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McMurry reaction in the synthesis of photochromic dihetarylethenes

M M Krayushkin, M A Kalik, V A Migulin

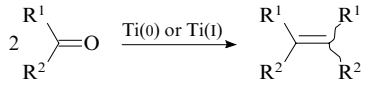
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Abstract. Application of the McMurry reaction in the synthesis of thermally stable photochromic dihetarylethenes is described. Hetaryl rings in the target molecules are connected to the double bond of the cycloalkenes or heterocycles formed upon the McMurry cyclization. The bibliography includes 66 references.

I. Introduction

A possibility of reductive dimerization of carbonyl compounds to yield alkenes in the presence of low-valence titanium species generated *in situ* was simultaneously discovered in the 1970s by three independent research groups: French,[1] Japanese[2] and American.[3] Subsequently, this new reaction was named after one of the discoverers — John E. McMurry.



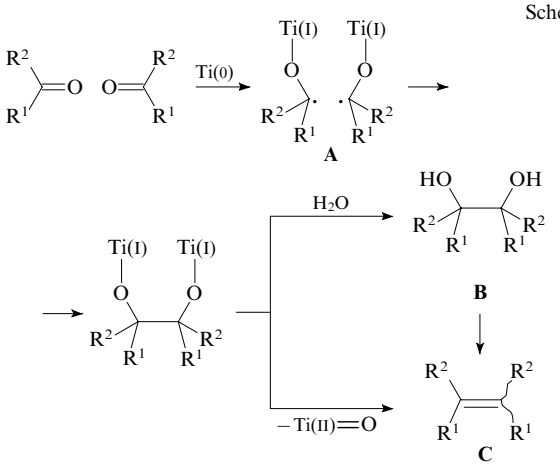
R^1, R^2 = H, Alk, Ar.

The condensation could be performed both intra- and intermolecularly. The mechanism of the McMurry reaction (Scheme 1) includes the formation of a ketyl radical **A** (or a biradical in the case of intramolecular cyclization), which then dimerizes to the corresponding pinacol **B**. The formed pinacols could be isolated in this stage of the reaction at low temperatures, while at higher temperatures, they easily undergo deoxygenation leading to the target alkene **C**. It was shown that these reactions occur on the surface of titanium.[4]

M M Krayushkin, M A Kalik, V A Migulin N D Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, Leninsky prosp. 47, 119991 Moscow, Russian Federation. Fax (7-499) 135 53 28, tel. (7-499) 137 69 39, e-mail: mkray@ioc.ac.ru (M M Krayushkin), mary@ioc.ac.ru (M A Kalik), tel. (7-916) 319 96 65, e-mail: vmiguli@mail.ru (V A Migulin)

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Scheme 1



Two ways of conducting the McMurry reaction are being employed. According to a primary developed two-step sequence, titanium chloride $TiCl_x$ (x = 3, 4) is reduced to metallic titanium with a powerful reducing agent (K, Na, Li, $LiAlH_4$, *etc.*) (first step), which is then followed by slow addition of either carbonyl, dicarbonyl, or a mixture of two carbonyl compounds (second step).

The reducing agent – solvent system turned out to be critical in the McMurry reaction. For instance, the reduction of $TiCl_3$ with Na or Mg could only be performed in tetrahydrofuran (THF), while with Li, K and $LiAlH_4$ this transformation could also be accomplished in glyme, diglyme or diethyl ether. It was shown that of all solvents tested, THF performed best in all instances.

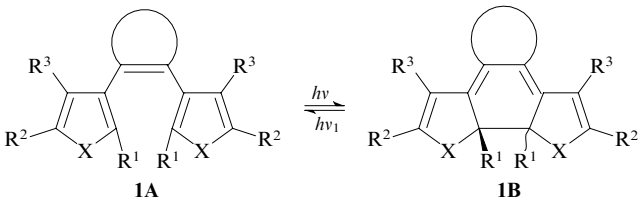
Subsequently this procedure was replaced by a more convenient method[5] where $TiCl_x$ is reduced to the low-valence titanium species in the presence of a carbonyl substrate by less active reducing agents such as zinc or iron powder. It was shown that metallic zinc is capable of reducing titanium chloride(III) or (IV) only to titanium(II). However, the redox potential of $TiCl_2$ becomes much lower upon coordination to a carbonyl compound, which allows further reduction to metallic titanium inducing the McMurry reaction. Thus, the pre-coordination of titanium species to the carbonyl oxygens alleviates the reaction, which usually results in lower reaction times and generally significantly higher yields.[6]

The McMurry reaction has been widely employed in organic synthesis; several reviews have appeared on this subject.[7–11]

Low-valence titanium turned out to be a unique reagent for the McMurry reaction. None of other transition metals gave consistently so high yields of alkenes, the products of reductive dimerization of carbonyl compounds. Despite a strong reducing power of low-valence titanium species, this method, however, is applicable to carbonyl compounds having other functional groups such as Hal, OH, SiR_3, NH_2, OR, SR, and CH=CHR. In some cases the substrates possess nitrile, amide, ester, or some other less active carbonyl groups that survive the reaction conditions. Incompatible with the McMurry reaction are carbonyl compounds containing nitro groups, fragments of allylic and benzylic alcohols, 1,2-diols, epoxides and halohydrins, as well as enones, quinones, α-halo ketones, oximes, sulfoxides.[8]

The reviews[8,9] show how the McMurry reaction is applied in the synthesis of natural products and their analogues including sterically hindered alkenes, macrocycles, carbo- and heterocycles, different monomers and polymers. Elaboration of the McMurry reaction resulted in developing of other synthetic areas: coupling of imines,[12] acyl silanes,[13] and intramolecular ketone–ester cyclization.[14]

Nowadays, the McMurry reaction serves as a key transformation in the synthesis of photochromic 1,2-dihetarylethenes **1** that undergo reversible interconversion under irradiation from the open form **A** to the cyclic form **B**.



