o-haloaryl)enamines 6 (X = Hal) the cyclization is car-

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*Here and subsequently: a) $R^1 = R^2 = H$; b) $R^1 = R^2 = CH_3$; c) $R^1 = H$, $R^2 = C_6H_5$.

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ried out by the action of NaH and CuI [21, 22] and $Pd(OAc)_2$ and PPh_3 [23]. N-Aryl-N-acetoxyenamines 6 (X = H, R^3 = OAc) are converted to carbazoles 7 by the action of trifluoroacetic acid [24] (also see [25]).

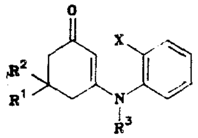
Enamines 8, in the β position of which there is a carbonyl function, undergo smooth cyclization to indoles. The reactions between diketones 1 and amino sugars are also included in this type of synthesis of indoles [26]. Aminoacetaldehyde acetals have been used for the synthesis of 3,4-unsubstituted 4-oxo-4,5,6,7-tetrahydroindoles [27].

Compounds of the 11 type were obtained from N-allyl derivatives 9 as a result of intramolecular photochemical 2 + 2 cycloaddition [28, 29].

It has been shown [30] that the cyclization of (β-bromoethyl) enamine 10 under the influence of $LiN[CH(CH_3)_2]_2$ in tetrahydrofuran gives indole derivative 12.

Refluxing enamines 6 (R^3 = X = H) with diacylethylenes in acetic acid leads to indole derivatives 13 [31]. These enamines react with pyruvic acid to give hydroxyindoles 14 [32].

2-Methyl-4-oxo-4,5,6,7-tetrahydroindole was obtained by thermolysis of 3-azido-2-allyl-2-cyclohexenone [33].



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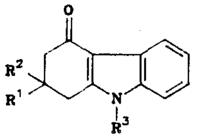

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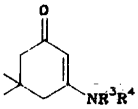

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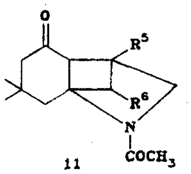

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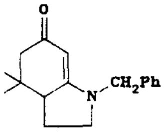

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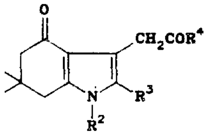

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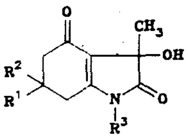

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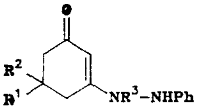
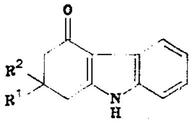
8 R^3=CHR⁵—COR⁶, R^4=H, Alk; *9* $R^3=CH_2—CR^5=CHR^6$, R^4=Ac; *10* $R^3=CH_2CH_2Br$, $R^4=CH_2Ph$

The behavior of enehydrazine 15 under the conditions of the Fischer reaction was studied in detail [34]. A mixture of carbazoles 17 and 18 is formed in the presence of sulfuric acid, while only 17 is obtained when the reaction is carried out in the presence of toluenesulfonic acid. The addition of ethylene glycol to the reaction mixture leads to the formation of carbazole 18. The reaction of enehydrazine 16 with cyclohexanone gives a mixture of carbazoles 19 and 20 [35].

Quinuclidines of the 21 type undergo rearrangement with cleavage of the quinuclidine ring and the formation of 5,6-dihydroindolizine structure 22 when they are heated with acetic anhydride [36].



15 R^3=H; 16 R^3=Ph



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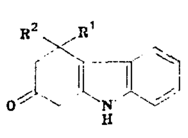

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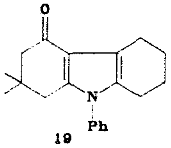

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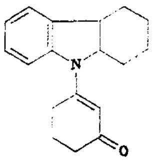

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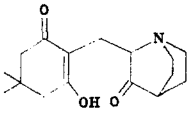

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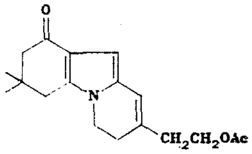

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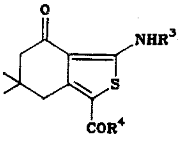
BENZOTHIOPHENES

2-Thiocarbamoylcyclohexane-1,3-diones react with bromomethyl ketones to give benzo[c]thiophene derivatives 23 [37].

Benzo[b]thiophene derivatives 24 were obtained by heating 3-dicyanomethylenecyclohexanones, obtained from cyclohexane-1,3-diones and malononitrile, with morpholine polysulfide or sulfur in the presence of morpholine [38].



