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Synthesis of Thienyl-Containing Photochromes (Dithienylethenes, Fulgides, Fulgimides, and Spirocompounds)

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INTRODUCTION

In the last 40 years, photochromic compounds have attracted considerable attention both from a theoretical standpoint and in view of their various practical applications.

Nowadays, thermally reversible photochromes are widely employed in actinometry, reprography, sun protective glasses, and tools for protection against light radiation [1–3], whereas thermally irreversible products are actively investigated for optical memory devices [4,5]. There are also numerous publications certifying that very diverse dihetarylethenes possess high-biological activities [6,7].

The expediency of further modification of the most prospective photochromes, with the aim to extend operating characteristics of the products, is obvious. The introduction of an electron excessive thiophene ring into the molecules of spirocompounds, fulgides, and fulgimides and modification of dihetarylethenes are one of the most obvious approaches to solve this problem. The present paper describes only the synthetic part of our research, taking out of context a section associated with spectral properties of photochromic products, which is quite attractive but requires extensive comments.

CONDENSED THIOPHENES AS THE PRECURSORS OF PHOTOCHROMES

Our data and those reported in the literature [8,9] show that photochromes based on condensed thiophenes possess the most promising, attractive physicochemical properties. Unfortunately, not all reported methods for their synthesis met our needs to the full extent. For that reason, some part of our research was devoted to the development of new approaches for the formation of condensed thiophenes.

Scheme 1 shows a sequence of reactions that leads to the condenced oxazines, which are themselves of interest for the subsequent synthesis of photochromes and are also readily modified into pyridine derivatives **4** and **5**.

At the first step, amino acids **1** react with the so-called Appel salt resulting in dithiazoles **2**, whose

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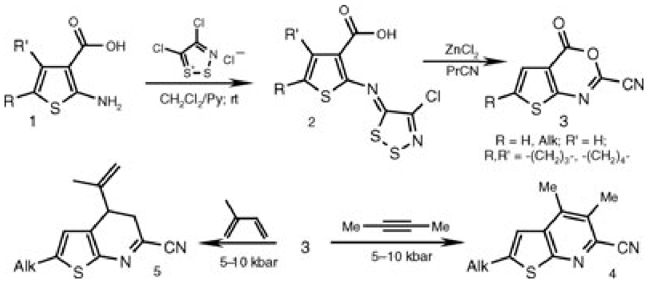

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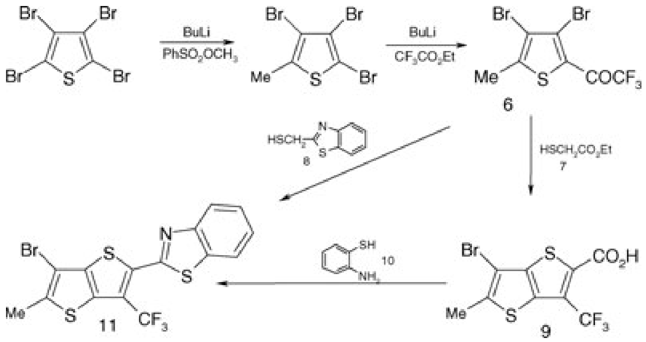

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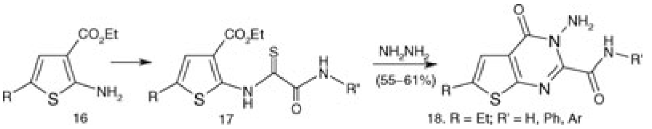

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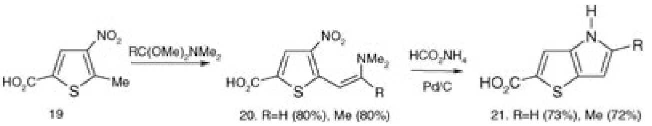


SCHEME 5

subsequent decomposition gives thienooxazines **3**. The latter contains two conjugated bonds that allow us to introduce them in the Diels-Alder reaction under high pressure of 5–10 kbar (under atmosphere pressure, the reactions do not occur) [10]. The synthesis of thienothiophene **11** containing benzothiazole ring is shown in Scheme 2. This substituent was introduced either by a direct interaction of dibromide **6** with thiol **8** or via the transformation of acid **9**, formed from the same dibromide [11].

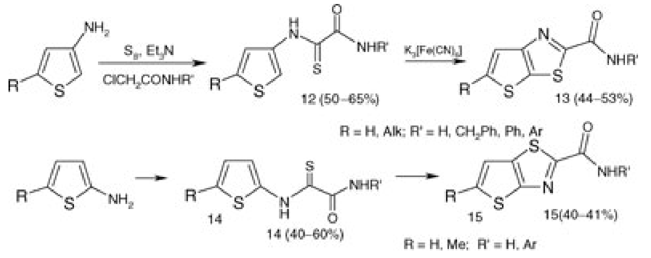
Considerable attention has been given to the elaboration of methods for the synthesis of condensed thiophenes, using exclusively available monothiooxamides [12,13] according to Scheme 3.

Heterocyclic amines were mixed with elemental sulfur and triethylamine in DMF and stirred for at least 30 min. Then, chloroacetamides were added to the obtained mixture to get in high yields monothiooxamides **12** and **14**, whose oxidation gave thienothiazoles **13, 15** [14,15]. In the same way, pyrrolo[3,2-*d*][1,3]thiazoles and thiazolo[4,5-*b*]pyridines were obtained [16,17].