1A **1B**

This type of photocyclization occurs only in *cis*-1,2-dihetarylethenes, which could be achieved by including the double bond into a cyclic fragment.

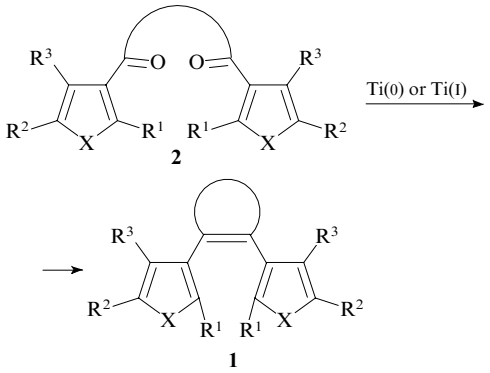
Photochromic materials have an extensive application worldwide: as a basic example, they have been widely used in manufacturing of sunglasses. Also photochromes are being employed in actinometers, dosimeters,[15,16] and in the field of optoelectronics.[17] Recent progress in the laser and information technologies makes photochromic materials promising for use as recording media in devices for recording, storage, and processing of information.[18–21] Obviously, the properties of such photochromes should satisfy the most diverse set of requirements including thermal stability (lack of interconversion of forms **A** and **B** in the absence of radiation), high fatigue resistance (ability to multi-switch without any loss of performance properties), high speed response to the effects of radiation, maximum remoteness of absorption bands of open **A** and cyclic **B** forms, sufficient solubility in organic solvents (film manufacture), ability to undergo photochromic transformations not only in solution, but also in the solid state, *etc.*[18,22,23]

Syntheses, phisycal properties and various applications of different photochromic materials can be found in reviews[24–28] and monographs.[29–31]

It should be noted that most of dihetarylethenes obtained under McMurry conditions satisfy these requirements, which largely determines the interest in this reaction.

II. Synthesis of photochromic dihetarylethenes by the McMurry reaction

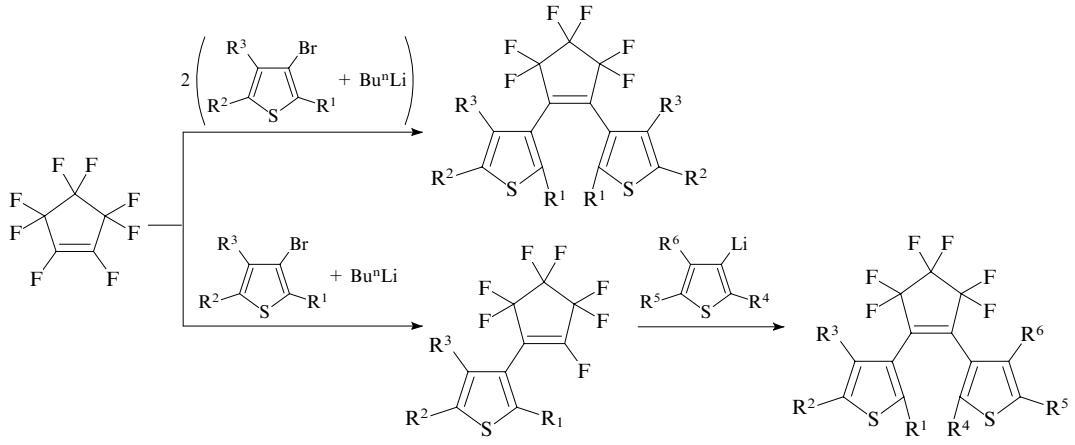
Photochromic dihetarylethenes **1** are usually synthesized either by a consecutive coupling of hetaryl fragments with a cycloalkene or heterocycle containing a C=C double bond or by construction of the cycloalkene fragment in the final steps of the synthesis. The McMurry reaction allows formation of the central cyclopentene bridge fragment in one step, starting from the corresponding diketones **2**.



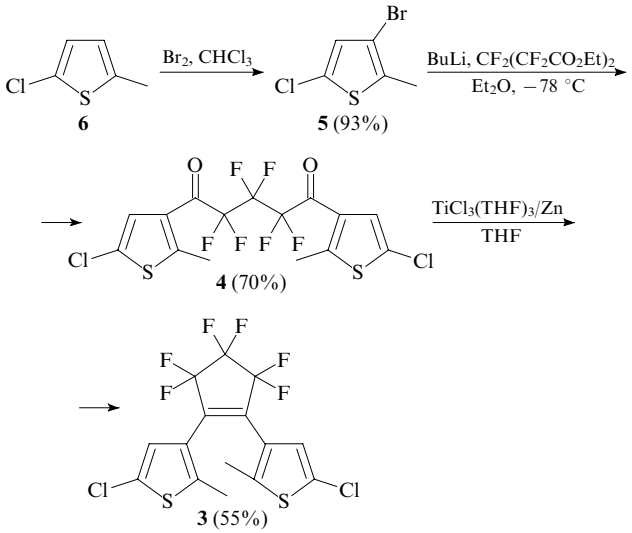
2 **1**

Presently, dithienylethenes with a perfluorocyclopentene bridge are the most popular due to their physical and chemical properties such as high fatigue resistance and thermal stability.[32,33] Photochromes of this class (both symmetrical and non-symmetrical) are usually synthesized by consecutive substitution of the thienyl anions for the fluorine atoms at the C=C bond in perfluorocyclopentene (Scheme 2).[34]