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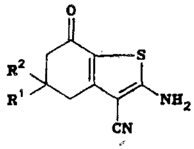

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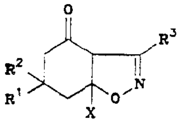
BENZOISOXAZOLES AND BENZOXAZOLES

The series of studies by Minsk chemists under the supervision of Akhrem dealing with the preparation of benzoisoxazoles and their reductive cleavage is included among the most important research in the entire field of the synthesis of heterocycles on the basis of cyclohexane-1,3-diones during the period covered by this review. Oxazolines 25, which are subsequently readily converted to benzoisoxazole derivatives 26, were obtained as a result of the cycloaddition of nitrile oxides to cyclohexenones, cyclohexane-1,3-diones, ethers of their enols, and enamines [39-42].

Only individual studies dealing with the synthesis of benzoxazoles have been published. Formylamino derivative 27, which undergoes cyclization to benzoxazole 29 under the influence of phosphoric anhydride, was obtained by the catalytic reduction of 2-nitrodimedone in a solution of the mixed anhydride of formic and acetic acids. In an acidic medium benzoxazole 29 is readily hydrolyzed to amide 27, whereas it reacts with ammonia to give enamine 28. Compounds 27 and 28 are readily converted to benzimidazole 30 [43].

A method for the synthesis of 2-acyloxy derivatives 31 from enamines of cyclohexane-1,3-diones and diacyl peroxides has been developed. Carbonates 31 (R^4 = OAlk) give oxazolones 32 under the influence of potassium hydroxide [44].



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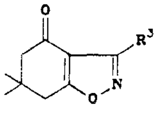

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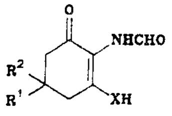

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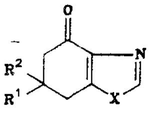

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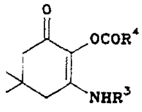

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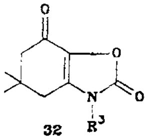

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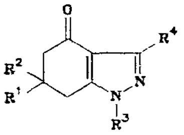
27, 29 X=O; 28, 30 X=NH

INDAZOLES AND OTHER PYRAZOLE-CONTAINING CONDENSED SYSTEMS

Indazoles 33 were obtained from hydrazines and 2-acylcyclohexane-1,3-diones or their derivatives [45-49]. A general method for the synthesis of 1,3-disubstituted 4-oxo-4,5,6,7-tetrahydroindazoles by the action of monosubstituted hydrazones of aldehydes on diketones 1 in refluxing benzene has also been developed [35, 50, 51]. This method is particularly convenient for the synthesis of 3-aryl-4-oxo-4,5,6,7-tetrahydroindazoles.

Pyrazole dioxide 34 was obtained by oxidation of dimedone dioxime with lead tetraacetate [52].



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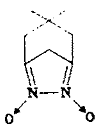

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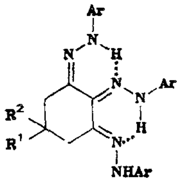
FIVE-MEMBERED HETEROCYCLES WITH THREE HETEROATOMS

Triazoles 36 and other products were obtained in the oxidation of tris(arylhydrazones) 35 with lead tetraacetate [53].

The simultaneous oximation and nitrosation of dimedone with subsequent treatment of the reaction product with trimethyl phosphite leads to furazan 37 [54]. Three-ring systems 38-43 were obtained in the reaction of dimedone dioxime or bis(phenylhydrazone) with selenium dioxide or sulfur chlorides [55-57].



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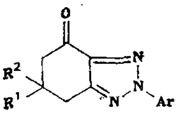

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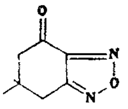

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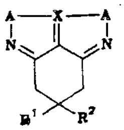


38 – 43

38 X=Se, A=O; *39* X=Te, A=O; *40* X=S, A=O; *41* X=Se, A=NPh; *42* X=Te, A=NPh; *43* X=S, A=NPh

BENZOPYRANS AND XANTHENES

Many original methods for the synthesis of these types of compounds have been published, which is associated with the increased interest in pyran derivatives, but the number of studies dealing with the synthesis of symmetrical xanthenes from diketones 1 and aldehydes has decreased. 5-Methyl- [58] and 5-(p-methoxyphenyl)cyclohexane-1,3-diones [59], aldehydes of the acetylenic series [60], and p-[N,N-bis(2-cyanoethyl)amino]benzaldehyde [61] have been subjected to the latter reaction; the synthesis has been realized using polymeric aminopropanesulfonic acids [62].

Methods involving catalytic and ionic hydrogenation have been used for the synthesis of hydrogenated benzopyran derivatives from 2-(3-oxoalkyl)cyclohexane-1,3-diones [63-66].