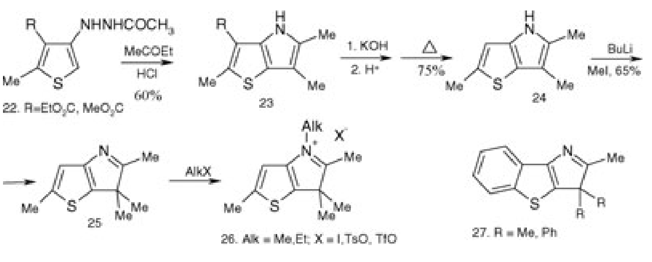
Apparently, monothiooxamides can enter not only the reactions of oxidative cyclization but nucleophilic replacement reactions as well, which allows further condensation into fused six-membered heterocycles (Scheme 4) [18].

Searching for new convenient methods for the synthesis of thieno[3,2-*b*]pyrroles, we investigated the synthetic potential of condensation of *o*-nitrotoluenes with dimethyl acetal of DFA followed by the reduction of nitroenamines obtained from the corresponding indoles [19]. It was demonstrated that the reaction can be successfully applied to the synthesis of thieno[3,2-*b*]pyrroles **21** (Scheme 5) [20].

We focused our attention on the synthesis of earlier not described condensed thieno[3,2-*b*]pyrrolenines, containing geminal methyl groups in the pyrrole ring. An approach to compounds of this type is presented in Scheme 6 [21]. Close transformations of the same type, including the Fisher's rearrangement and giving rise to



SCHEME 3



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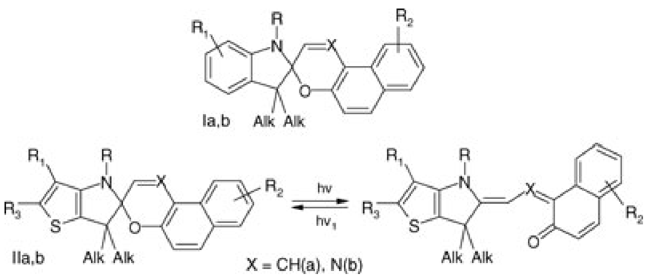


SCHEME 7

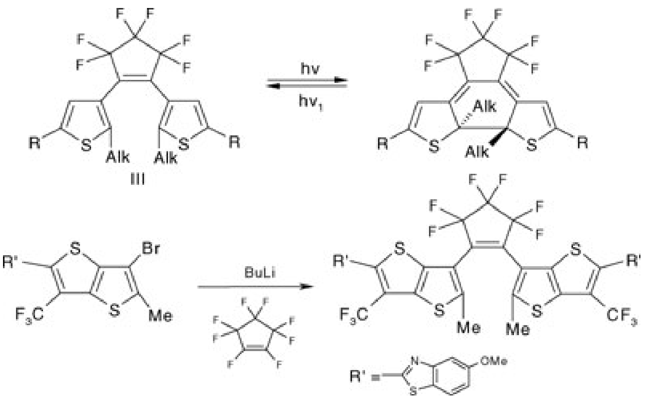
2,3,3-trimethyl-bezo[*b*]thieno[3,2-*b*]pyrrolenine **27** and its analogues, were described by us in [22].

SPIROCOMPOUNDS

It is known that the vast majority of thermally reversible spiropyranes (**Ia**) and spyrooxazines (**Ib**) are obtained from indole derivatives (Scheme 7) [3, pp. 9–14].

We were the first to synthesize spiropyranes **IIa** and spirooxazines **IIb** on the basis of thienopyrroles, that is to create spirophotochromes, in which a benzene ring was replaced with thiophene or benzothiophene.

First representatives of spiropyranes **30–35** were obtained by boiling iodide and triflate salts of the above-mentioned thieno[3,2-*b*]pyrrolenines and benzo[*b*]thieno[3,2-*b*]pyrrolenines with salicylic aldehydes (**28**) or 1-nitroso-2-phenol (**29**) in the presence of organic bases in equimolar quantities (Scheme 8) [17, 23, 24].