Scheme 2



An alternative synthetic route to symmetrical 1,2-dithienylperfluorocyclopentenes can be based on the intra-molecular McMurry reaction. In this condensation, the central perfluorocyclopentene fragment is formed from the starting 1,5-dithienylperfluoropentane-1,5-diones. For example, photochrome **3** was obtained in good yield:[35, 36]

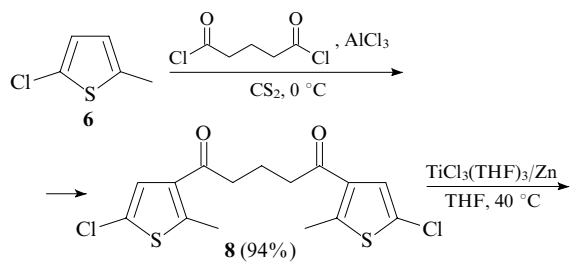


Ti(0,I) species were obtained from $TiCl_3$ by zinc reduction. In the course of this reaction the solvent molecules stabilize the titanium species formed. In addition, the reduction rate is higher where the solvent molecules participate in coordination of titanium. In this case, THF was used as a solvent, which formed the complex $TiCl_3(THF)_3$.

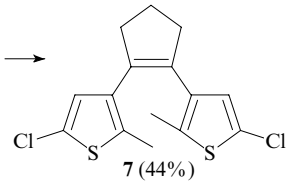
Synthesis of the key intermediate **4** included generation of an anion from bromide **5** by treatment with butyllithium, followed by its attack on diethyl hexafluoroglutarate. The direct acylation of 2-chloro-5-methylthiophene **6** with hexafluoroglutaryl dichloride was unsuccessful in this case.[36]

Analogous dihetarylethenes with a cyclopentene bridge are much less studied; however, they also possess attractive photochromic properties and can be considered as a promising alternative to dithienylperfluorocyclopentenes.† To date, the McMurry reaction proved to be the most effective and efficient method for the synthesis of this class of compounds. At the same time, 1,5-diketones, as precursors for the intramolecular McMurry reaction, usually are conveniently obtained by simple Friedel–Crafts acylation of the corresponding heterocycle with glutaryl dichloride in the presence of a Lewis acid. Being combined these two methods afford an easy synthetic access to photochromes with a cyclopentene bridge.

Using this strategy, the Feringa research group synthesized dithienylethene **7** in 1998.[39]



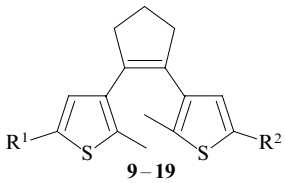
† A comparison of spectral and photochromic properties for these two classes of photochromes has been published recently.[37, 38]

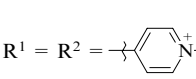


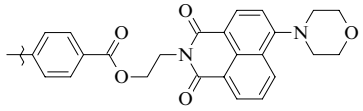
The key intermediate **8** was obtained in excellent yield by direct acylation of commercially available 2-chloro-5-methylthiophene **6** with glutaryl dichloride.

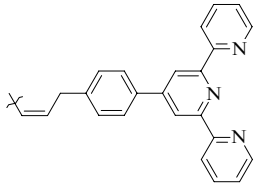
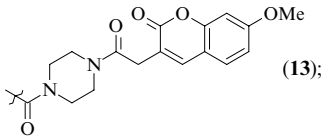
In the crystal structure of **7**, the ring skeleton of the molecule is not planar, having two substituted thiophene rings in *cis* position with respect to the double bond of the cyclopentene ring; this explains the lack of photochromic properties of this compound in the solid state.[40]

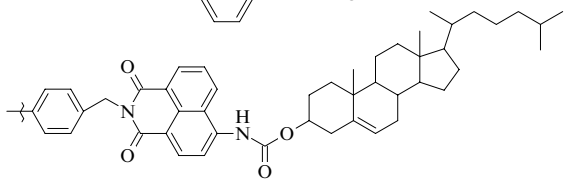
The possibility of lithiation of chlorides in the target molecule **6** allowed further chemical modification of this photochrome into a variety of photochromic 5,5′-disubstituted dithienylperhydrocyclopentenes with different physical and chemical properties. Some of the synthesized compounds **9**–**19** are of applied interest.

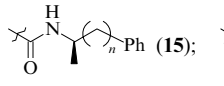
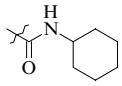


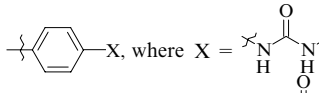
$R^1 = R^2 =$  (**9**);  (**10**);

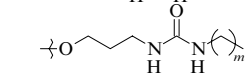
 (**11**);

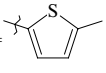
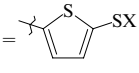
 (**12**);  (**13**);

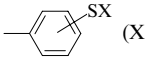
 (**14**);

 (**15**);  (**16**);