Coumarin derivatives 44 were obtained from diketones 1 and β-ketoglutaric acid esters [67, 68].

The reaction of 2-acylcyclohexane-1,3-diones with malononitrile has been studied in quite some detail. (Some of the material has already been published in reviews [69].) Quinoline and isoquinoline derivatives are primarily formed in these reactions (see below), but coumarin derivatives 45 have been obtained along with quinoline derivatives from the reactions of 2-acetyl- [70] and 2-formylcyclohexane-1,3-diones [71] with malononitrile. In contrast to other 2-formylcyclohexane-1,3-diones, 2-formyl-5-phenylcyclohexane-1,3-dione reacts with cyanoacetamide to give coumarin 45 b [72]. The latter was also obtained from 2-dimethylaminomethylene-5-phenylcyclohexane-1,3-dione and cyanoacetamide [73]. 2-Amino-3-cyano-4-(2-furyl)-benzopyran derivatives were obtained from diketones 1 and furfurylidenemalononitrile [74].

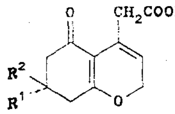
The reactions of diketones 1 with o-bromobenzoic acids in the presence of NaH and CuBr have been used for the synthesis of dibenzo[b, d]pyrans 46 [75, 76]. 2,5-Dibromoterephthalic acid has also been used in reactions of this type [75].

The reactions of diketones 1 with alkylthiodiphenylpropenium salts lead to 2-alkylthio-3,4-diphenyl-2H-pyrans 47 [77-79].

Dimethylselenonium dimedonylylid [80] reacts with diphenylcyclopropene to give pyrones 48 and 49 [81]. (Formula, top, following page.)

The reaction of diketones 1 with 2-acetyl-2-cyclohexenones in the presence of sodium methoxide gave tetraketones 50, which were then converted to dibenzopyran derivatives 51 [82]. Similar compounds 52 were obtained from α-chloromethoxynaphthalene and dimedone or 2-methyldimedone [83].

The simple synthesis of benzopyrandiones 54 on the basis of dimedone and the simplest anhydrides — acetic or propionic anhydride — in the presence of sodium hydroxide should particularly be noted; the reaction proceeds through intermediate O,C-diacyl derivatives 53 [84].



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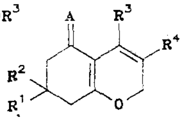

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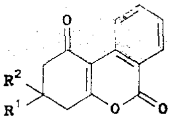

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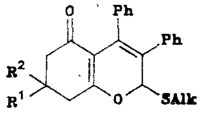

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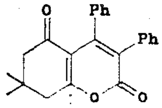

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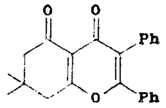

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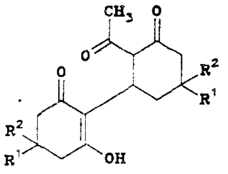
45 a A=C(CN)$_2$, R^3=H, CH$_3$, R^4=CN; b A=O, R^3=H, R^4=CONH$_2$

The reaction of 2-aminomethylenedimedone with N,N-dimethylacetamide diethylacetal gives aminal 55, which subsequently readily gives 7,7-dimethyl-5-oxo-5,6,7,8-tetrahydrocoumarin [85]. α-Chloro-β-dimethylamino-acrolein aminals and aminoacetals react with dimedone to give enamine 56, which, according to the results of physicochemical investigations, undergoes partial conversion to isomer 57 in solutions [86].

Chromanones 58 were obtained by acylation of diketones 1 with β,β-dimethylacrylic acid chloride in the presence of TiCl$_4$ [87], while 2,2-disubstituted α-chromenes 59 were obtained from diketones 1 and β,β-disubstituted acroleins [88].

Indolo[2,3-c]benzopyrylium perchlorates (60) were obtained in the reaction of 2-(3-indolyl)dimedone with a mixture of equimolar amounts of a carboxylic acid anhydride and perchloric acid [89].



