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6-ARYL-2,2-DIMETHYL-1,3-DIOXIN-4-ONES: SYNTHESIS AND CHEMICAL CHARACTERISTICS. (REVIEW)

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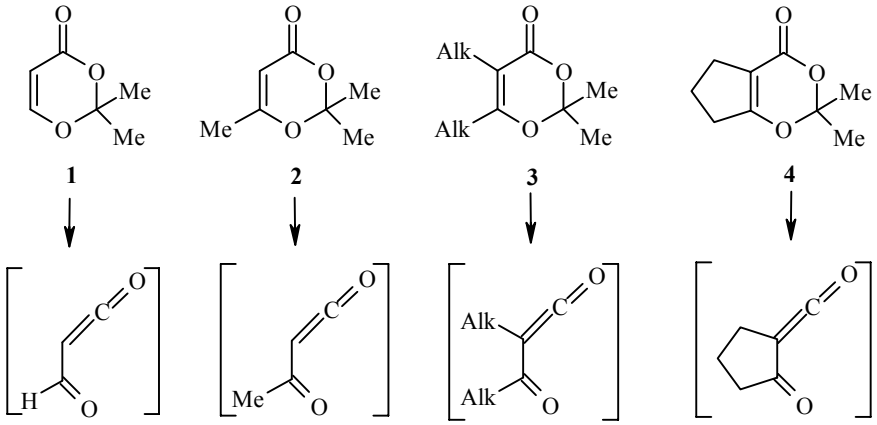
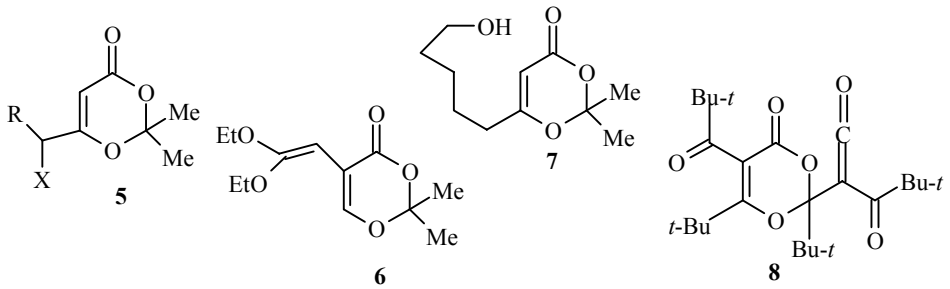
Methods for the production of 6-aryl-2,2-dimethyl-1,3-dioxin-4-ones and their thermolysis in the presence of nucleophiles and dienophiles are discussed.

Keywords: 6-aryl-2,2-dimethyl-1,3-dioxin-4-ones, aroylketenes, aroylacylation, biological activity, [4+2] cycloaddition, heterocyclization, thermolysis.

Acylketenes are often used as "building blocks" in chemical design [1, 2]. 1,3-Dioxin-4-ones **1-4** can be used as sources of these highly reactive particles [3-5].



The more "exotic" dioxinones **5-8** have not found widespread use in this connection [2].



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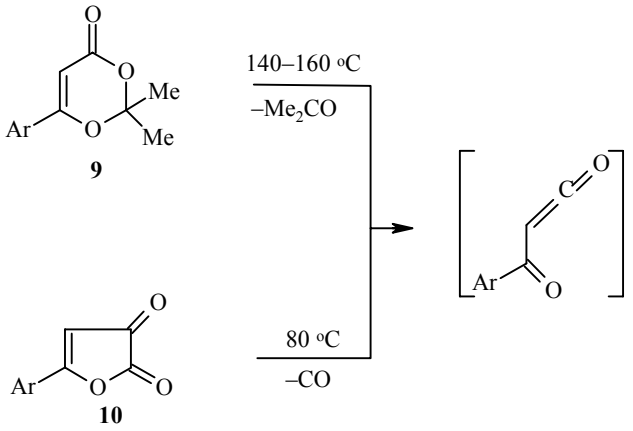
The presented types of dioxinones **1-8** do not cover the whole range and only give a general idea of the structure of such compounds. In the reviews devoted to 1,3-dioxin-4-ones [3-5] there are no data on the chemical transformations of their 6-acyl derivatives. Aroylketenes generated from 6-aryl-1,3-dioxin-4-ones **9** often enter into reactions analogous to the transformations of the aroylketenes formed from 5-aryl-2,3-dihydrofuran-2,3-diones **10** [6].



Ar = Ph, *p*-C_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

However, there are differences in the reactivity of these ketenes due to the different conditions of their formation (temperature difference 60-80°C).

