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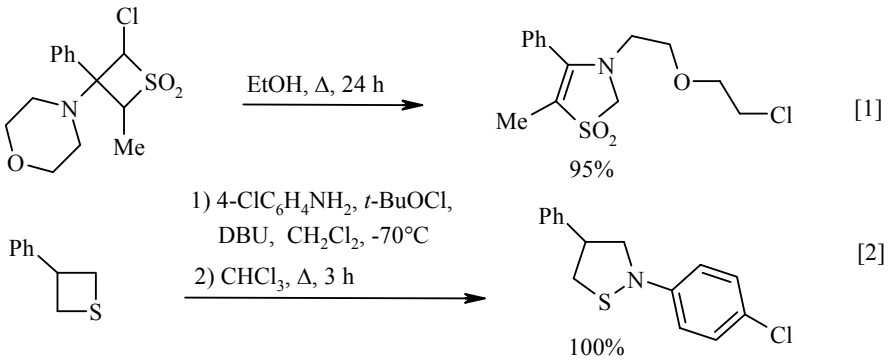
METHODS FOR THE SYNTHESIS OF DERIVATIVES OF 3-AMINOTHIETANE (REVIEW)

A. N. Butkevich, V. V. Sokolov, A. A. Tomashevskii, and A. A. Potekhin*

Methods for the production of derivatives of 3-aminothietane and 3-aminothietan-2-one are reviewed.

Keywords: 3-aminothietanes, thietane 1,1-dioxides, thietanylation of heterocycles, thiirane–thietane rearrangement, [2+2]-cycloaddition.

Derivatives of thietane have so far remained little-investigated compounds both in the sense of methods of preparation and with respect to synthetic applications. This is due to the almost complete lack of such compounds among natural compounds and to the insufficient attention paid to them on the part of chemists because of the poor synthetic accessibility of functionally substituted thietanes and the lack of information on useful biological activity directly associated with the presence of the small sulfur-containing heterocycle. Nevertheless, the synthetic potential of thietane derivatives is extremely significant, as can be illustrated by effectives examples of the construction of difficultly obtainable heterocyclic systems from them.



The main reason for the limited use of thietane derivatives in multistage synthesis is the low yields obtained in reactions leading to the formation of the four-membered heterocycle. In this connection research into new effective methods for the production of derivatives of 3-aminothietane with various substituents at the nitrogen atom is needed.

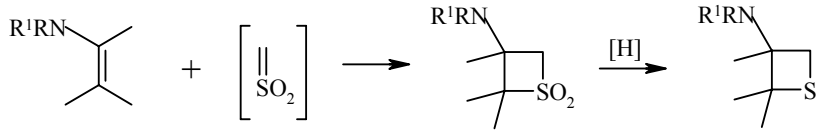
Dedicated to Prof. Ivars Kalvinsh on the occasion of his 60th birthday.

*Deceased.

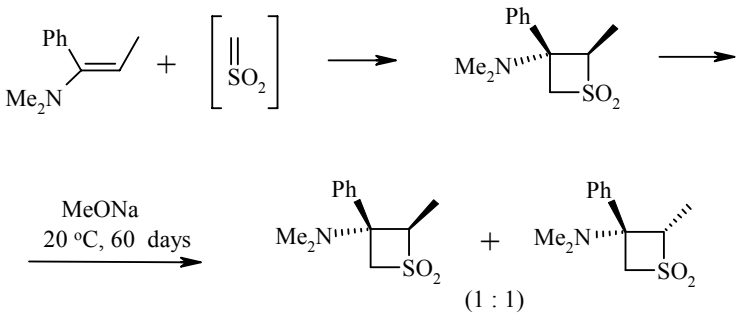
St. Petersburg State University, Chemical Faculty, St. Petersburg, Russia; e-mail: vsokolo@mail.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 5, pp. 655-682, May, 2007. Original article submitted March 20, 2006.