 X, where X = (n = 5, 7, 11) or

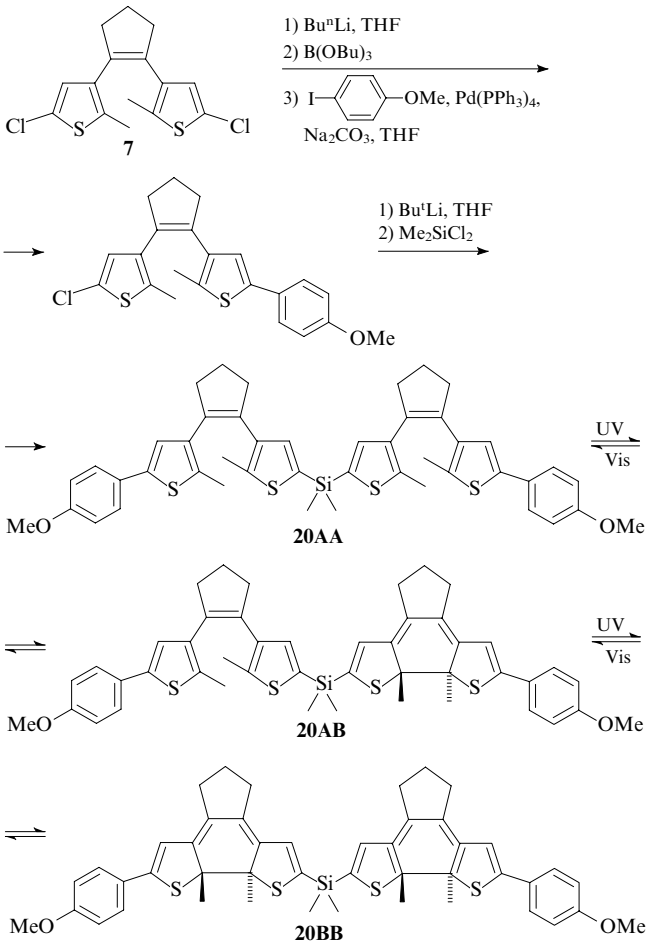
 (m = 3, 5, 11) (**17**);

$R^1 =$ , $R^2 =$  (X = Ac, Au nanoparticles) (**18**);

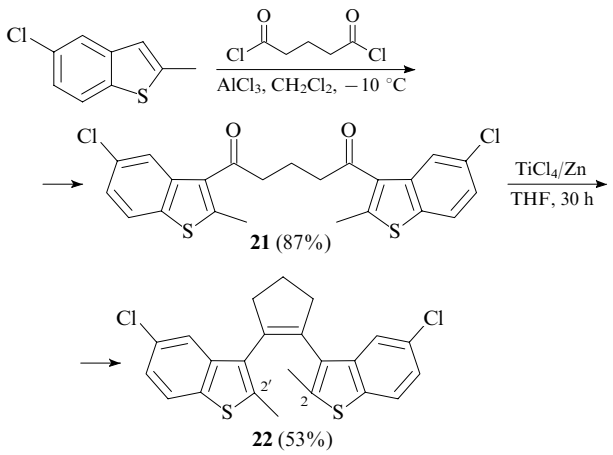
R^1 = Ph, $R^2 =$  (X = Ac, Au nanoparticles) (**19**).

Thus water soluble pyridinium zwitter-ions **9** were used in assembly of a monolayer-functionalized electrode that allow the sequential electrochemical and photochemical storage of information;[41] bis[5-(4-pyridyl)-2-methylthiophen-3-yl]cyclopentene (**10**) was employed as a photoswitch responding to Zn^{2+} ions, protons, and alternating UV/Vis radiation;[42] 5,5′-disubstituted bis(thienyl)cyclopentenes with naphthalimide (**11**)[43] or terpyridine (**12**)[44] units, as fluorescent optical switches; bis(3-thienyl)cyclopentene containing coumarin fragments (**13**), as a tuning energy transfer system;[45] compounds **14**–**17**, as photoresponsive organogels that show gel-to-liquid transition upon irradiation;[46–48] and compounds **18** and **19**, as spacer-dependent uni- and bi-directional light-induced switches of diarylethens on gold nanoparticles.[49, 50]

Another synthetic application of dichloride **7** has recently been demonstrated by the Feringa group.[51] Two-component molecular photo-switches **20** were synthesized, which consisted of two dithienylcyclopentene moieties bridged through a silicon atom.



Similarly, photochromic dihetarylethenes containing benzothienyl units were described.[52, 53] For instance, 1,5-diketone **21**, employed in the McMurry condensation, was prepared by the direct Friedel–Crafts acylation of 5-chloro-2-methylbenzo[*b*]thiophene with glutaryl dichloride in excellent yield. The chlorine atoms in the benzothiophene fragments could serve as functional precursors for further chemical modifications of **22**.



Krayushkin *et al.*[53] have studied photochromic behaviour of compound **22** in both toluene solution and crystalline state. It was shown that physicochemical properties of **22** in toluene solution were similar to those of other known photochromes with a cyclopentene bridging unit.[37, 38] X-Ray diffraction analysis of a crystal of **22** (Fig. 1) showed the benzothiophene rings to be in *anti*-parallel conformation, which is a prerequisite for photochromic interconversion of open and ring forms. Nevertheless, the distance between the centres C(2) and C(2′) is equal to 5.358 Å, which considerably exceeded the calculated[54] limit of ~4.2 Å for such photocyclization to proceed. As a result, no photochromism of crystalline compound **22** was observed.