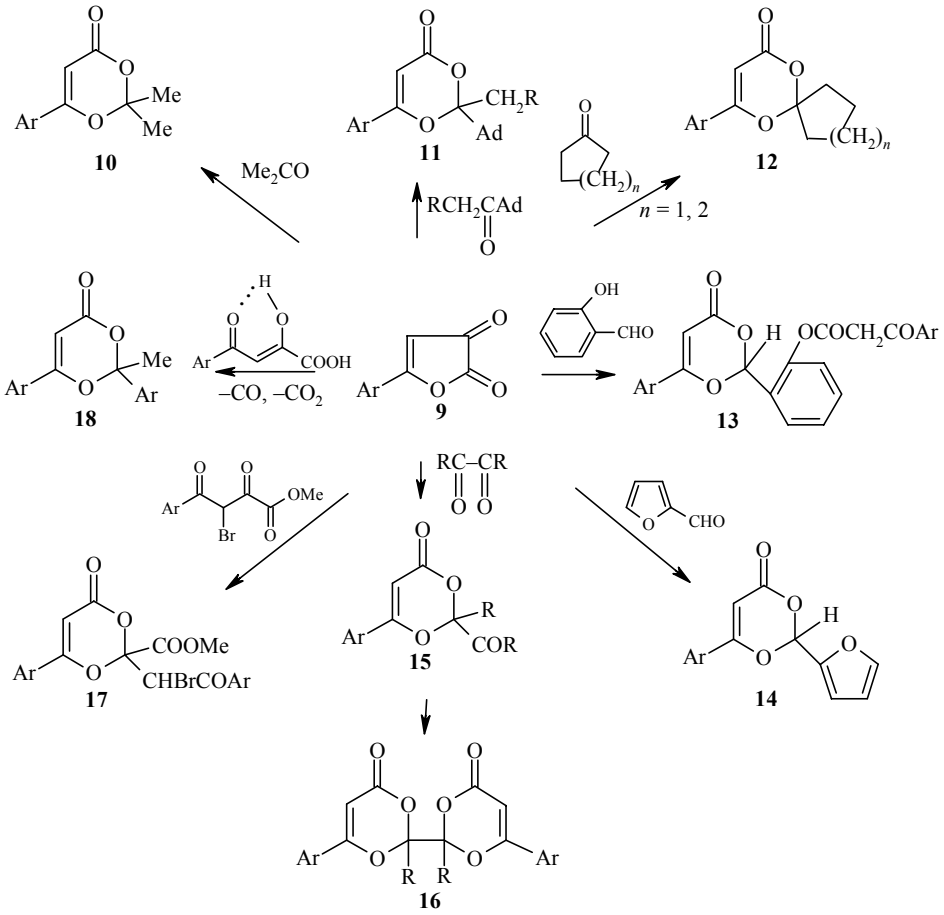
The material presented here supplements the above-mentioned reviews with new data not only on the dioxinones but also on the acylketenes.

1. Synthesis of 2-Substituted 6-Aryl-1,3-dioxin-4-ones

The largest number of 2-substituted 6-aryl-1,3-dioxin-4-ones **9**, **11-18** were obtained on the basis of the reactions of 5-aryl-2,3-dihydrofuran-2,3-diones with carbonyl compounds (Scheme 1). Ketones [7-12], aldehydes [9, 13], diketo esters [14], and enolized [15, 16] and nonenolized [9] dioxo compounds were used in the reaction.

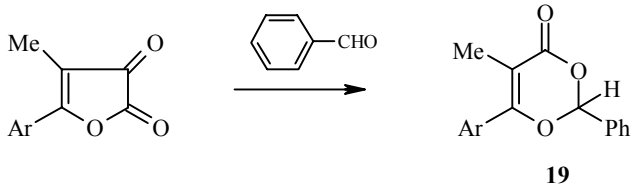
The highest yields of the aryldioxins presented in scheme 1 were obtained from aromatic aldehydes and aliphatic or alicyclic ketones (90-99%) [9]. The decrease in the yield of the dioxinones **11** (29-78%) synthesized from adamantyl methyl ketones is due, on the one hand, to steric hindrances created by the adamantyl substituent and, on the other, to the lower polarization of the carbonyl group in the reagent. The presence of an electron-withdrawing substituent in the methyl fragment (R = Cl, Br) increases the yield of the products **11** by 10-15% [10]. The yields of the dioxinones **17** and **18**, obtained from aroylpyruvic acids and their methyl esters, do not exceed 40-56% [14, 16]. Diacetyl reacts with furandione with the participation of both carbonyl groups irrespective of the ratio of the reagents. In the case of benzil both possible products **15** and **16** are formed [9].

Scheme 1



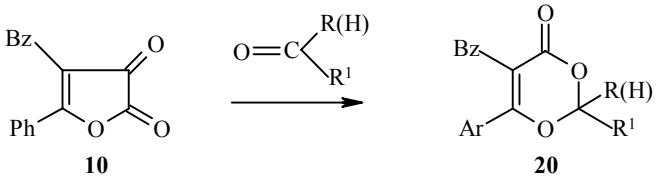
Ar = Ph, *p*-C_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

By using 5-aryl-4-methyl-2,3-dihydrofuran-2,3-diones it is possible to obtain the dioxinones **19** with yields of 64-99% [9]:



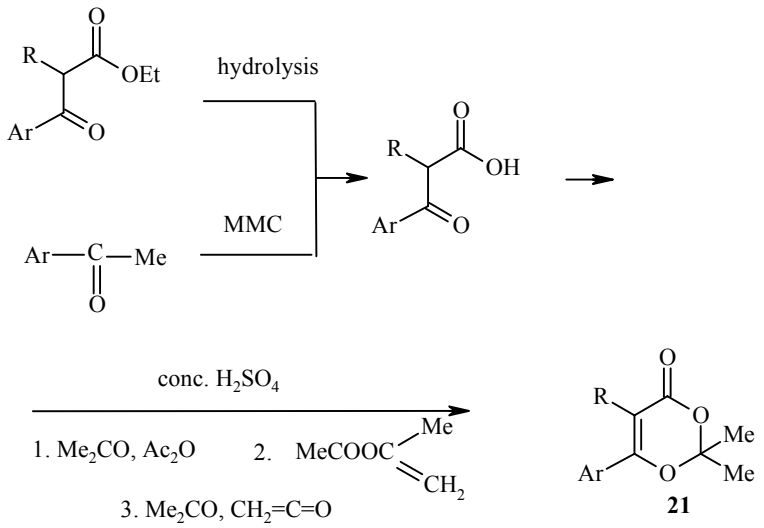
Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$

In the presence of a benzoyl substituent at position 4 of the furan ring in compounds **10** the yields of the dioxinones **20** amount to 55-89% [17].