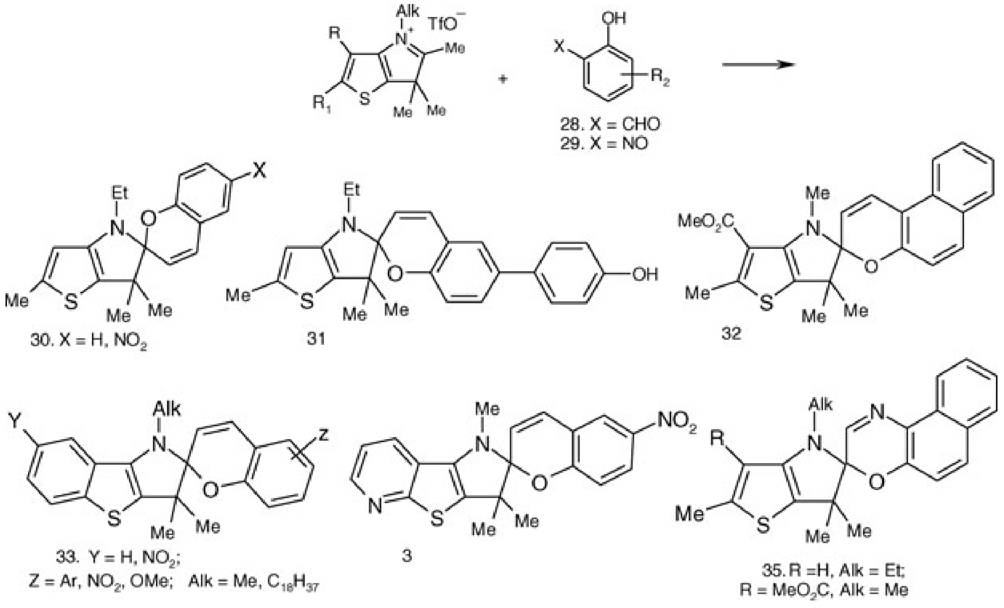


SCHEME 9

Generally, the spectral properties of spiropyranes **30–35** as well as their structures, determined by X-ray crystallographic analysis, resemble to those of traditional indolenine derivatives [22–24]. Possibly, in future research may reveal more subtle differences in optical properties between the benzene and thiophene derivatives of spirocompounds; however, it can be said by now that the presence of a thienyl ring in the products in some cases led to the appearance of thermochromism, electrochromism, and other desired operating properties.

THERMALLY IRREVERSIBLE DIHETARYLETHENES

Scheme 9 illustrates a well-known general approach to the synthesis of dihetarylethenes in which



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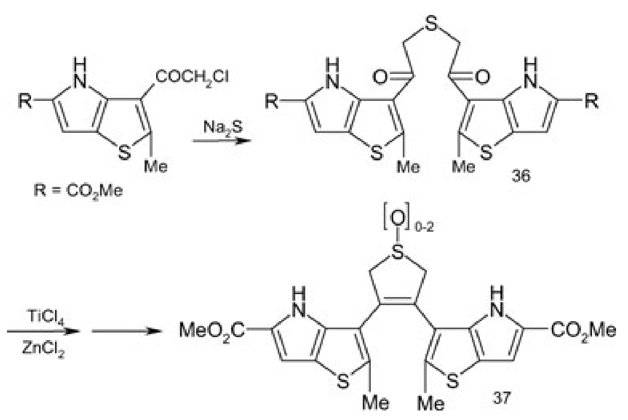

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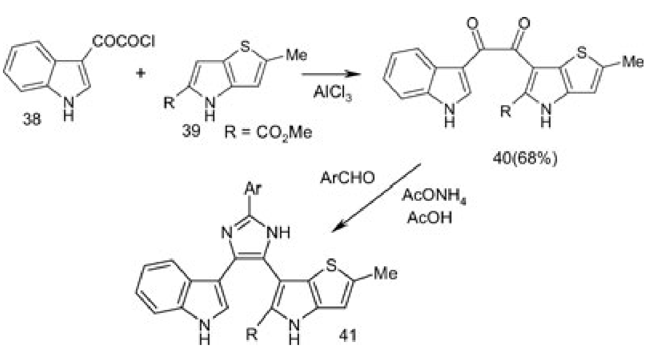


SCHEME 12

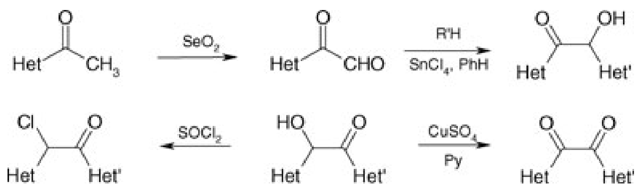
thiophene rings are separated by a perfluorocyclopentene bridge. As a rule, thiophene bromides are used as the starting compounds. They are converted into the corresponding lithium derivatives, which are made to react with perfluorocyclopentene without isolation [11].

Precisely, the method was used for synthesizing a wide range of photochromic compounds in which a perfluorocyclopentene ring is connected with thienyl cycles [25] as well as with benzothienyl [26], thienothiophene [11,27], and dithienothiophene groups [28].

An alternative method for preparation of perfluorocyclopentene, or cyclopentene dihetarylethenes, is a sequence of transformations that ends with the McMurry reaction [29]. The process was employed to obtain products **37** from diketone **36**—a typical subject of this reaction [30] (Scheme 10).

In the search of general approaches to the synthesis of photochromes containing diverse bridges, we paid our attention to such available starting compounds as heterocyclic hydroxyketones, diketones, and chloroketones presented in Scheme 11.

Dihetarylethenes with various bridges were prepared on the basis of these products. The synthesis of trisubstituted imidazoles **41** (in good yields) from diketone **40**—the result of reaction of compounds **38** and **39** under the condition of the Friedel–Crafts process—is reported in [31] (Scheme 12). A vast range of photochromes with dimethylthienyl substituents at positions 4 and 5 of the imidazole cycle is presented in [32].



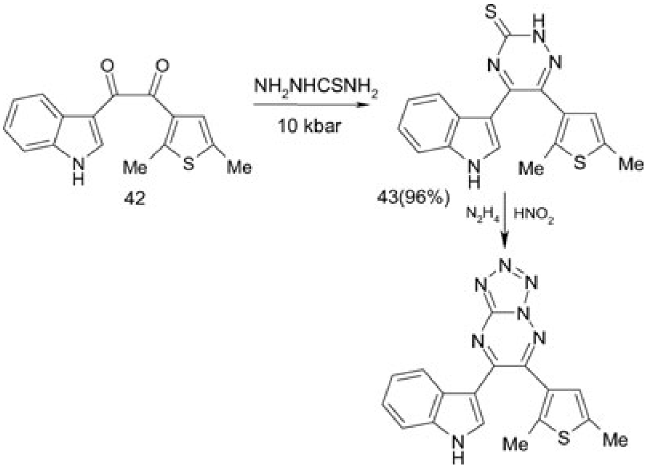
SCHEME 11

We failed to prepare triazine **43** under an atmospheric pressure probably because an electron excessive dimethylthienyl ring decreases the electrophilicity of system **42**, synthesized from indole **38** and dimethylthiophene. However, on increasing pressure up to 10 kbar, the product was obtained in a nearly quantitative yield [31] (Scheme 13).