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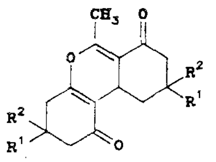

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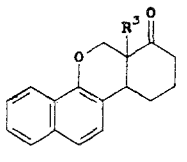

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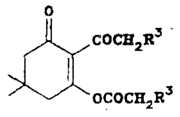

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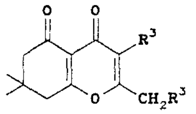

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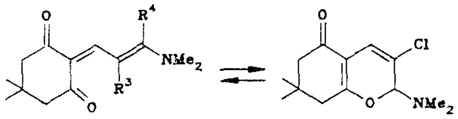

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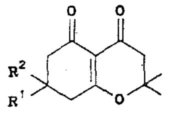

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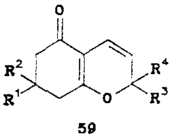

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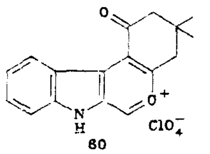

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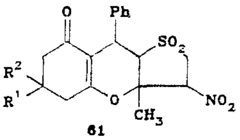

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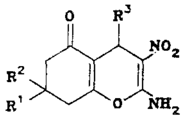


62

55 R^3=H, R^4=NMe$_2$; 56 R^3=Cl, R^4=H

The synthesis of heterocycles using unsaturated nitro compounds and cyclohexane-1,3-diones has been developed. Thus the reaction of 2-benzylidene-3-methyl-4-nitro-3-thiolene 1,1-dioxide with diketones 1 in the presence of an alkoxide gives three-ring chroman derivatives 61 [90]. Chromenes 62 were obtained from dimedone and cyanonitroethenes [91].

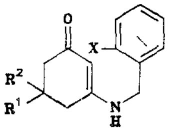
CONDENSED PYRIDINES

With respect to the number of studies devoted to it, the synthesis of condensed systems that include a pyridine ring on the basis of cyclohexane-1,3-diones occupies the number-one position. Some of these investigations are genetically related to studies dealing with six-membered O-containing heterocycles (for example, [81, 82, 85, 92]), while some pertain to the synthesis of dihydropyridines via various variations of the Hantzsch method using cyclohexane-1,3-diones and their enamines. The interest in 8-azasteroids, in the synthesis of which cyclohexane-1,3-diones are often used, is not diminishing. Investigations of the reactions of cyclohexane-1,3-diones and their derivatives with malononitrile have enriched this area of research substantially.

Under conditions that are similar to those used for the cyclization of N-aryleneamines 6 to carbazoles, N-benzyleneamines 63 are converted to phenanthridines 64 [21, 25]. The cyclization of o-bromobenzyleneamines was also carried out photochemically [93] and under the influence of $LiNEt_2$ [94, 95].

Polyphosphoric acid was used as the cyclizing agent in a known method for obtaining 7-oxo-7,8,9,10-tetrahydrophenanthridines from N-arylaminoalkylidenecyclohexane-1,3-diones [96, 97].

Research on condensation reactions with the participation of diketones 1, an aldehyde, and an enamine (or an enamine of cyclohexane-1,3-dione, an aldehyde, and a methylene ketone), which lead to the formation of hexahydroquinolines 65 [98-100], which have hypotensive properties [100], continued. Aldoses were used as the aldehydes in reactions with dimedone enamine [101].



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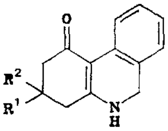

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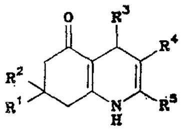


65

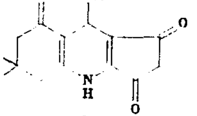
The reactions of enamines of cyclohexane-1,3-diones with α,β-unsaturated carbonyl compounds have been studied extensively. Thus condensed dihydropyridines 66 were obtained with 3-benzylidenecyclopentane-1,2,4-trione [100], while 5-oxo-5,6,7,8-tetrahydro- and 5-oxo-1,2,3,4,5,6,7,8-octahydro-2-methylquinolines were obtained with methyl vinyl ketone [102]. The same tetrahydroquinoline was obtained via a similar pathway [103, 104] from 2-butynone and an enamine, while 4-methyl-5-oxo-5,6,7,8-tetrahydroquinoline was synthesized from diketone 1 and 1-amino-2-butyne [104].

Benzo[*a*]phenanthridine derivatives 68 were obtained from 2-benzalaminonaphthalene and the corresponding 4,5-disubstituted cyclohexane-1,3-diones [105].

Cyclohexane-1,3-diones 1 and their enamines react with α-methyl-β-amino(and β-alkoxy)acroleins to give 3-methyl-5-oxo-5,6,7,8-tetrahydroquinolines [106]. Dimedone enamine reacts with ethyl acrylate in diglyme to give quinolone derivative 67 [107], which was also obtained from 2-(3-cyanoethyl)cyclohexane-1,3-dione [108]. 4-Azabenz[f]indan derivatives 69 were obtained in the reactions of 3-acylcyclopentane-1,2,4-triones with dimedone enamine [109, 110]. In the reaction of β-benzoylacrylonitrile with cyclohexane-1,3-dione enamine the product is 4-oxo-2-phenyl-4,5,6,7-tetrahydroquinoline, which is formed as a result of splitting out of HCN [111].