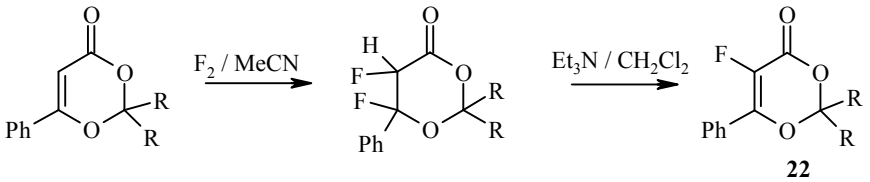
R = Me, Ph; R^1 = CCl_3, Ph, *p*-MeC_6H_4, *p*-ClC_6H_4, *m*-$NO_2C_6H_4$, C_5H_4N; R+R^1 = $(CH_2)_5$

A convenient method for the synthesis of aryldioxinones **21** was described in [18]. The authors proposed three versions of the method. Irrespective of the method, the yields of the products **21** amounted to 53-82%. The first method was based on the reaction of ethyl aroylacetates with acetone in the presence of acetic anhydride. The second method involved the reaction of the above-mentioned esters with isopropenyl acetate. In the third version these esters reacted with a mixture of acetone and ketene. In all three cases the role of catalyst was played by concentrated sulfuric acid. The dioxinones **21** are also formed during treatment of acetophenones with methylmagnesium carbonate (MMC). The method is simplified by the use of *tert*-butyl aroylacetates on account of the elimination of the hydrolysis stage.



R = H, Me; Ar = Ph, *p*-$MeOC_6H_4$, *p*-$NO_2C_6H_4$

Fluorination of the 2-substituted 6-phenyl-1,3-dioxin-4-ones gave the 5-fluoro derivatives **22** [19]; the yield of the 2,2-dimethyl-substituted derivative amounted to 30%, while that of the 2,2-spirocyclohexyl-substituted compound was 61%.

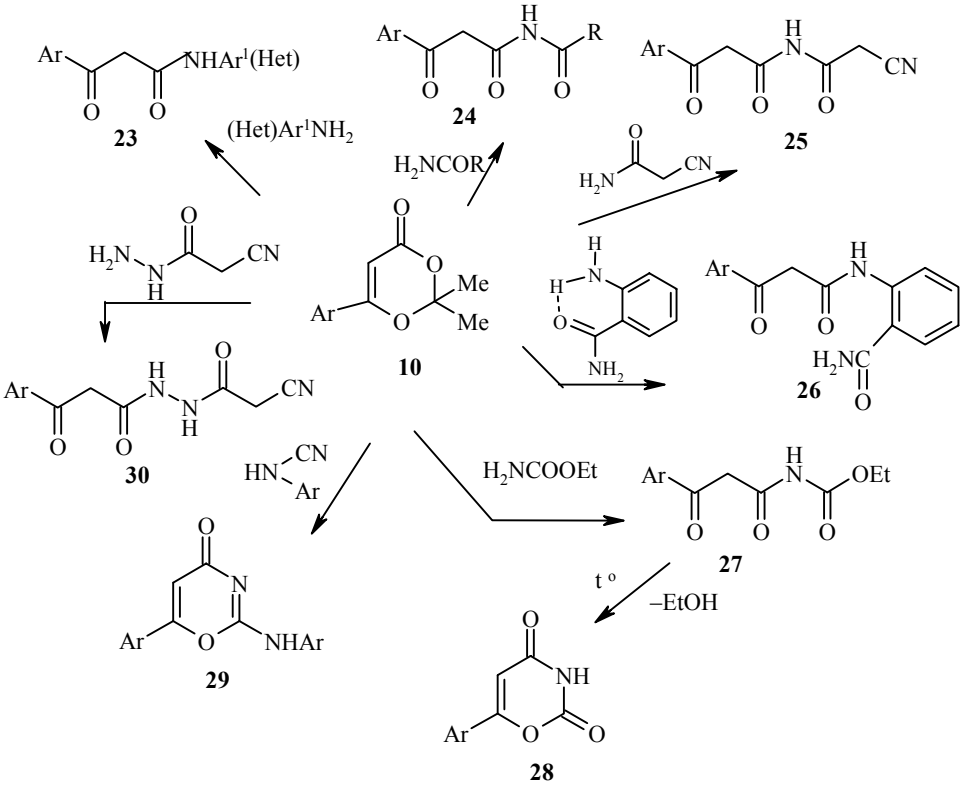


R = Me, R + R = $(CH_2)_5$

2. Reactions of Aryldioxinones with Amines and Amides

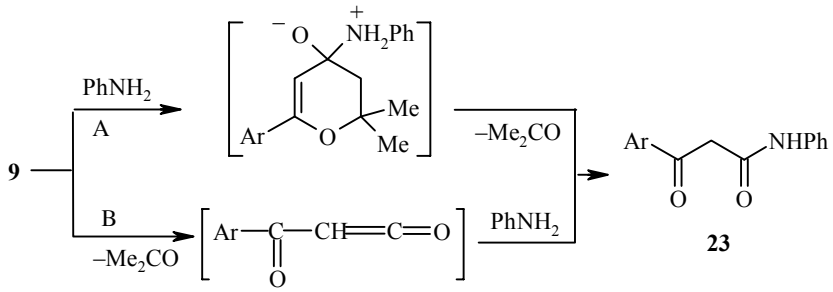
Aroylacetamides **23** were obtained with high yields by melting the aryldioxinones **9** with aromatic and heterocyclic amines [20, 21]. Aniline, *p*-toluidine, *p*-anisidine, 2-pyridylamine, and 2-pyrimidylamine were used.