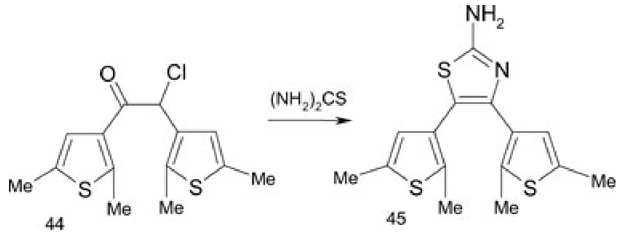
When heated in water, ethanol 2-chloroethane-1-one (**44**) with excess of thiocarbamine affords aminothiazole **45** in 87% yield (Scheme 14) [33]. Thenoin **46** is a very convenient starting compound for synthesis of pyrazine 47 [32] (Scheme 15).

Thenoin forms furan **48** in the reaction with malononitrile, whereas with cyclic enaminone it forms indolone **49** in the presence of DBU base [34] (Scheme 16).

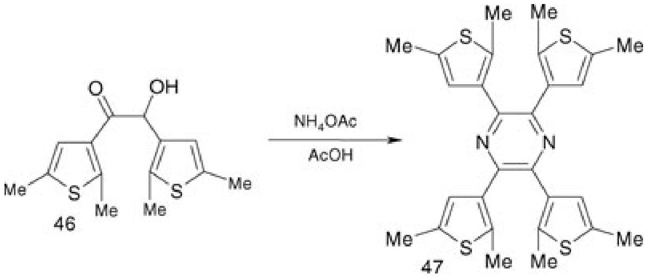
Krayushkin and coworkers [34] describe the reactions of hyrdroxyketone **46** with chloro anhydrides of benzoic acids and ammonia acetate or carbamide



SCHEME 13



SCHEME 14

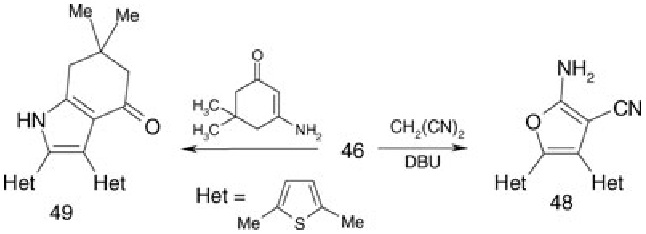


SCHEME 15

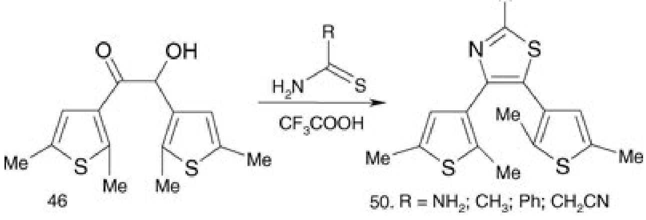
leading to the corresponding oxazoles [33] and imidazolones [35].

A series of publications have been devoted to the interaction of α-hydroxyketones having as a rule dimethylthienyl cycles with di-nucleophiles in organic acids. Thus, the reaction of thiocarbamide (or thioamides) with thenoin **46** smoothly leads to the formation of thiazoles **50** (Scheme 17) [36].

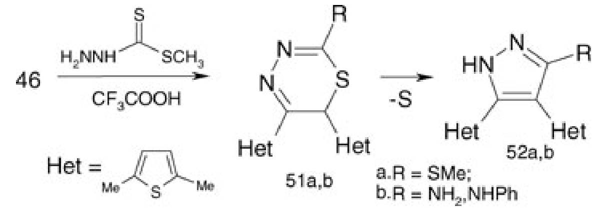
Similar conditions are required to prepare unstable thiadiazine **51a** that spontaneously converts to pyrazole **52a**. Unlike thiadiazine **51a**, products **51b** proved to be stable and were isolated from the reaction mixture. The subsequent transformation on prolonged heating in dioxane gives the respective pyrazole **52b** with good yield [36] (Scheme 18).



SCHEME 16



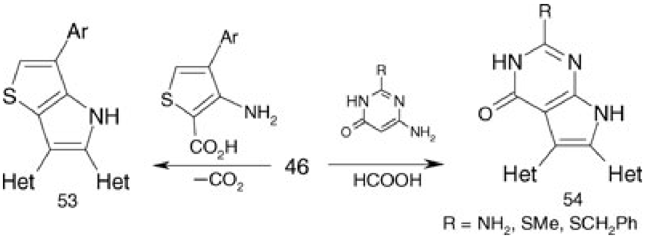
SCHEME 17



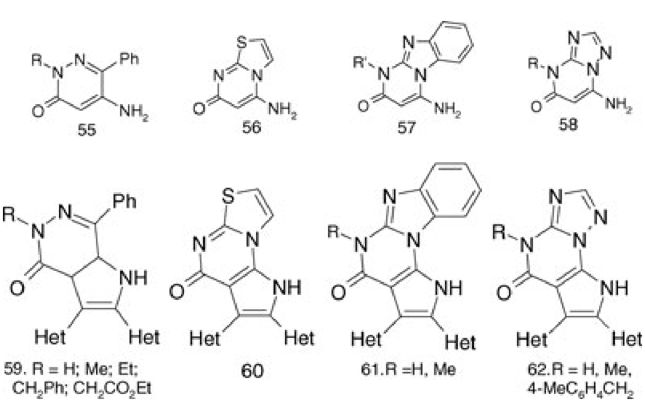
SCHEME 18

Good yields have also been obtained when thenoin **46** reacted with heterocyclic enamines in acid medium. In a similar way photochrome **53** with a thienopyrrole bridge was prepared [37]. The process is naturally accompanied by decarboxylation (Scheme 19). The reaction of thenoin with pyrimidine enamines leads to the corresponding pyrimidine derivatives **54**.