1. [2+2] Cycloaddition

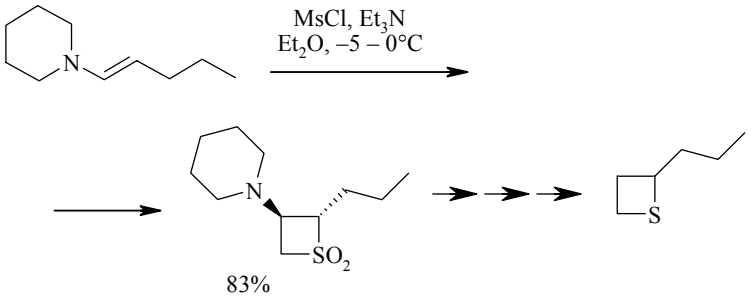
Derivatives of 3-aminothietane were first prepared by a method based on the [2+2] cycloaddition of sulfene $CH_2=SO_2$ or its analogs to enamines followed by reduction of the products – 3-aminothietane 1,1-dioxides. Despite the numerous shortcomings and limitations, most of the 3-aminothietanes described in the literature were synthesized by this method. The reaction takes place regioselectively and can be represented by the following general scheme:



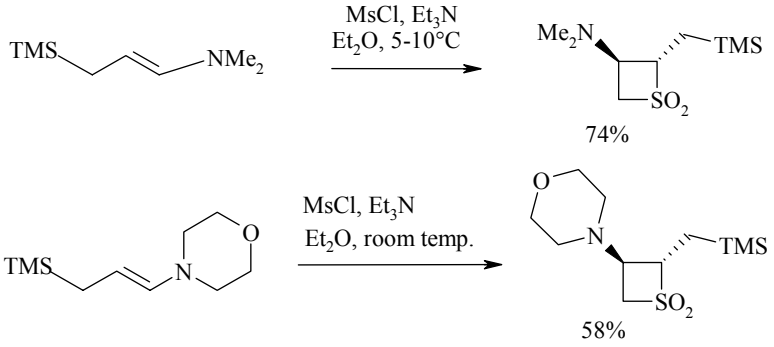
The stereoselectivity of the process (retention of the configuration, see [3] and the literature cited therein) indicates that the addition of the sulfene to the enamine is a concerted [π^2s + π^2a]-cycloaddition process, taking place under kinetic control.



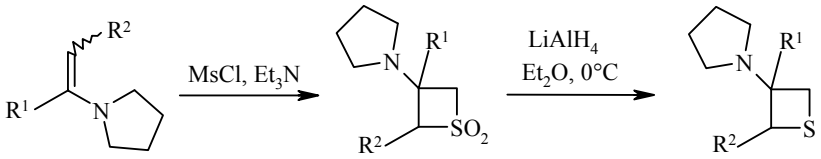
The usual method for the generation of sulfenes is by the action of a base (most often triethylamine) on alkanesulfonyl chlorides in aprotic solvents, often with cooling. The methanesulfonyl chloride usually employed for this purpose gives the simplest sulfene during dehydrohalogenation. The yields in the addition reaction (usually from moderate to high) depend on the substituent at the double bond of the enamine. The addition of sulfene to 1-(pent-1-enyl)piperidine was suggested as first stage in the synthesis of 2-propylthietane [4].



3-Amino-2-(trimethylsilylmethyl)thietane 1,1-dioxides were obtained similarly [5].

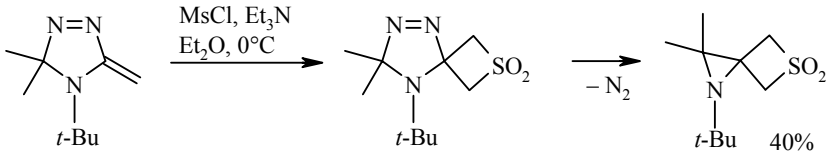


The reduction of the 3-aminothietane 1,1-dioxides to the corresponding 3-aminothietanes is realized by the action of an excess of lithium aluminum hydride (e.g., [6]).

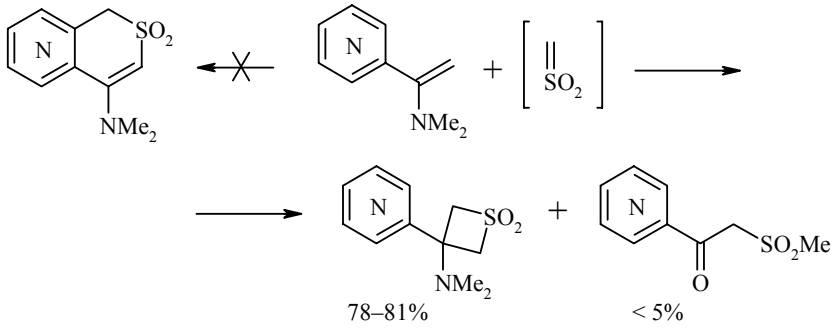


R^1–R^2 = $(CH_2)_4$; R^1–R^2 = $(CH_2)_5$; R^1 = Et, R^2 = Me; R^1 = *n*-Pr, R^2 = Et