Scheme 2



Ar = C_6H_5, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

Compounds **23** can form by two mechanisms. The acylation mechanism (path A) applies to nucleophilic substitution reactions at the carbonyl carbon atom. Path B involves the intermediate formation of the aroylketene.



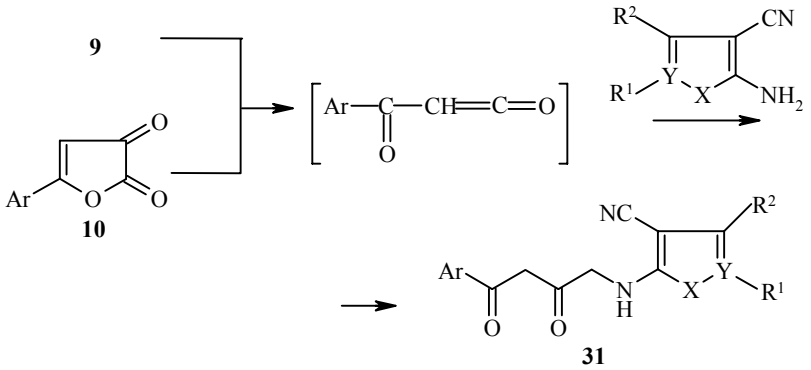
Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-$EtOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

It was established by kinetic investigations that path B, in which the controlling stage is the formation of the aroylketene by a [4+2] retrocycloaddition mechanism, is preferred [22].

Acylation of the amide groups in formamide and acetamide probably takes place according to a similar scheme, and the reaction products are compounds **24**, **25**. The yields of these compounds amount to 49-93% [23, 24]. The presence of the electron-withdrawing cyanoacetyl group in the cyanoacetamide does not affect the course of the reaction. At the same time the convenient leaving group EtO of the acyl carbamate **27** promotes their cyclization to the 1,3-oxazine-2,4-diones **28** [25]. In anthranilamide the amino group is acylated more

readily, but its involvement in an intramolecular hydrogen bond in the initial reagent significantly reduces the yields of the final products **26** [24]. In arylcyanamides the nucleophilicity of the amino group is probably reduced to an even greater degree than in the amides, as a result of which the aroylketene reacts not at the amino group but at the cyano group. The reaction products in this case are 1,3-oxazin-4-ones **29** [26]. In cyanoacetylhydrazide aroylacylation takes place at the amino group. An attempt to bring the NH and NHCO groups into the reaction simultaneously was unsuccessful. The absence of an intramolecular hydrogen bond in the reagent makes it possible to obtain the final products **30** with high yields [27].

Heterylaminonitriles containing pyrrole, furan, and pyrazole rings react with the dioxinones **9** and furandiones **10** like heterylamines [28] with the formation of N-aroylacetyl derivatives **31** [29]. 2-Amino-1,5-diphenylpyrrole-3,4-dicarbonitrile, 2-amino-4,5-diphenylfuran-3-carbonitrile, and 5-amino-1-phenylpyrazole-4-carbonitrile were also brought into the reaction. The yields of the products **31** amounted to 64-80%.

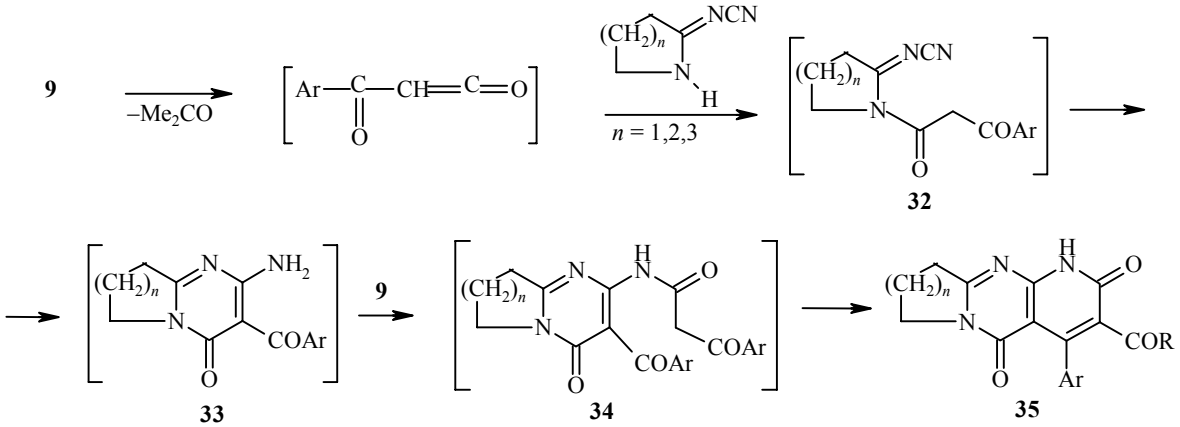


31

Ar = Ph, *p*-MeC_6H_4, *p*-$EtOC_6H_4$, *p*-ClC_6H_4

The reaction is one of the few examples where the dioxinones **9** and furandiones **10** react with amino compounds according to one scheme. The exception is 2-amino-4,5-tetramethylenethiophene-3-carbonitrile, which with furandiones forms not the N-aroylacetyl but the N-aroylpyruvyl derivatives. This is probably due to the more nucleophilic characteristics of the amino group in this reagent [29].The cyano group of heterylaminonitriles, unlike the cyano group of arylcyanamides, does not take part in the reaction.