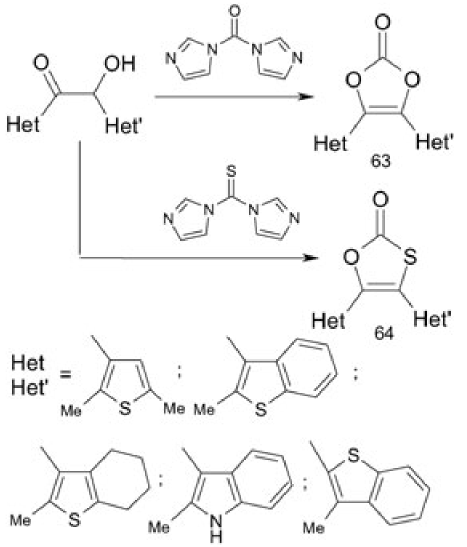
The reaction in acetic acid was also applied to aminopyridazines **55** and condensed aminopyrimidines **56–58** that allowed us to synthesize compounds **59–62** in 40–80% yields [37] (Scheme 20).



SCHEME 19



SCHEME 20

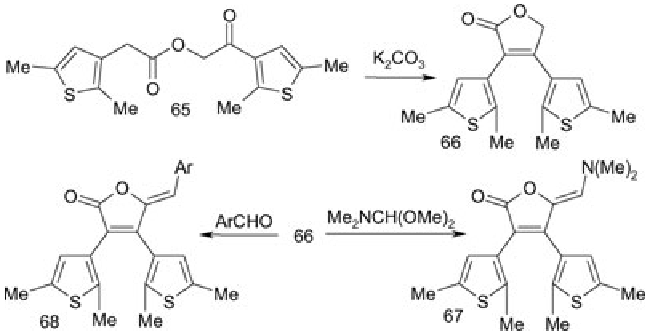


SCHEME 21

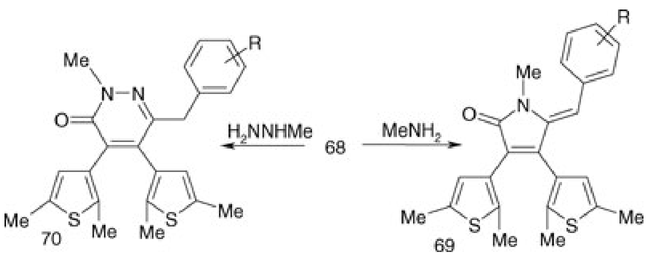
The reaction of thenoins with 1,1′-carbonyl diimidazole went in a traditional direction to give dioxalone 63 (Scheme 21).

However, using 1,1′-thiocarbonyl diimidazole in the reaction with thenoins (Scheme 21), we observed a new unexpected route of the reaction: instead of the expected thione, we prepared in high yields the products **64** whose structure was proved by X-ray diffraction analysis [38]. The reaction with 1,1′-thiocarbonyl diimidazole is of a general character and can be extended, in particular, to benzoins and aroins.

Lactones **66**, obtained from acids, haloketones, or from the corresponding esters of type **65**, exhibit photochromic properties and can be also very convenient for the synthesis of photochromic dihetarylethenes with various bridges [39] (Scheme 22).



SCHEME 22



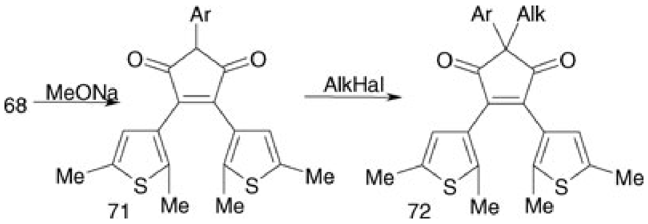
SCHEME 23

Thus we have shown that lactone **68** converts to lactam **69** in a very good yield while reacting with methylamine in trifluoroacetic acid. We have also demonstrated that the reaction of furanones **68** with hydrazines gives pyridazinones **70** (unpublished data) (Scheme 23). This reaction proceeds smoothly and gives high yields (80–95%) that allow us to consider it as a convenient method for the synthesis of photochromes containing a pyridazinone-binding fragment.