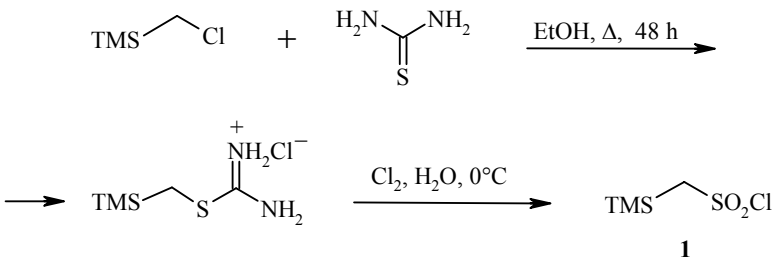
The use of the enamine, in which the double bond was in conjugation with the azo group, led to the production of a spiroaziridine instead of the expected triazoline as the only product. The intermediate compound was characterized by spectral methods but was not isolated since ejection of the nitrogen molecule takes place at temperatures lower than room temperature [7].



In the reaction of isomeric (1-dimethylaminovinyl)pyridines with sulfene [8] the [2+2] cycloaddition products 3-aminothietane 1,1-dioxides and not expected the [4+2] cycloaddition products are formed with yields in the order of 80%. The parallel formation of a small amount of acyclic sulfone was observed.



Trimethylsilylmethanesulfonyl chloride (**1**) [9], which is easily obtained from trimethyl-(chloromethyl)silane with a total yield of 58%, was proposed as another source of sulfene.



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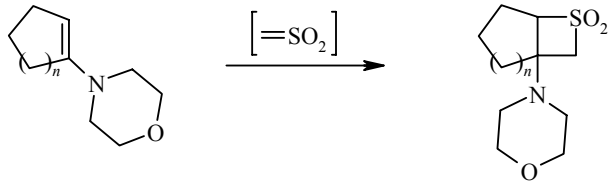
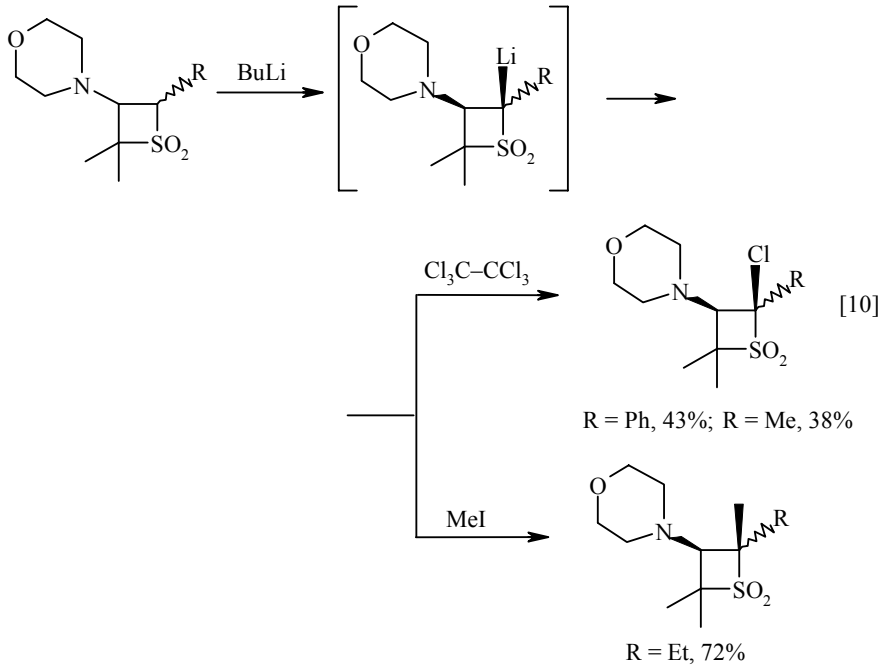
The action of cesium fluoride in acetonitrile leads to desilylation of compound **1** with the formation of sulfene (and trimethylfluorosilane as side product). Generation of sulfene by this method gives higher yields of the [2+2] cycloaddition products than the standard procedure (MsCl + Et_3N) [9].