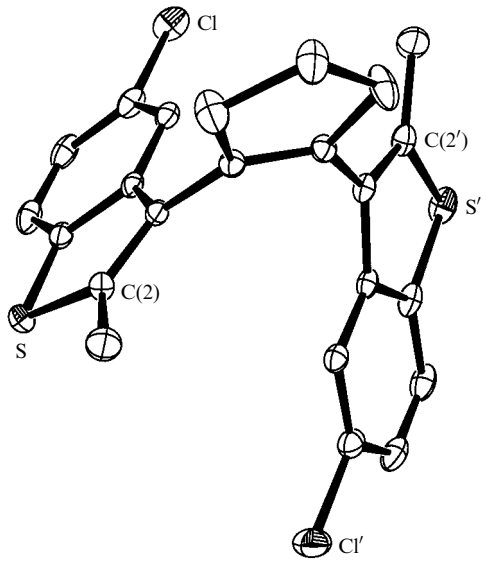
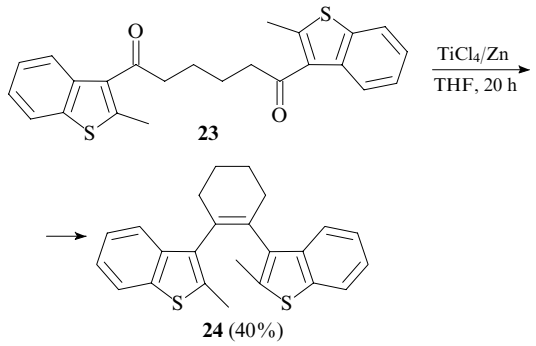


Figure 1. The crystal structure of 1,2-bis(2-methyl-5-chlorobenzo[*b*]-thiophene-3-yl)cyclopentene (**22**).

A similar pattern is typical of several photochromes with a perfluorocyclopentene bridging unit.[55]

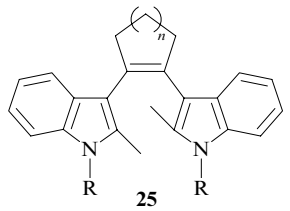
The use of adipic dichloride in the acylation instead of glutaric dichloride gave rise to the corresponding 1,6-diketone **23**, which also condensed under the McMurry conditions. However, the reported yield (40%) of formed 1,2-bis(2-methylbenzo[*b*]thiophen-3-yl)cyclohexene (**24**) turned out to be inferior to its cyclopentene analogue.[52]



23

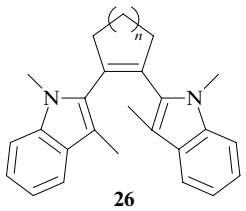
24 (40%)

Besides the photochromes with thienyl fragments, indole-based 1,2-bis(indolyl)cycloalkenes, *e.g.*, **25** and **26**,[52, 56, 57] and furan-based 1,2-bis(2,5-dimethylfuran-3-yl)-cyclohexane (**27**)[52] are known. The synthetic strategy of these compounds also includes the McMurry cyclization of the corresponding 1,5- or 1,6- diketones.



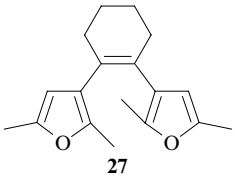
25

R = Alk; *n* = 1, 2.



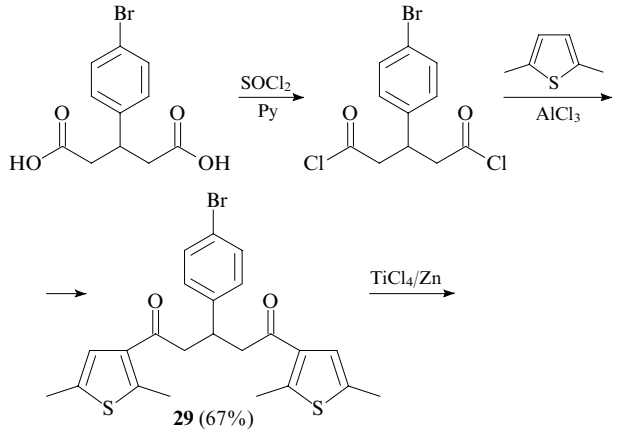
26

n = 1, 2.

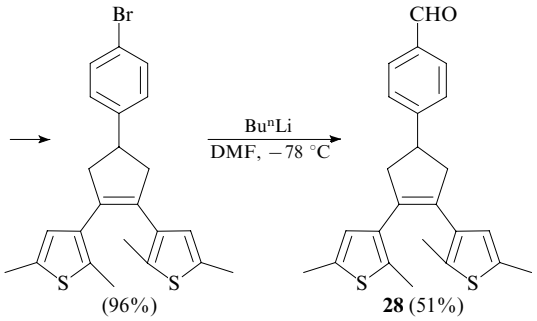


27

Modification of the connecting bridge in 1,2-bis(hetaryl)cycloalkenes is a promising way of acquiring novel types of photochromes. This can be achieved by introduction of different substituents into the cyclopentene fragment or by replacing a CH_2 group by a heteroatom. Thus photochrome **28** was synthesized under the McMurry conditions from 1,5-diketone **29**, the latter being obtained from 3-(4-bromophenyl)glutaric acid.[58]



29 (67%)