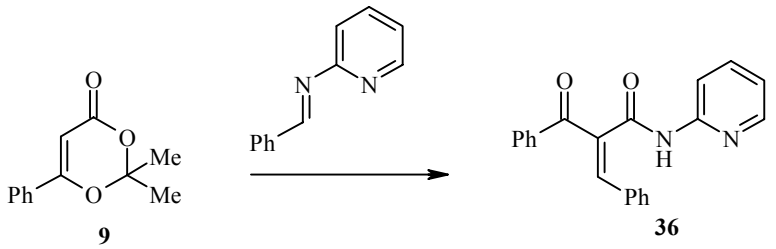
The reaction of the dioxinones **9** with 2-(N-cyanoimino)pyrrolidine is a multistage process. At the first stage 1-N-aroylacylation of the reagent occurs with the formation of the derivatives **32**. Condensation of the cyano group in them with the active methylene group leads to the aminopyridines **33**. During further aroylacylation of compounds **33** their N-aroylacetyl derivatives **34** are formed. Intramolecular cyclization of the latter leads to the polymethylenepyrido[2,3-*d*]pyrimidines **35** [30], the yields of which amount to 48-56%.



Ar = Ph, *p*-MeC_6H_4, *p*-ClC_6H_4

Unlike the heterylamino nitriles, N-cyaniminopyrrolidine does not enter into reaction with furandiones **10**.

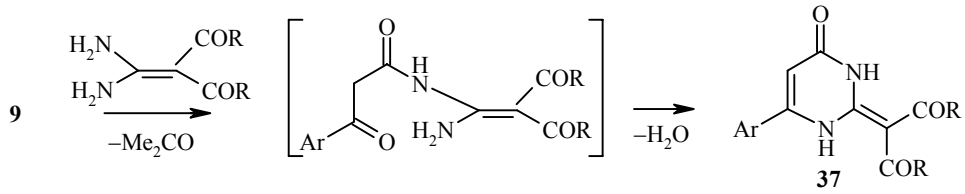
With the dioxinone **9** 2-(benzylideneamino)pyridine forms 2-benzoyl-N-(2-pyridyl)cinnamamide **36** with a yield of 69% [31].



9 **36**

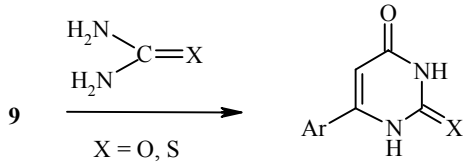
3. Reactions of Aryldioxinones with 1,1- and 1,2-Diamines

During the reaction of aryldioxinones **9** with ketene aminals (2-diaminomethylene-1,3-diphenyl-1,3-propanedione or ethyl 2-diaminomethylene-3-oxobutyrate), which can be regarded formally as 1,1-diamines, the substituted 6-aryl-2-methylene-4H-pyrimidin-4-ones **37** are formed with yields of 64-93% [32, 33].



Ar = Ph, *p*-MeC_6H_4, *p*-ClC_6H_4

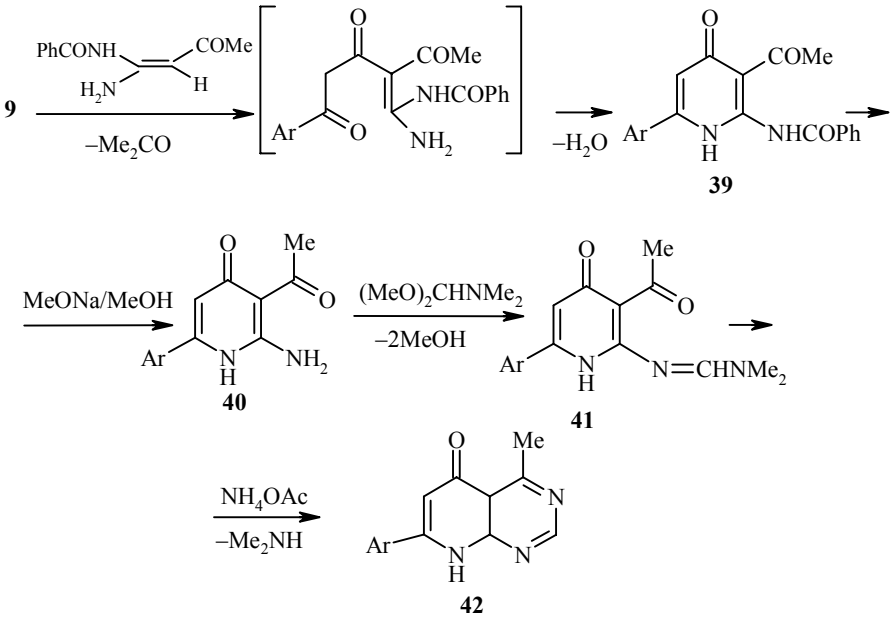
Urea and thiourea react according to a similar scheme. The reaction products are the corresponding uracils and thiouracils **38** [34]. The yields of these compounds are between 42 and 80%.



38

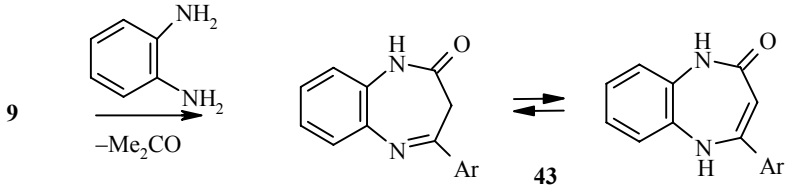
Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

The ketene aminal 1-amino-1-benzoylamino-1-buten-3-one reacts with the aryldioxinones **9** not as a N,N-nucleophile but as a C,N-nucleophile, leading to the formation of the pyridones **39**, which are readily debenzoylated to compounds **40**. The latter are converted by treatment with dimethylformamide dimethyl acetal into compounds **41**, which undergo cyclization under the influence of ammonium acetate in butanol with the formation of pyrido[2,3-*d*]pyrimidin-5-ones **42** [33]. The reaction is a multistage process, and the yields of the final products do not therefore exceed 60%.