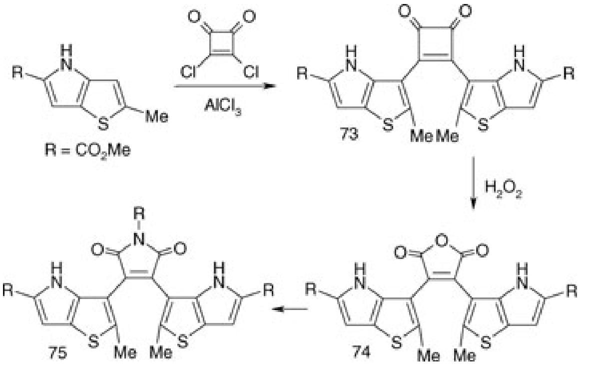
We have found that a rearrangement takes place upon the reaction of furanones **68** with sodium methylate, which leads to the formation of cyclopentadiones **71** (unpublished data) (Scheme 24). The reaction is of a general character and gives a possibility to obtain products with diverse aromatic and heterocyclic substituents in 86–95% yields.

Special attention has been given to derivatives of maleic anhydride and amide that are very promising substances for optical memory [40]. The problem was that the methods had been developed for their synthesis prior to the beginning of our investigations and they were rather far from being perfect [41].

We have developed some quite effective approaches to these products. According to Scheme 25, we synthesized the squaric acid derivative **73** in good yield [42]. The cyclobutanedione moiety in this case, like in many others [43], is the key structure in the synthesis of photochromes derived from maleic anhydride or maleimide.



SCHEME 24



SCHEME 25

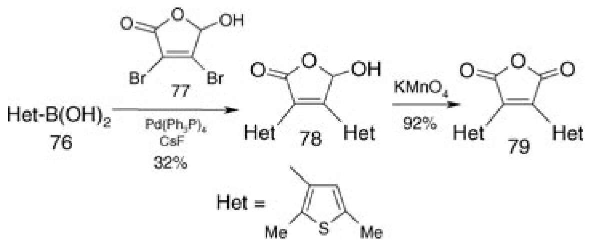
Oxidation of the product **73** gives maleic anhydride **74** in almost quantitative yield whose further amination also leads to maleimide **75**.

The second approach through cross-coupling reactions also proved to be convenient and reliable one for the synthesis of thienyl derivatives of maleic anhydride **79** [44] (Scheme 26). In this scheme, thienylboronic acid **76**, which can be readily obtained from the suitable bromide, serves as one of the component. The second component is rather cheap and is available as mucobromic acid **77**. Derivatives of palladium in the presence of fluoric cesium act as typical catalysts in this type of reactions.

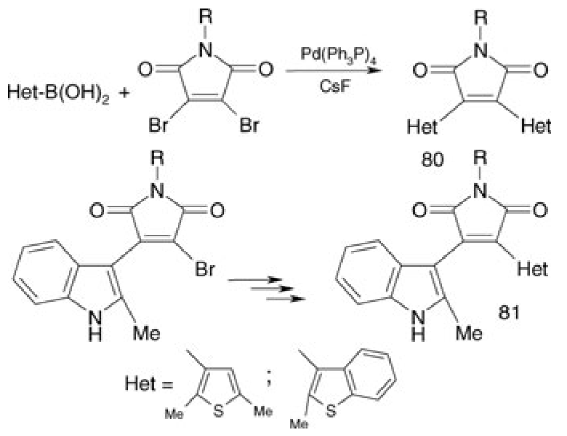
Generally, it can be stated that despite moderate yields of the target product **79**, the suggested method deserves attention: the starting compounds are available rather cheap, and the techniques are simple and can be reliably reproduced.

We have studied in detail the reaction of thienylboronic acids with 3,4-dibromo-*N*-butylmaleimide by an example of the synthesis of products **80**, optimized the process conditions, and demonstrated that this cross-coupling reaction is of a general character and can be successfully employed in the case of other heterocyclic and aromatic boronic acids. The suggested modification of the cross-coupling reaction has been successfully extended to the synthesis of nonsymmetrical products **81** [45] (Scheme 27).

The obtained amides **80** and **81** were hydrolyzed in a dioxane alkaline solution to give the respective maleic anhydrides [45]. No doubt, this is a rather paradoxical approach, because as a rule, in most of cases these are maleimides that are obtained from anhydrides. However, in this very case such an approach to the synthesis of maleic anhydrides seems to be quite efficient, taking into account incomparable availability of maleimides.



SCHEME 26

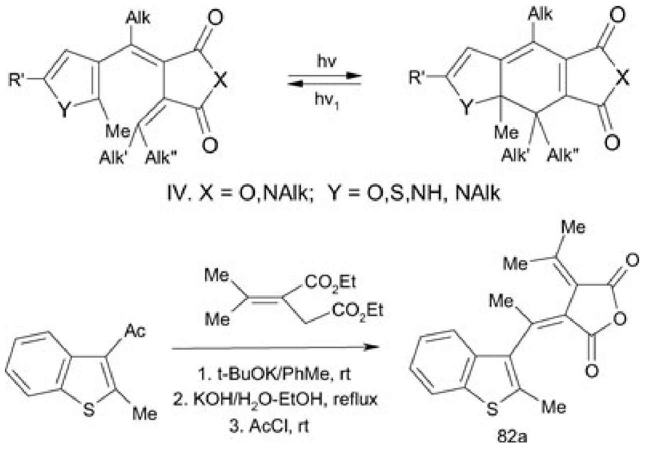


SCHEME 27

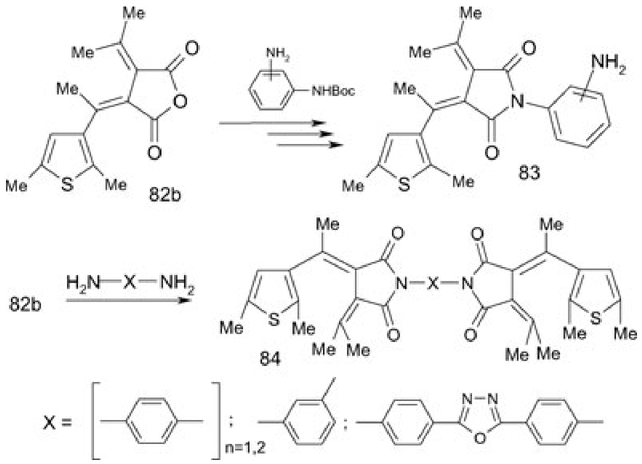
On the whole, it is possible to say that the problems of the synthesis for photochromes with maleic anhydride and maleimide bridges have been successfully solved.