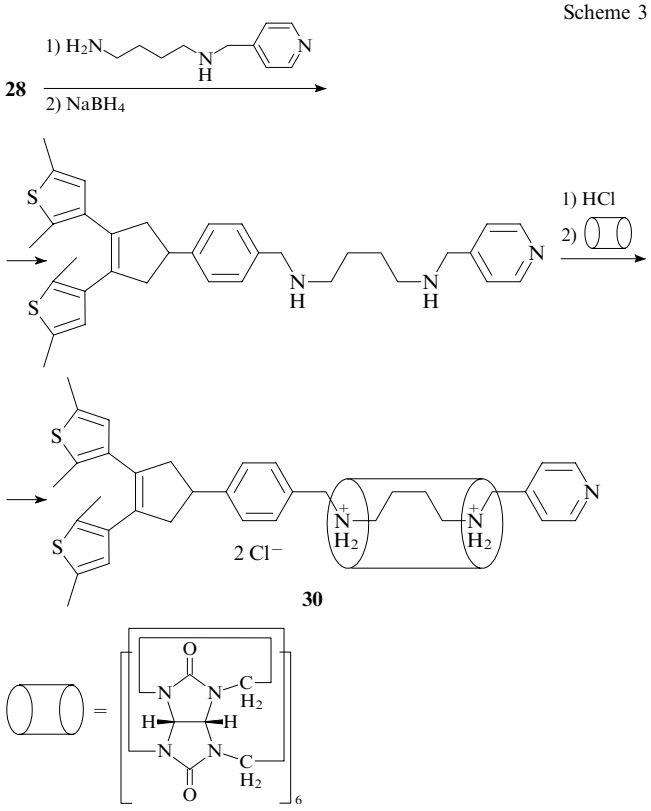


(96%)

28 (51%)

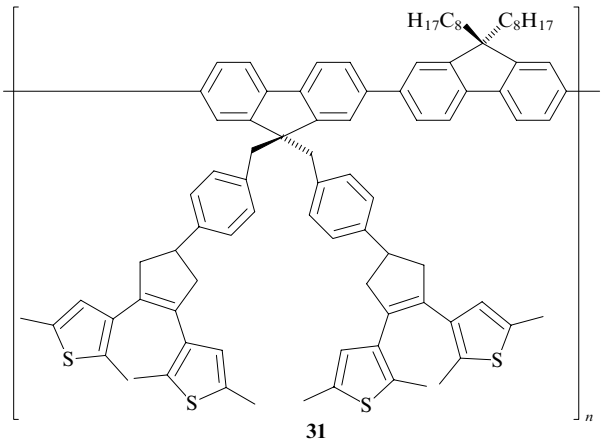
Starting from aldehyde **28**, *N*-substituted diaminobutane, and cucurbit[6]uril, a novel pseudorotaxane **30** with diarylethene as the functional stopper was recently assembled (Scheme 3). This type of compounds can be used in the construction of the prototype of molecular machines.[59]

Scheme 3



30

Photochrome **28** also served as a precursor to photochromic copolymer **31** with two dithienylethene substituent

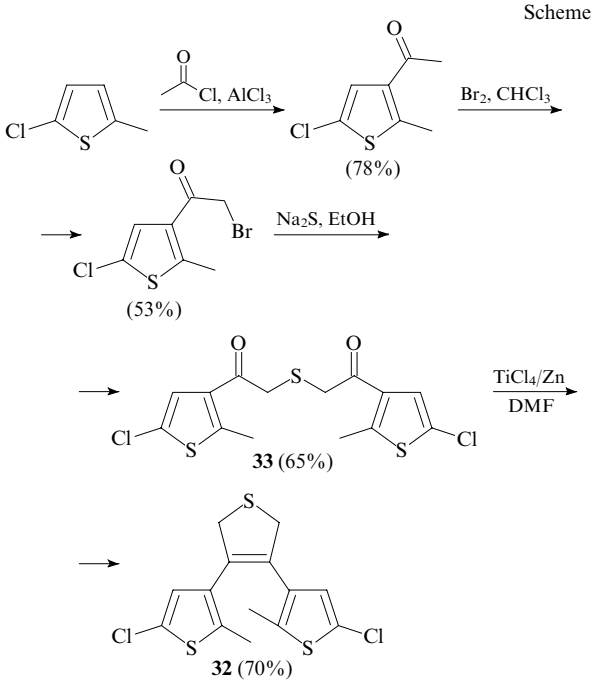


31

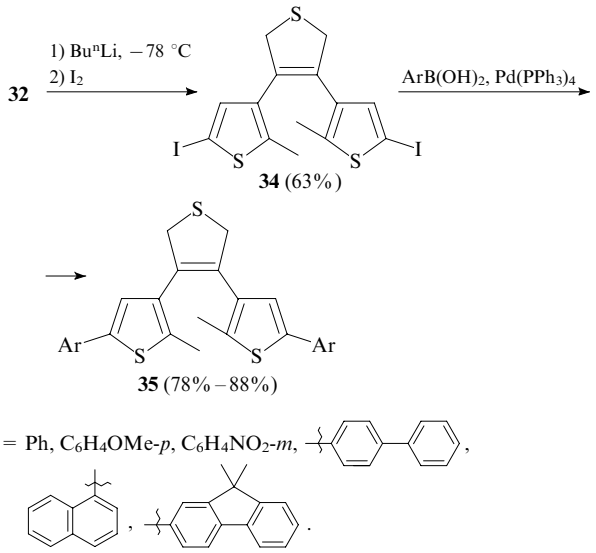
at one fluorene segment. The target polymer **31** displayed photochromic properties in both solution and the solid state.[58] This type of polymers could be applied in optoelectronic technologies.

1,2-Dithienylethene **32** contains a 2,5-dihydrothiophene fragment as a bridge instead of the cyclopentene unit.[60] This compound was synthesized by the intramolecular McMurry reaction from 3-thia-1,5-diketone **33** according to Scheme 4.

