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A one-pot synthesis and mild cleavage of 2-[2- or 5-(alkylsulfanyl)pyrrol-1-yl]ethyl vinyl ethers by *t*-BuOK/DMSO: a novel and facile approach to *N*-vinylpyrroles

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ABSTRACT

Substituted *N*-[2-(vinyloxy)ethyl]pyrroles, prepared in good yield through an allenic or acetylenic carbanion/isothiocyanate one-pot methodology from 2-(vinyloxy)ethyl isothiocyanate and allyloxyallene, methoxyallene, *N*,*N*-dimethyl-2-propyn-1-amine, and 3-methoxy-1-(methylsulfanyl)-1-propyne, are smoothly converted into the corresponding *N*-vinylpyrroles using *t*-BuOK/DMSO (room temperature). The reaction proceeds via elimination of vinyl alcohol from the *N*-[2-(vinyloxy)ethyl] substituent and represents a novel approach to *N*-vinylpyrroles.

N-Vinylpyrroles are of interest as monomers, as useful synthetic intermediates and building blocks for the design of various polymeric materials and new azole-containing mono-, poly-, and macrocyclic molecular architectures for diverse purposes (optoelectronics, non-linear optics, materials science, supramolecular chemistry, medicine, pharmacology, etc.).[1,2] Traditionally, these compounds are prepared by the addition of NH-pyrroles to alkynes in the presence of various catalytic systems,[2,3] less often by indirect vinylation with dihaloalkanes (through substitution–elimination),[1a,n,2,4] by vinylic substitution with vinyl halides[1k,2,5] and vinyl triflates,[2,6] or by isomerization of *N*-allylic into *N*-propen-1-ylic derivatives.[2,7] In particular, the one-pot syntheses of diversely substituted *N*-vinylpyrroles directly from ketones and acetylene (via ketoximes) in the superbasic MOH/DMSO (M = Na, K, Cs) systems (Trofimov reaction) are synthetically attractive.[8]

It has also been well documented that the introduction of heteroatoms or heteroatom-containing substituents at different positions of the pyrrole ring can change drastically the physical, chemical, and biological properties of the corresponding pyrrole derivatives.[9–12] Among the heterosubstituted pyrroles, demanded are those bearing amino,[10] alkoxy,[11] and alkylsulfanyl[12] groups, which are recommended for the design of optoelectronic materials, new biologically relevant porphyrins and as pharmaceutically important substrates. However, the availability of such compounds is poor, and the corresponding *N*-vinylpyrroles, to the best of our knowledge, are still unknown. In fact, until recently, general and efficient methods for the direct synthesis of heterosubstituted pyrroles were absent. Apart from our own previous work,[13,14] only a few reports in this field have been published.[15]

We recently developed conceptually novel general strategies for the highly selective one-pot assembly of heterosubstituted pyrroles from simple and readily available reagents, that is, isothiocyanates, allenes or alkynes and alkylating agents (through synthesis and intramolecular cyclization of 1-aza-1,3,4-trienes),[2b,13] or from allyl isothiocyanate (through its transformation into an *N*,*S*-centered pyrrolylsulfide dianion in the presence of KDA).[14] In this Letter, we describe a novel and general synthetic approach to new classes of *N*-vinylpyrroles, hitherto unknown, and synthetically unavailable 2-(alkylsulfanyl)-*N*-vinylpyrroles bearing functional substituents—based on synthesis (via allenic or acetylenic carbanion/isothiocyanate one-pot methodology)[13] and an unprecedented mild cleavage of 2-[2- or 5-(alkylsulfanyl)pyrrol-1-yl]ethyl vinyl ethers using *t*-BuOK/DMSO (see Schemes 1–6).

In continuation of our interest in the development of novel pyrrole ring-construction strategies,[13,16] we have examined the one-pot preparation [from 2-(vinyloxy)ethyl isothiocyanate (**1**), allyloxyallene, and MeI (Scheme 1)] of polyfunctionalized

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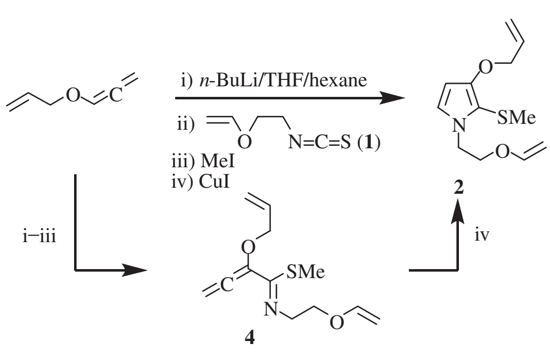
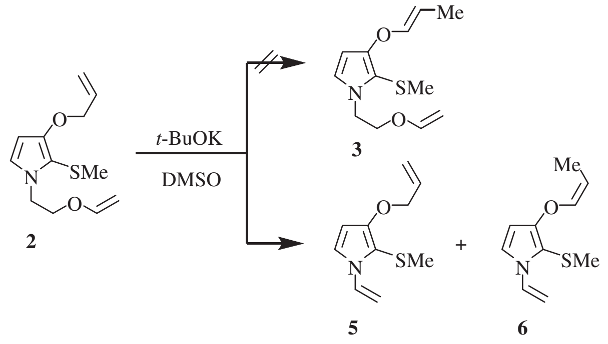
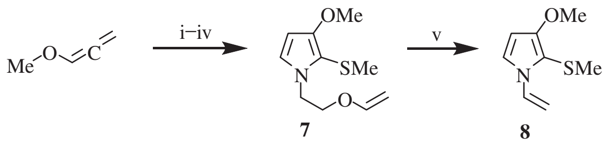