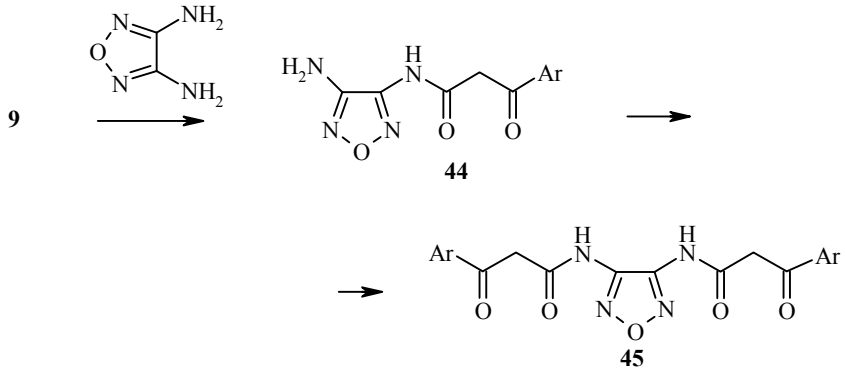
Ar = Ph, *p*-MeC_6H_4, *p*-ClC_6H_4

The reaction of the aryldioxinones **9** with *o*-phenylenediamine leads to 1,5-benzodiazepin-2-ones **43** [22], the yields of which amount to 60-84%.



Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

In contrast to *o*-phenylenediamine, diaminofurazan is aroylacylated by the dioxinones **9** at the amino groups, and both the monoaroylacetyl **44** and the bisaroylacetyl **45** derivatives are formed, depending on the ratio of the reagents [35]. The yields amount to 27-60% in the first case and 49-78% in the second.



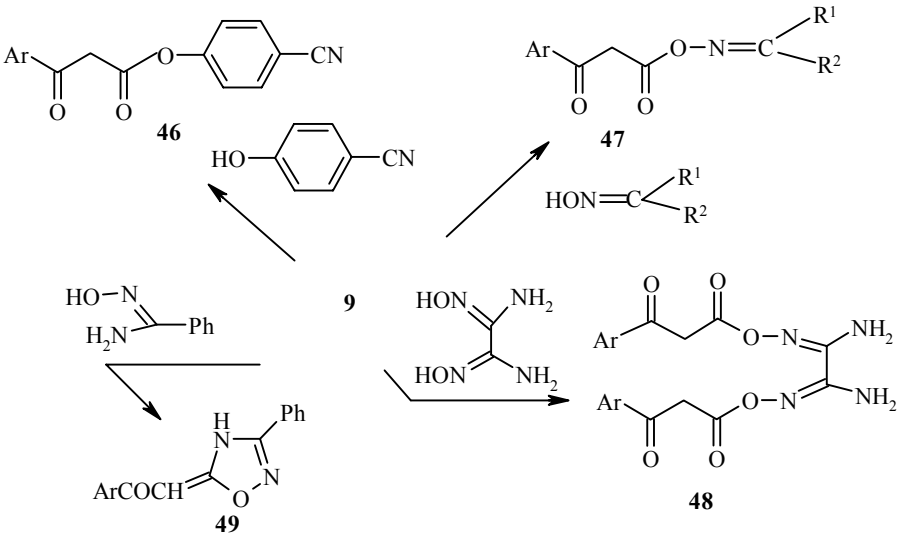
Ar = Ph, *p*-MeC_6H_4, *p*-ClC_6H_4, *p*-BrC_6H_4

4. Reactions of Aryldioxinones with OH-Nucleophiles

During the reaction of the aryldioxinones **9** with *p*-hydroxybenzonitrile *p*-cyanophenyl aroylacetates **46** are formed with yields of 55-67% [36].

The O-aroylacetyl derivatives **47** were also obtained from oximes [37]. The reaction was carried out with the oximes of acetophenone and salicylaldehyde. The yields of the products **47** were 65-75%. Diaminoglyoxime takes part in the reaction not as an NH-nucleophile but as an OH-nucleophile, and the bis-O,O-aroylacetyl derivatives of diaminoglyoxime **48** are formed irrespective of the ratios of the reagents.

Scheme 3

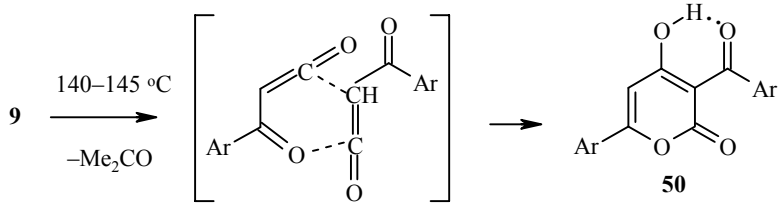


Ar = Ph, *p*-MeC_6H_4, *p*-ClC_6H_4, *p*-BrC_6H_4

The previously described structure of these compounds as N,N-aroylacetyl derivatives [35] was rejected in [38]. Benzamidoxime takes part in the reaction as an O,N-binucleophile, and the products are 4,5-dihydro-1,2,4-oxadiazoles **49** [35]. The yields of the latter amount to 82-95%.