Scheme 4

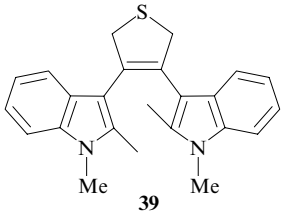


Further functionalization of **33** could be accomplished by replacing chlorine atoms in the thiophene units by other substituents. For example, Cl/I halogen exchange in **33** furnished diiodide **34**, which reacted with a variety of arylboronic acids under the Suzuki conditions to give different 5,5′-diaryl derivatives in high yield (78% – 88%).[61]



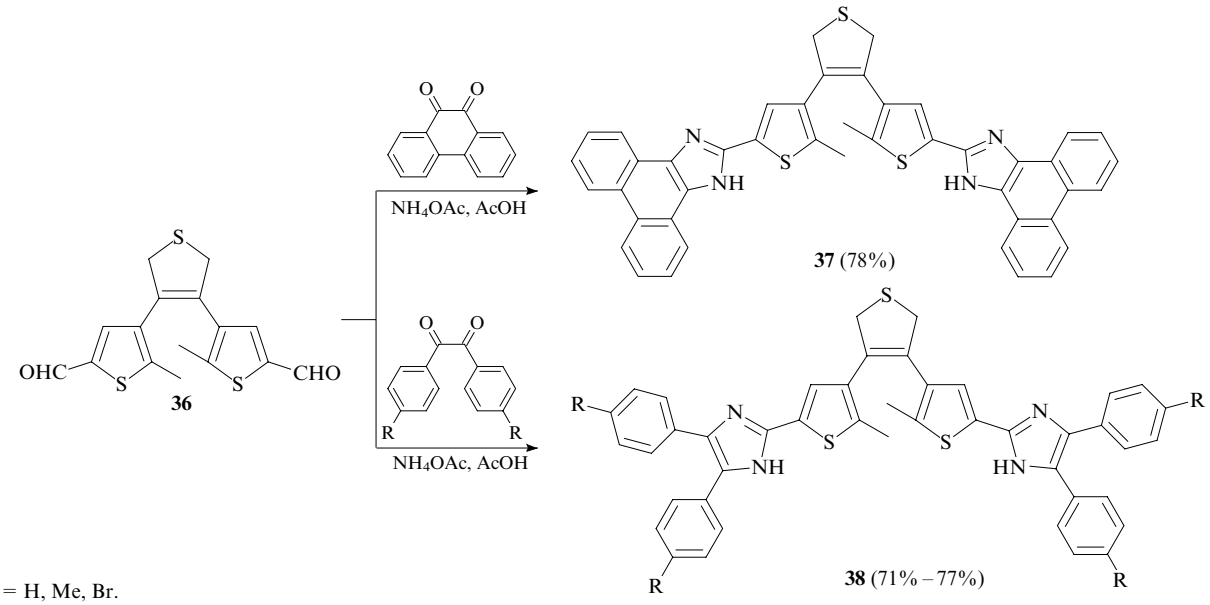
5,5′-Diformyl derivative **36** reacts with either phenanthroquinone or substituted benzils in acetic acid in the presence of ammonium acetate to give fluorescent dithienylethenes **37** and **38**, respectively (Scheme 5).[62]

Photochromes with nitrogen-containing heterocycles at the 2,5-dihydrothiophene bridge were also described. For example, 3,4-bis(indol-3-yl)-2,5-dihydrothiophene (**39**)[63] and thienopyrrole-based compound **40** (Ref. 64) were synthesized by the McMurry condensation. However, the yields of target photochromes were rather low.



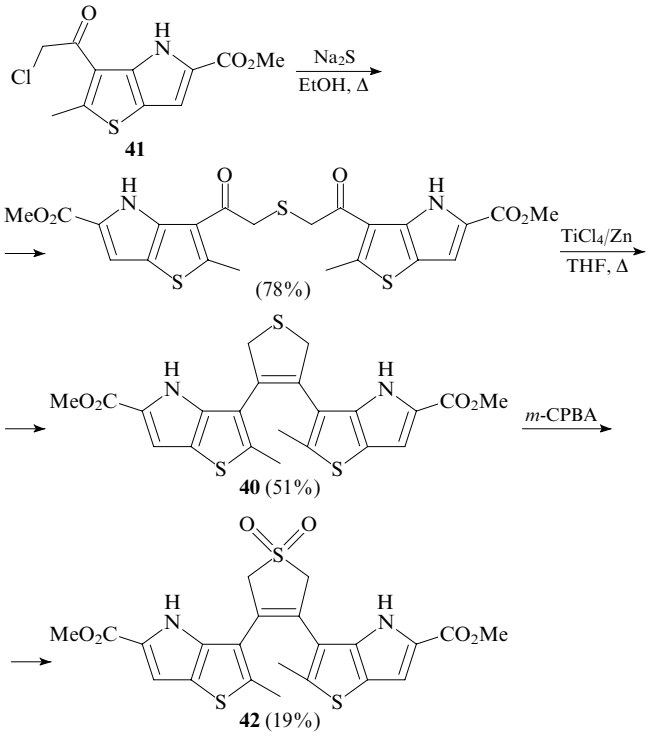
Thieno[3,2-*b*]pyrrole **41** was employed as a starting material to photochrome **40**, which was subsequently con-