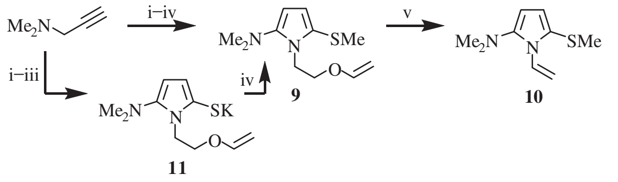
Scheme 1.



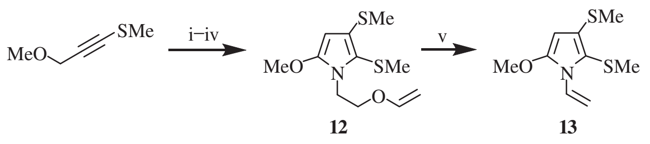
Scheme 2.



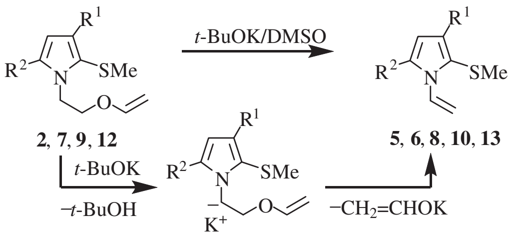
Scheme 3. Reagents and conditions: (i) *n*-BuLi/THF/hexane; (ii) CH$_2$=CHOCH$_2$CH$_2$N=C=S (**1**); (iii) MeI; (iv) CuI; (v) *t*-BuOK/DMSO: 1.2 equiv, rt, 60 min (74%); 2 equiv, ∼50 °C, 20 min (95%).



Scheme 4. Reagents and conditions: (i) *n*-BuLi/THF/hexane; (ii) CH$_2$=CHOCH$_2$CH$_2$N=C=S (**1**); (iii) *t*-BuOH/*t*-BuOK/DMSO; (iv) MeI; (v) *t*-BuOK/DMSO, 2 equiv, rt, 60 min or ∼112 °C, 10 min.



Scheme 5. Reagents and conditions: (i) *n*-BuLi/THF/hexane; (ii) CH$_2$=CHOCH$_2$CH$_2$N=C=S (**1**); (iii) MeI; (iv) CuBr (74%); (v) *t*-BuOK/DMSO, 2 equiv, rt, 60 min.



Scheme 6. Reagents: R^1 = OCH$_2$CH=CH$_2$, R^2 = H (**2**, **5**); R^1 = OCH=CHCH$_3$, R^2 = H (**6**); R^1 = OMe, R^2 = H (**7**, **8**); R^1 = H, R^2 = Me$_2$N (**9**, **10**); R^1 = SMe, R^2 = OMe (**12**, **13**).

3-(allyloxy)-2-(methylsulfanyl)-*N*-[2-(vinyloxy)ethyl]pyrrole (**2**) as a highly reactive monomer, synthon, and precursor of 2-(methylsulfanyl)-3-(1-propenyloxy)-*N*-[2-(vinyloxy)ethyl]pyrrole (**3**) which is the first representative of vinyloxy-substituted pyrroles, combining the useful properties of both pyrroles and vinyl ethers (Scheme 2).

The reaction proceeds via formation and Cu$^+$-catalyzed[16a] intramolecular cyclization of allenylimidothioate (1-aza-1,3,4-triene) **4**, which can be easily isolated, if necessary. Pyrrole **2** was obtained in 72% isolated yield (Scheme 1).

However, our attempt to carry out smooth allyl–propenyl isomerization of 3-(allyloxy)pyrrole **2** into 3-(1-propenyloxy)pyrrole **3** under basic conditions (0.54 equiv of *t*-BuOK, DMSO, 45–55 °C, 45 min) gave an unexpected result. Instead of target pyrrole **3**, previously unknown 3-(allyloxy)-2-(methylsulfanyl)-*N*-vinylpyrrole (**5**) and 2-(methylsulfanyl)-3-[(*Z*)-1-propenyloxy]-*N*-vinylpyrrole (**6**) were isolated in ∼55% total yield (ratio of **5**/**6** = ∼50:50), together with starting pyrrole **2** (∼60% in a mixture of products), the conversion of **2** being ∼59% (Scheme 2).