Various heterocyclic systems that include a pyridine ring were obtained in the reactions of diketones 1 with 4-chloro-5-formyl-6,7-dihydroindazoles [112], 4-(3-indolyl)pyrimidine [113], benzylidenecyanothioacetamide [114], and 3,1-benzoxazine-2,4-dione [115].

Secondary aromatic amines react with formaldehyde and diketones 1 in the presence of mineral acids and a mild dehydrogenating agent to give tetrahydroacridinium salts 70 [116] rather than phenanthridinium salts, as was originally assumed [117]. β-Naphthylamines have also been subjected to these reactions [118].



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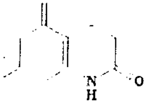

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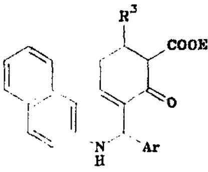

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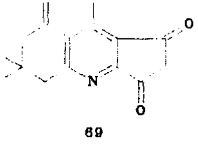

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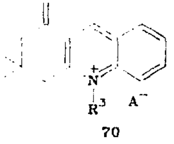
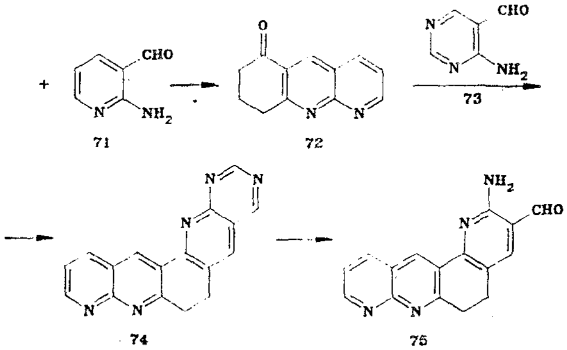
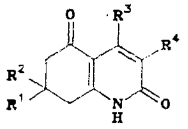


70

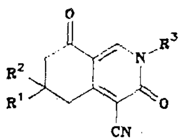
A method for annelation of the two-ring naphthyridine structural fragment using cyclohexane-1,3-diones has been developed. The reaction of diketones 1 with 2-amino-3-formylpyridine (71) gives 6-oxo-6,7,8,9-tetrahydrobenzo[b]-1,8-naphthyridine (72) [119], which then reacts with, for example, 4-amino-5-formylpyrimidine (73) to give 6,7-dihydropyrimido[4,5-b]-pyrido[2',3'-j]-1,7-phenanthroline (74) [120], which is readily hydrolyzed to amino aldehyde 75; the latter reacts with, for example, naphthyridine 72 or diketones 1 to give complex polycondensed systems.



The synthesis of quinoline and isoquinoline derivatives on the basis of the reactions of 2-acylcyclohexane-1,3-diones and their derivatives with malononitrile has undergone intensive development in the last decade [69]. Quinoline derivatives 76 were obtained from 2-acetylcyclohexane-1,3-diones and malononitrile [70, 121, 122]. The condensation of 2-arylaminomethylenecyclohexane-1,3-diones with malononitrile in refluxing ethanol in the presence of morpholine led only to isoquinoline 79, whereas a mixture of quinoline derivatives 77 and 80, as well as isoquinoline 79, is formed at 20°C [123]. These principles were later confirmed for a large number of 2-arylaminomethylenecyclohexane-1,3-diones [124], as well as in a study of the reaction of 2-dimethylaminomethylenecyclohexane-1,3-diones with malononitrile and cyanoacetamide [73]. The reaction of 2-formylcyclohexane-1,3-diones and their potassium salts with malononitrile also gives quinolinedione 77 as the chief product [71], while the reaction of formyl derivatives and their potassium salts [72] and 2-dimethylaminomethylenecyclohexane-1,3-diones [73] with cyanoacetamide leads to quinoline derivative 78.



76 – 78



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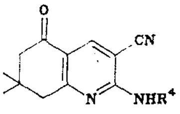


80

76 R^3=CH_3, R^4=CN; 77 R^3=H, R^4=CN; 78 R^3=H, R^4=$CONH_2$

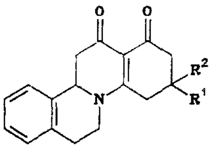
2-Acetylcyclohexane-1,3-diones and other acetyl-containing cyclic triacylmethanes are used in reactions with 3,4-dihydroisoquinoline N-oxide [125-127]. The addition products are converted to quinolizidine derivatives 81 when they are refluxed in DMF. Thus a simple route to analogs of 8-azasteroids was discovered (see earlier reviews [128, 129]).