FULGIDES AND FULGIMIDES

An analogous mechanism of cyclization, when an open-ring hexatriene is converted reversibly to a closed-ring cyclohexadiene with UV-light, is realized in the case of the derivatives of maleic anhydride and maleimide the so-called fulgides (X = O) and fulgimides (X = NAlk) (Scheme 28). Depending on their structure, these compounds can



SCHEME 28

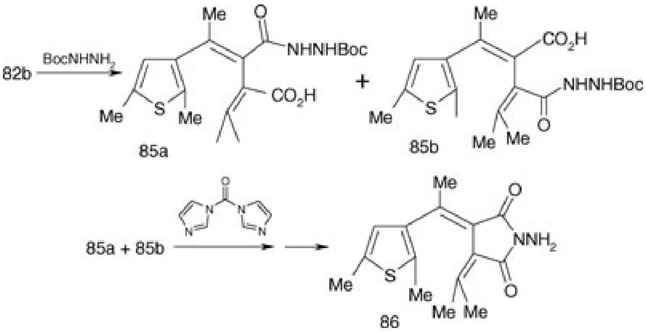


SCHEME 29

possess properties both of spiroproducts and dihetarylethenes and then can be the components of color compositions or elements of optical memory. In our research, fulgides IV. X = O were mainly used as the starting compounds. Their typical synthesis by the example of product **82a** is shown in Scheme 28.

The interaction of the initial fulgide **82** with the prior prepared *m*- and *p*- Boc-substituted anilines, leading to the synthesis of amines **83** and symmetrical bis-fulgimides **84**, which are attractive as components for the three-dimensional optical memory devices, is described in [46] (Scheme 29).

Aiming at the synthesis of *N*-aminofulgimide **86**, we have attempted to introduce fulgide **82** in the reaction with hydrazine hydrate under different conditions (Scheme 30). However we could not obtain the target product because of the formation of multicomponent mixtures which were difficult to separate. The subsequent transformations were carried out using the Boc-protection. In this case, the reaction proceeded in a specific manner: we showed that substance **82** reacted with Boc-hydrazine in boiling benzene for 10 h and gave the mixture of hydrazides of acid **85a** and **85b** in 80% yield. We found that various cyclizing reagents, normally used for cyclization of amide acids to give imides, in this case had no effect. The reaction was a success only when *N*,*N*′-carbonyl diimidazole was involved as a cyclizing reagent [46]. The quantitative removal of the protective Boc-group took place in the final stage of the synthesis [47]. Summing up, we can state that carbonyldiimidazole is a novel versatile reagent for transformation of fulgides into fulgimides, which has a number of advantages against the traditional reagents employed for this purpose.



SCHEME 30

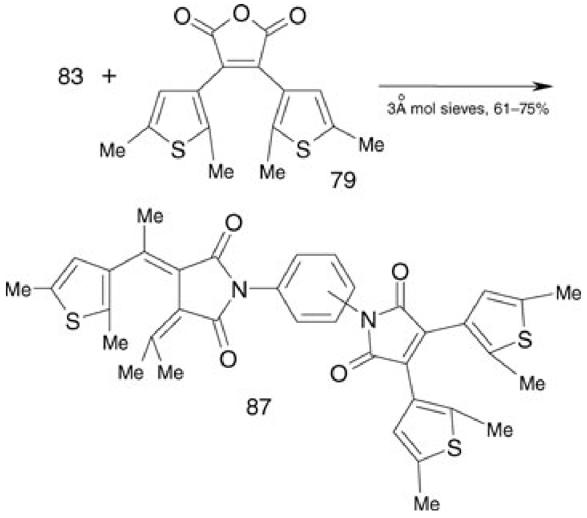
All fulgimides synthesized manifest photochromic properties, forming cyclized forms under UV light.

Having fulgimides **83** and **86**, as well as dihetarylethenes with maleic anhydride bridge, we have the possibility to synthesize the new specific hybrids—the compounds in which fulgimide photochromic fragments are connected with photochromic dihetarylethene moieties.

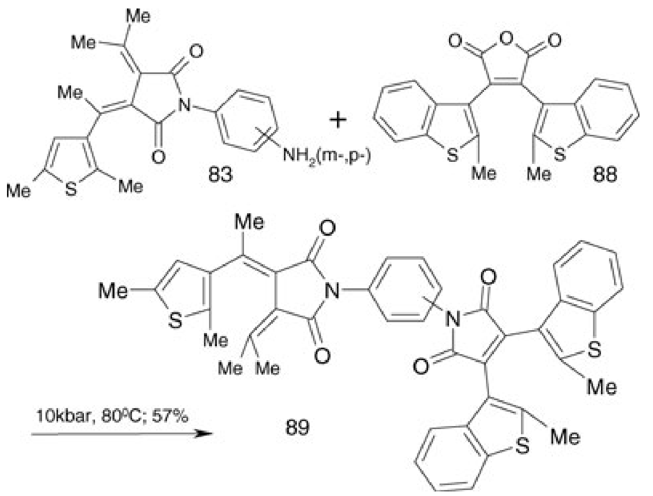
The first two hybrid photochromes **87** with meta- and paraorientations of the parts have been prepared after prolonged boiling of amines **83** with anhydride **79** in ethanol, under an atmosphere of argon, in the presence of molecular sieves 3Å, in 61–75% yields (unpublished data) (Scheme 31).