A more detailed investigation of the reaction showed that these transformations of pyrrole **2** occurred even at rt. Thus, treatment of pyrrole **2** with 2 equiv of *t*-BuOK in DMSO at rt for 60 min led to pyrroles **5** and **6** in ∼66% total yield (ratio of **5**/**6** = 27:73), the conversion of **2** being ∼87%. At ∼40 °C for 30 min, pyrroles **5** and **6** were obtained in ∼76% total yield (ratio of **5**/**6** = ∼5.3:94.7), the conversion of **2** being ∼94%. In all the cases, formation of pyrrole **3** was not evident by analytical methods (NMR, IR and MS).

The generality of the reaction leading to *N*-vinylpyrroles was confirmed using a number of other 2,3-, 2,5- and 2,3,5-substituted *N*-[2-(vinyloxy)ethyl]pyrroles. Thus, 3-methoxy-2-(methylsulfanyl)-*N*-[2-(vinyloxy)ethyl]pyrrole (**7**)[13a,17] was obtained in one preparative step from isothiocyanate **1**, methoxyallene and MeI (Scheme 3) in 62% isolated yield. The treatment with *t*-BuOK/DMSO easily transformed **7** into 3-methoxy-2-(methylsulfanyl)-*N*-vinylpyrrole (**8**) in good to excellent yield, depending on the reaction conditions.

Interestingly, attempts to effect this transformation with *t*-BuOK/THF or KOH/DMSO (in both cases with 1.2 equiv of base at ∼50 °C for 20 min) did not afford the expected *N*-vinylpyrrole **8**. When ∼0.5 equiv of *t*-BuOK (DMSO, ∼50 °C, 20 min) was used, the amount of *N*-vinylpyrrole **8** in the mixture with pyrrole **7** was not higher than 50%.

The reaction of *N*,*N*-dimethyl-5-(methylsulfanyl)-*N*-[2-(vinyloxy)ethyl]pyrrol-2-amine (**9**), obtained from *N*,*N*-dimethyl-2-propyn-1-amine and isothiocyanate **1**, with 2 equiv of *t*-BuOK/DMSO gave *N*,*N*-dimethyl-5-(methylsulfanyl)-*N*-vinylpyrrol-2-amine (**10**) in 97% yield (the conversion of **9** being 98%) when the reaction was carried out at ∼112 °C (Scheme 4). At rt the yield of **10** was ∼91%, but the conversion of **9** was much lower (70%).

Unlike pyrroles **2** and **7**, the synthesis of pyrrole **9** occurs via five-membered ring-closure promoted by *t*-BuOK/DMSO, followed by methylation of the intermediate pyrrolylsulfide **11** (Scheme 4).[13a,18]

It was also shown, that tetrasubstituted pyrrole **12**,[19] derived from isothiocyanate **1**, 3-methoxy-1-(methylsulfanyl)-1-propyne and MeI, underwent sequential cleavage of the *N*-[2-(vinyloxy)ethyl] moiety using *t*-BuOK/DMSO to give 5-methoxy-2,3-

bis(methylsulfanyl)-*N*-vinylpyrrole (**13**) in 96% yield, the conversion of **12** being 97% (Scheme 5).

The most probable mechanism for the transformation of *N*-[2-(vinyloxy)ethyl]pyrroles into *N*-vinylpyrroles involves deprotonation of the *N*-[2-(vinyloxy)ethyl] moiety followed by β-elimination of vinyl alcohol as depicted in Scheme 6.

Previously, a similar superbase-catalyzed elimination of vinyl alcohols from vinyl ethers containing heteroatom (oxygen) at the β-position of a vinyloxy group was reported for the vinyl ether of glycerol[20] and the divinyl ether of ethylene glycol.[21]

The structures of synthesized pyrroles were assigned unambiguously by IR and ^{1}H, ^{13}C, HSQC and HMBC NMR experiments.

In conclusion, we have developed a novel and general synthetic approach to the efficient preparation of *N*-vinylpyrroles from 2-(vinyloxy)ethyl isothiocyanate (**1**), lithiated allenes or alkynes and alkyl halides in two preparative steps via a one-pot synthesis of *N*-[2-(vinyloxy)ethyl]pyrroles and elimination of the vinylic alcohol from *N*-[2-(vinyloxy)ethyl] moiety in the presence of *t*-BuOK/DMSO. Moreover, this approach to *N*-vinylpyrroles is applicable to other *N*-[2-(vinyloxy)ethyl]pyrroles. We are continuing to study the scope and limitation of this approach to *N*-vinylpyrroles.

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Supplementary data

Supplementary data (experimental section, characterization of all compounds) associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2010.07.179.

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