Depending on the ratios of the reacting substances, piperidine-containing bridged systems 82 and 83 were obtained from diketone 1c, benzylamine, and formaldehyde under the conditions of the Mannich reaction [130]. Ethylenediamine has also been used in place of benzylamine in these reactions. Data on the participation of cyclohexane-1,3-diones and their enamines in Mannich reactions have also been presented in a review [131] (see also [132]).

6-Substituted 7-oxo-7,8,9,10-tetrahydrophenanthridines were synthesized from 3-(2-acylaminophenylthio)-2-cyclohexenone [133] under conditions of cyclization by means of polyphosphoric acid.

Treatment of triketone 84 with hydroxylamine leads to the formation of a mixture of quinoline derivatives 85 and 86 [134].

Pyridine-containing systems were also obtained in the course of allylation of enamines of cyclohexane-1,3-diones [135].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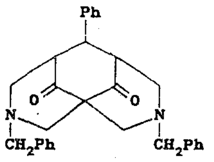
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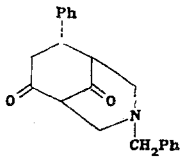
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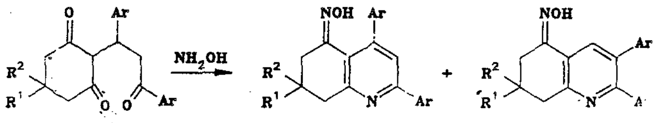
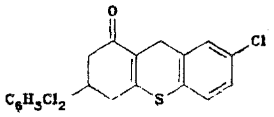


84 85 86

CONDENSED THIOPYRANS

The reaction of 3-bromo-5-(2,4-dichlorophenyl)-2-cyclohexenone, which was obtained from the corresponding cyclohexane-1,3-dione and PBr$_3$, with 2-mercapto-5-chlorobenzoic acid in DMF in the presence of sodium hydride leads to the corresponding sulfide, which, under the influence of polyphosphoric acid, gives thioxanthene 87 — a substance with antimalarial activity [136].

Thiobenzopyrans 88 were obtained by the action of hydrogen sulfide on triketones 84 [137, 138].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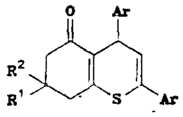
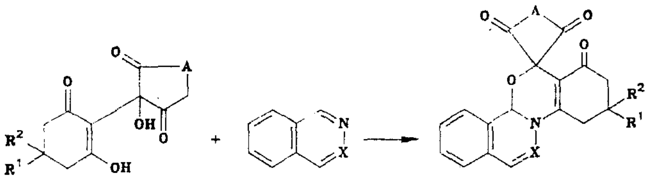

88

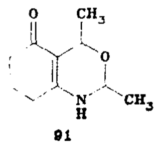
1,3-OXAZINE DERIVATIVES

A general method for the synthesis of complex condensed compounds 90, which include a 1,3-oxazine ring with a β-dicarbonyl function spiro-connected in the 6 position, has been developed [139-142]. The essence of the method consists in the reaction of tosylates of hydroxytetraketones 89, which were obtained as a result of the addition of diketones 1 to 1,2,3,-triketones, with isoquinoline and phthalazine.

A simple method for the synthesis of benzoxazines 91 from dimedone enamine and acetaldehyde has been proposed [143].

Diazabutadienes of the 92 type react with diketone 1a to give benzoxazine derivatives 93 and quinazoline derivatives 94 [144].


89 90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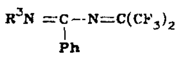
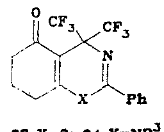
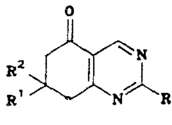

$R^3N{=}\underset{\mid \atop Ph}{C}{-}N{=}C(CF_3)_2$

92


93 X=O; 94 X=NR^3

SIX-MEMBERED HETEROCYCLES WITH TWO NITROGEN ATOMS

A large number of quinazolines 95 have been obtained via a known scheme from 2-aminomethylenecyclohexane-1,3-diones and amidines [145]. Under the influence of butoxide, dimedone o-nitroanil undergoes cyclization to N-oxide 96, which is converted to quinoxaline 97 as a result of dehydration and reduction [146]. The reaction of phenylhydrazonocyclohexane-1,3-dione with malononitrile led to cinnoline derivative 98 [147].