An analogous approach was applied for the synthesis of a hybrid photochrome **89**, starting from amine **83** and 4,5-bis(benzothienyl)maleic anhydride **88** (Scheme 32).

However, prolonged boiling of the reagents in ethanol did not lead to the desired result. Even after 100 h, only the starting compounds were observed in the reaction mixture. Attempts to increase

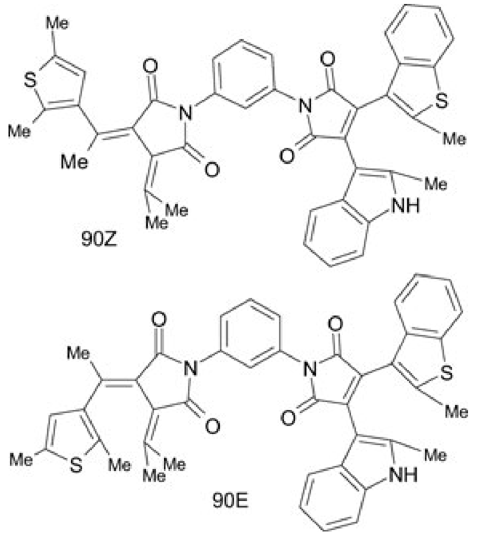


SCHEME 31



83 88 89

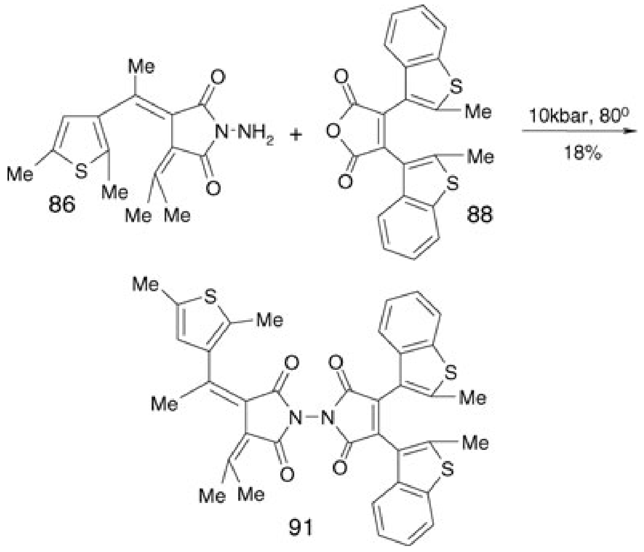
SCHEME 32



90Z

90E

SCHEME 33



86 88 91

SCHEME 34

the rate of the reaction by replacing ethanol with higher boiling solvents gave no result. The addition of *p*-toluenesulfonic acid in catalytic amount or such bases as DBU and 1,1,3,3-tetramethylguanidine also gave no result.

We supposed that a method of high pressure would promote the formation of maleimide cycle in this case. In fact, after keeping the solution of amine **83** and anhydride **88** in equimolar amounts in THF for 12 h at 800°C, under pressure of 10 kbar, followed by flash chromatography on SiO_2, the target hybrid photochromes **89** with *m*- or *p*-maleimide substituents were isolated from the reaction mixture in about 50% yields (unpublished data).

The method of high pressure was also successfully applied to the synthesis of hybrid photochromes **90**, in the forms of *Z* and *E* isomers, which are separated by chromatography on silica gel (Scheme 33).

The synthesis of the hybrid **91** (Scheme 34) in which the fulgimide and dihetarylethene fragments are connected directly by nitrogen atoms is of special interest.

We have not found any other method for the formation of such products reported in the literature. Photochrome **91** was obtained in the reaction of *N*-aminofulgimide **86** with anhydride **88** at pressure 10 kbar. PMR-spectra showed that the hybrids prepared under high pressure have *E*-configurations of the fulgimide fragments. The nature of this phenomenon is not quite clear, taking into account that the starting amines **83** and **86** had *Z*-configuration. Probably, the molecules of *E*-isomers are of a smaller volume and the transfer of *Z*-isomers to *E*-isomers is accompanied by a partial decrease in the volume of the system, which weakens the external action of high pressure. The cases of alkene isomerization under high pressure have been reviewed in [48].

CONCLUSION

Our data show that most of dihetarylethenes, fulgimides, and their hybrids we synthesized are thermally irreversible. Taking into account high fatigue resistance of their close analogues, reviewed in the literature, it can be supposed that they can be very useful as switches and for optical memory devices.

These above examples will suffice to show that thienyl-containing compounds play an increasingly important part among photochromes. An introduction of thiophene cycles into spiropyrane or spirooxazine products, fulgides, and fulgimides makes it possible to extend substantially the range of their physicochemical and photochromic properties. In turn, it is thienyl-containing substituents in dihetarylethenes that provide the uniqueness to these substances, their high fatigue resistance, and thermal irreversibility. It is apparent that further investigations in this direction will enlarge practical application of photochromic materials.

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