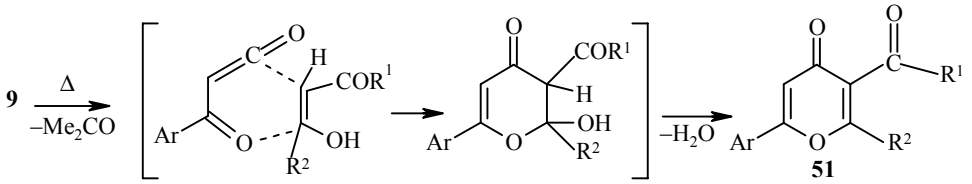
5. [4+2] Cycloaddition Reactions between Two Molecules of Aroylketene

If the reaction mixture contains an inert reagent, the aroylketenes formed during the thermolysis of the dioxinones **9** enter into [4+2] cycloaddition, and one molecule of the aroylketene acts as heterodiene while the other acts as heterodienophile. The products of such dimerization, i.e., 4-hydroxy-2H-pyran-2-ones **50**, are stabilized by an intramolecular hydrogen bond [9, 39].



Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

In the reaction of the dioxinones **9** with acetylacetone and methyl acetyl- and aroylpyruvates the role of 2π-component is played by the ethylene bond on the enol fragment of the molecules [40, 41].



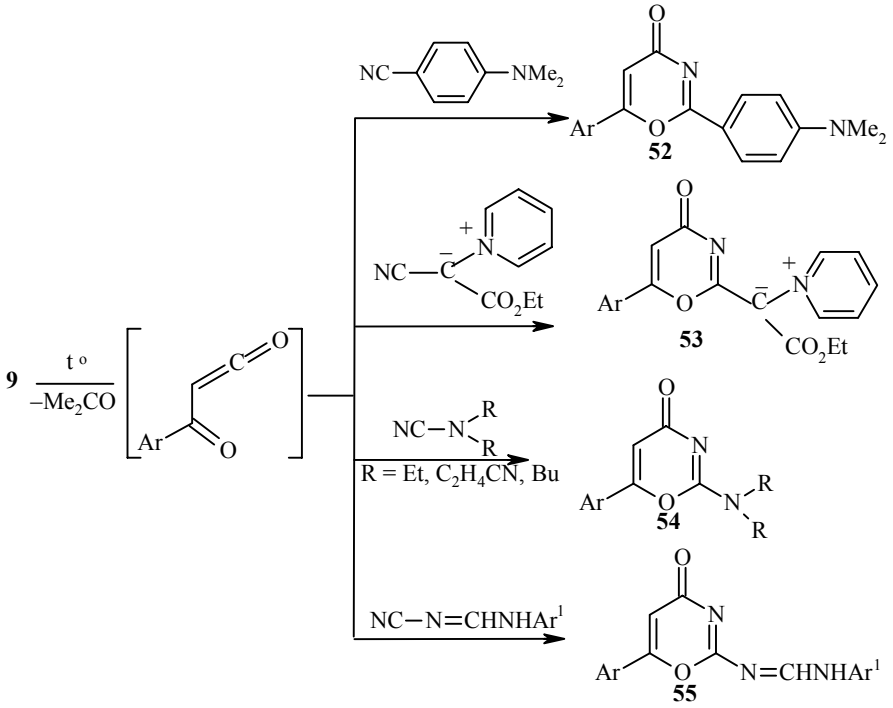
Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4

The initially formed 3-acyl-1,3-dihydro-4-pyranones undergo dehydration under the reaction conditions with the formation of the pyranones **51**. The yields of the latter are low (6-41%), and this is due to the competing dimerization of the aroylketenes to the pyranones **50**.

Reagents containing an acetylene bond do not enter into [4+2] cycloaddition with the aroylketenes generated from the dioxinones **9** [9].

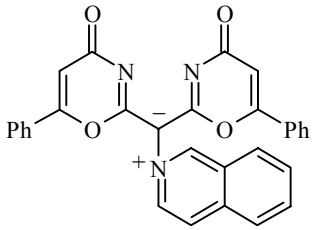
In order to bring the carbon–nitrogen triple bond into the heterodiene synthesis it is necessary to have strong electron-donating substituents, which promote polarization of this bond. Examples of such reagents are shown in scheme 4. In all cases the reaction products are 2-substituted 6-aryl-1,3-oxazin-4-ones **52-55** [36, 39, 42]. In the review [43] it was shown for the case of cyanamides that the reaction of aroylketenes with these reagents represents a Diels–Alder reaction with an inverse electronic effect in the addends. In spite of the presence of three reaction centers in the molecule of the N-tolyl-N'-cyanoformamidines (the double and triple carbon–nitrogen bonds and also the NH group) the process takes place regioselectively at the C≡N bond with the formation of the oxazinones **55**, which is probably due to the positive mesomeric effect of the amino group [44, 45]. The reaction makes it possible to obtain the oxazinones **55** with yields of 55-85%.