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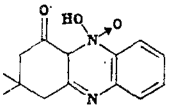

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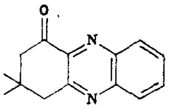

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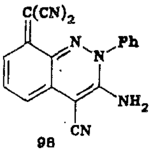


98

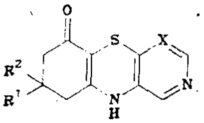
PHENOTHIAZINES AND OTHER SIX-MEMBERED SULFUR-CONTAINING HETEROCYCLES

5-Amino-4-mercaptopyrimidines react with 2-bromocyclohexane-1,3-diones to give pyrimidothiazine derivatives 99 [149, 149]. Phenothiazine derivatives 100 were obtained from 2-mercaptoaniline and diketones 1 [133, 150].

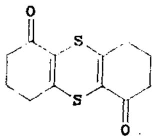
It has been shown [151] that cyclohexane-1,3-diones 1 react with sulfur in the presence of secondary amines to give the corresponding salts. Dithiin derivative 101 is formed in the acidification of salts obtained from diketone 1a, while treatment with acetic anhydride leads to benzoxathiins 102.

Phenoxatellurane derivative 103b was synthesized from dimedone enol acetate and $TeCl_4$ with subsequent reduction with sodium sulfite [152].

Condensed systems that include a triazine ring were obtained from diketones 1, 1,2,4-triazole-3-diazonium sulfate [153], and 3-diazopyrazolo[3,4-b]-pyrazine [154].



99 X=N; 100 X=CH



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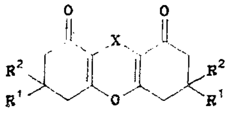


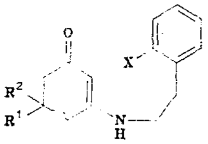
102 X=S; 103 X=Te

SEVEN-MEMBERED NITROGEN-CONTAINING RINGS

Cyclohexane-1,3-dione enamines of the 104 type (X = H, Hal) can be cyclized to the corresponding azepines 105 using the electrochemical [24], photochemical [93], and chemical [94] methods that were examined above in the synthesis of five- and six-membered nitrogen-containing rings. Azepine 106 was obtained by photochemical cyclization of the corresponding enamine containing a γ,δ-double bond [155].

A large number of dibenzodiazepines 107 have been synthesized from dimedone N-(o-aminophenyl)enamines and acyl chlorides [156].

Azepines 108 were obtained by Beckmann rearrangement of 1,2-diaryl-4-oxo-4,5,6,7-tetrahydroindole oximes [157], while the thermolysis of 3-azido-2-cyclohexenone in methanol gives azepine 109 [33].



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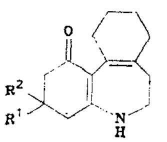

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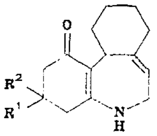

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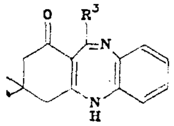

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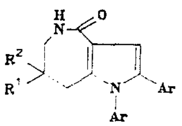

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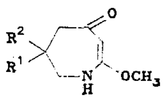


109

SPIROCYCLES

New methods for the synthesis of spirans — griseofulvin analogs 110 — have not been described during the period under review; however, a general photochemical method for the synthesis of 1,1,2,2-tetraacylcyclopropanes on the basis of them has been developed [158, 159].

It has been found [160] that the reaction of diketones 1 with selenium dioxide leads not to phenooxaselenin, but rather to spirobenz-1,3-oxaselenolcyclohexane derivative 111 (see also [161-163]).

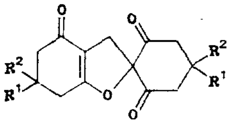
N,N'-Disubstituted sulfur diimides [164] and sulfoxylic acid diamides [165] are used as sulfur carriers in obtaining spirobenz-1,3-oxathiolanecyclohexane 112 from diketones 1. Compound 112 was also obtained by the successive action of bromine and triethylamine on didimedonyl disulfide [164]. Oxathiin 102 was prepared from spiro compound 112 by the action of triphenylphosphine, as well as by the action of acetic anhydride on didimedonyl sulfide [164].

Spirodithiolane 114 was synthesized photochemically from disulfide 113 [166]. Dithiin 115 was obtained from cyclohexane-1,3-dione and 1,3-ditosylmercaptopropane [167], while spiro compound 116 was obtained from dimedone and acyl isocyanates [168].

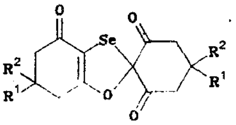
The sodium derivatives of cyclohexane-1,3-diones react with 4-chloromethyldihydropyridines to give spiro compounds 117 [169].

Didimedonylmethane enamines react with aldehydes to give spiro compounds 118 [170].

An unusual spiro compound — the 3-cyano-5,5-dimethyl-2-cyclohexenethione trimer — was obtained in the course of a systematic study [171] of thio analogs of cyclohexane-1,3-diones [172].