Scheme 5



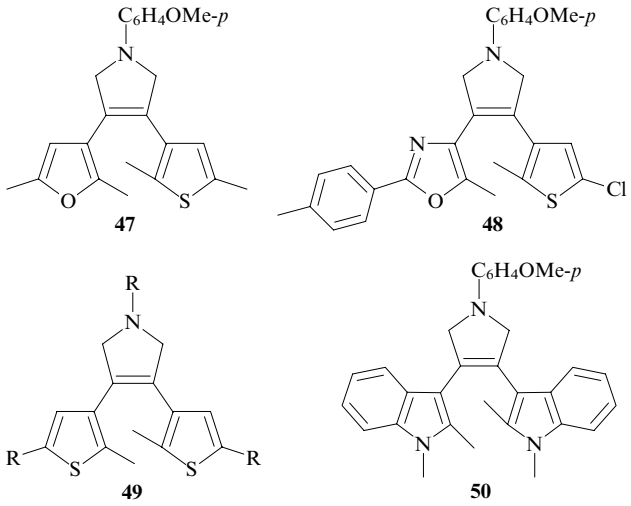
R = H, Me, Br.

verted into photochromic sulfone **42** in 19% yield by oxidation with *m*-chloroperoxybenzoic acid (*m*-CPBA).[64]



The McMurry reaction could be successfully applied to the synthesis of dihetarylethenes with a dihydropyrrole bridge from 3-*aza*-1,5-diketones as the starting compounds. Thus coupling of 4-aminophenol with 2 equiv. of 3-bromoacetyl-2,5-dimethylthiophene provided diketone **43**, which was condensed to photochrome **44** under the McMurry conditions. Further treatment of **44** with either benzoyl chloride or benzene-1,3,5-tricarbonyl trichloride furnished chromophore **45** and symmetrical chromophore **46**, respectively (Scheme 6).[65]

In an analogous manner, various symmetrical and non-symmetrical photochromic 3,4-dihetaryl-2,5-dihydropyrroles **47**–**50** with furyl, thienyl, oxazolyl, and indolyl heterocycles were synthesized in good yields (60%–80%).[66]

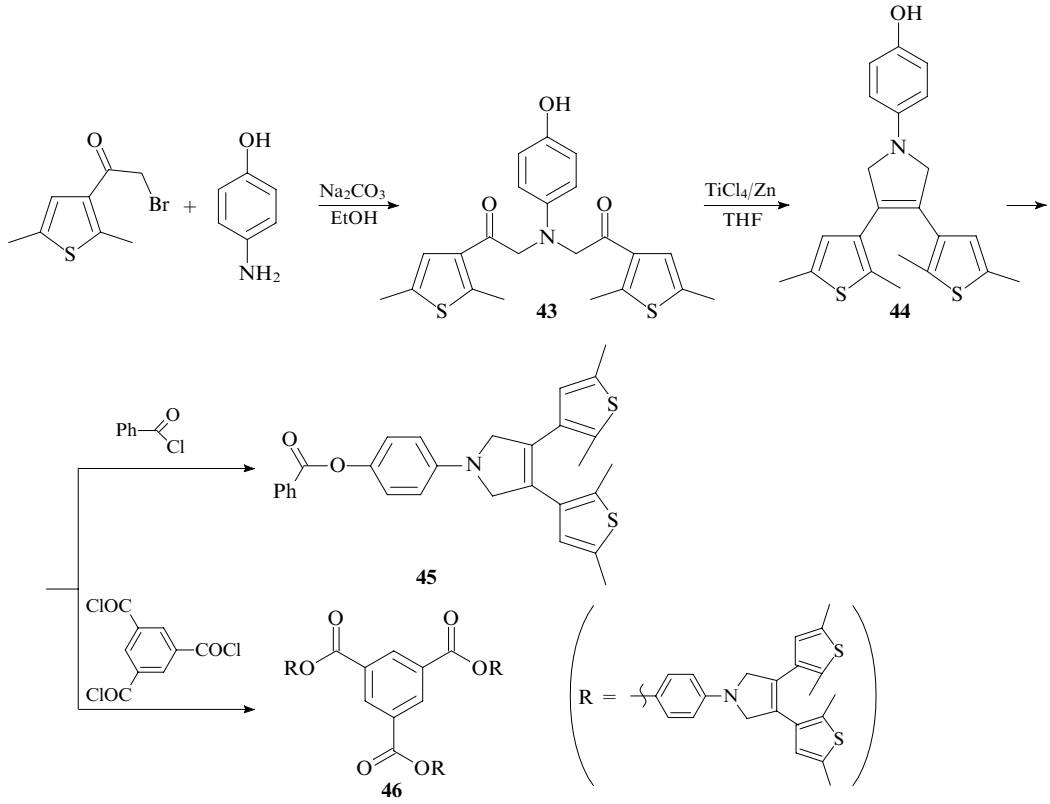


R = Me, Cl, CHO.

III. Conclusion

The described examples demonstrate an extremely high efficiency of the McMurry reaction in the synthesis of a variety of photochromic dihetarylethenes. At the same time, it has several drawbacks such as high dependence on reaction conditions, sometimes low reproducibility, and a huge range of yields from excellent to inferior — the facts

Scheme 6



that strongly encourage further search for optimal and general reaction conditions for the McMurry reaction.

On the other hand, most of the reported to date photochromic molecules are obtained from symmetrical diketones, which predetermines their symmetrical structure. It is apparent that further elaboration of the McMurry reaction in the synthesis of thermally stable irreversible dihetarylethenes should be focused not only on extension of its use in terms of numbers of reported novel structures, but also on finding a general method for the synthesis of both symmetrical and non-symmetrical photochromes. Obviously, this search should also include a development of simple and reliable methods of synthesis of the precursor non-symmetrical diones.

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