Scheme 4



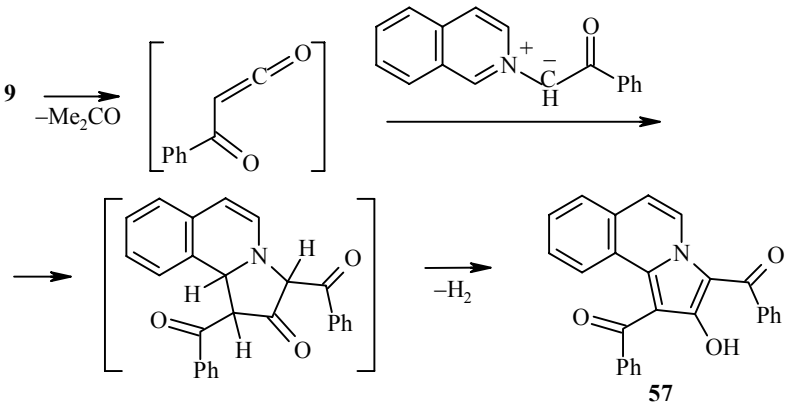
Ar = Ph, *p*-MeC_6H_4, *p*-$MeOC_6H_4$, *p*-ClC_6H_4, *p*-BrC_6H_4, Ar' = Ph, *p*-$MeOC_6H_4$

The presence of a second cyano group in the isoquinolinium cyanomethylide leads to the formation of the bisoxazinone **56** with a yield of 74% [39].



56

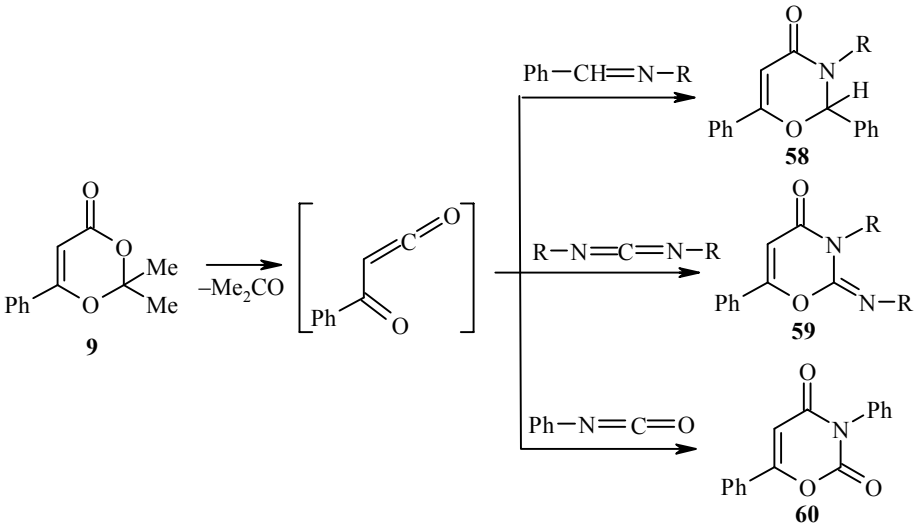
6-Phenyl-1,3-dioxin-4-one reacts abnormally with isoquinolinium phenacylylide. The reaction follows the pattern of 1,3-dipolar cycloaddition. The final product is the pyrroloisoquinoline **57**, the yield of which amounts to 25% [39].



57

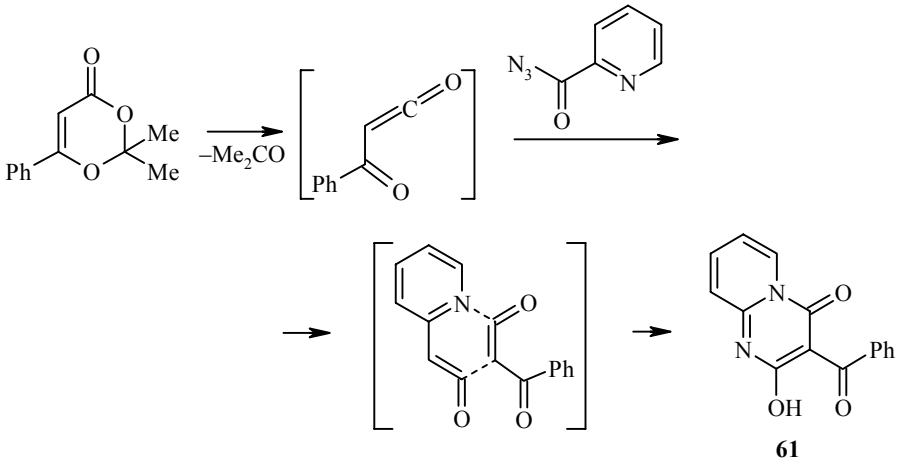
In contrast to the C≡N bond, the C=N double bond in heterodiene synthesis requires activation by electron-donating substituents. Schiff bases (R = Et, *t*-Bu, Ph), carbodiimides (R = Ph, cyclohexyl), and phenyl isocyanate were used in the [4+2] cycloaddition reaction with the aroylketenes generated from compounds **9** (Scheme 5).

Scheme 5



The main products of these reactions are 3,4-dihydro-1,3-oxazin-4-ones **58**-**60** [31], the yields of which lie in the range of 43-84%. In a number of cases with Schiff bases the substituted acetylacetamides are also formed together with the oxazines **58**, and the yields of compounds **58** are then 17-20% lower [39].

The isocyanate from picolinoyl azide reacts with benzoylketene with the formation of pyrido[1,2-*d*]-pyrimidin-4-one **61**.



61

In the reaction in question, as also in the dimerization of aroylketenes, the benzoylketene acts as dienophile [31].

2-2-Disubstituted aryldioxinones have antimicrobial activity against *Escherichia coli* and *Staphylococcus aureus* [12, 14, 15]. In addition, they are used in the synthesis of biologically active compounds possessing anti-inflammatory and analgesic activity [29, 37, 38, 46].

Thus, the presented published data on the synthesis and characteristics of 2,2-disubstituted 6-aryl-1,3-dioxin-4-ones indicate that these compounds may become widely used synthons for the production of substituted aroylacetamides and nitrogen-containing heterocycles.

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