110



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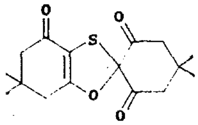

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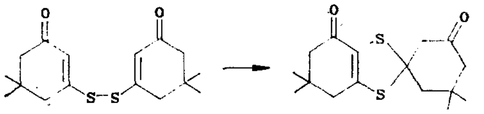

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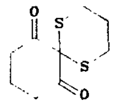

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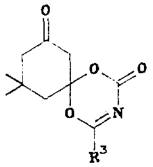
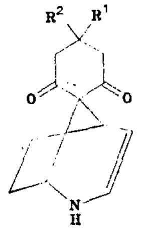


116



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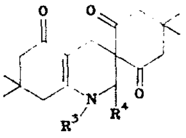
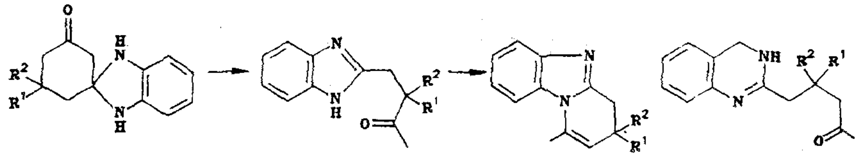


118

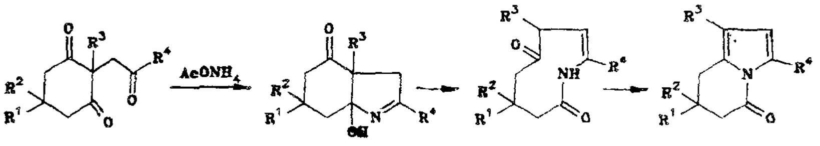
FORMATION OF HETEROCYCLES INVOLVING A CHANGE IN THE CARBON SKELETON OF CYCLOHEXANE-1,3-DIONES

Treatment of enamines 6 (X = NH_2) with toluenesulfonic acid in DMSO gives pyridobenzimidazoles 121 [173, 174]. The formation of the latter occurs through spiro compounds 119 with subsequent cleavage of the carbocycle and cyclization of benzimidazole derivative 120 to pyridobenzimidazole 121. Similar transformations of amide 6 (X = $CONH_2$) lead to quinazoline 122 [173]. The formation of benzothiazole derivatives via a similar scheme was noted in [133].

Treatment of triketone 123 with ammonium acetate in acetic acid in order to obtain the corresponding indole led to acylpyrrole 126 [175].



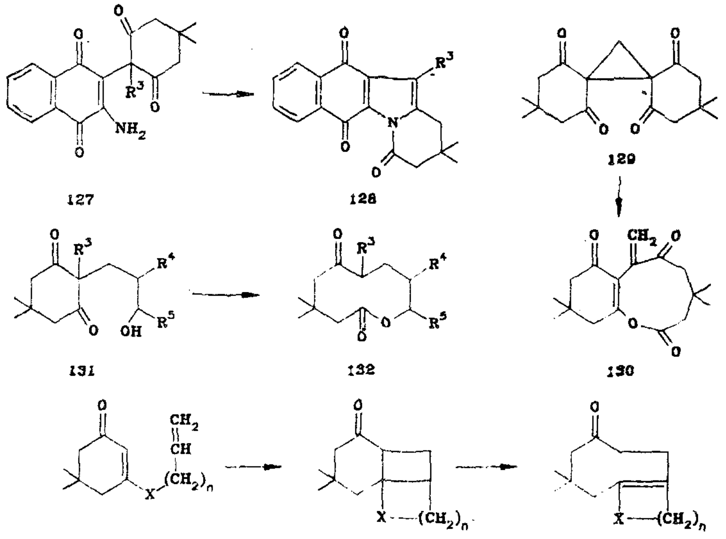
119 120 121 122



123 124 125 126

Similar transformations — opening of the ring of the addition product and subsequent cyclization with the participation of the other electrophilic center — were observed when aminoquinone 127 was treated with sodium hydride in tetrahydrofuran; pyrrole derivative 128 was formed as a result [176].

Spiro compound 129 undergoes isomerization to lactone 130 in the presence of tertiary amines [177].



127 128 129 131 132 130 133 134 135

An intramolecular reversible Dieckmann reaction leads to opening of the ring of 2,2-disubstituted cyclohexane-1,3-diones 131 under the influence of catalytic amounts of sodium hydride in benzene with the formation of lactones 132 [178, 179].

Enamines and enol ethers of the 133 type undergo photochemical transformations to give cycloaddition products 134 [180, 181], which are converted to bicyclic compounds 135 under the influence of boron trifluoride etherate.

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