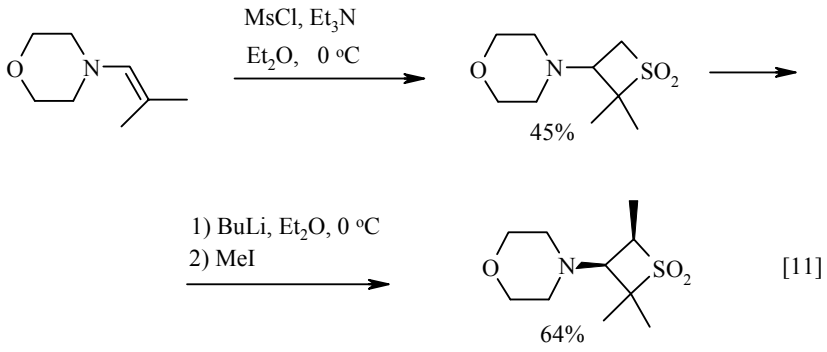


The dependence of the yield of the products, %, on the method of generation of the sulfene:

n	MsCl + Et_3N	**1** + CsF
1	77	81
2	54	65



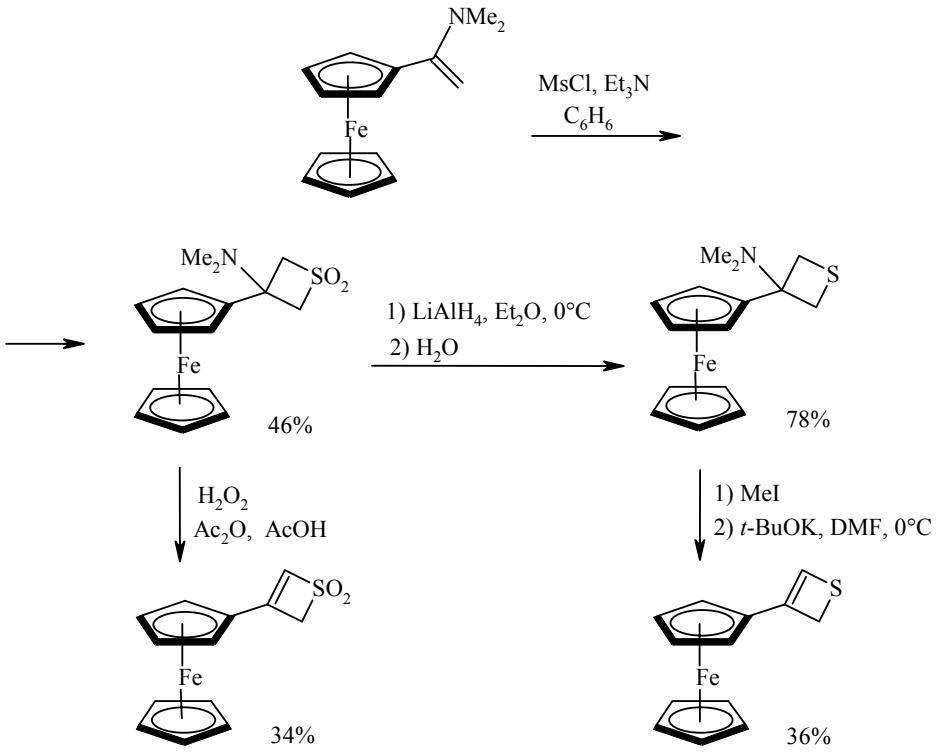
[10]



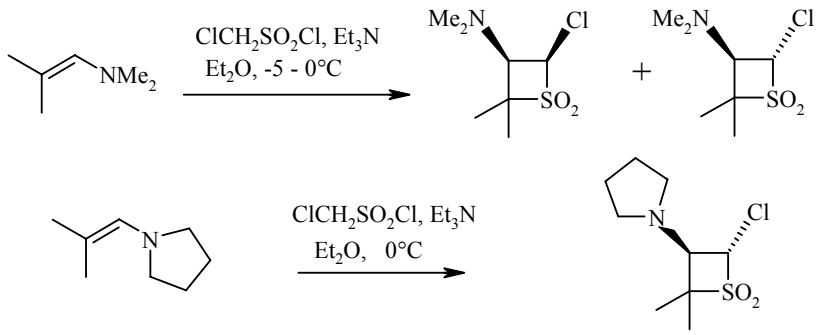
[11]

The cycloaddition products (3-aminothietane 1,1-dioxides) can be submitted to further functionalization without opening the heterocycle. The substituent at position 2 of the thietane ring can be introduced by metallation with *n*-butyllithium, which takes place stereoselectively. This is due to coordination of the lithium atom in the metallated sulfone at the *cis* position to the amine nitrogen atom.

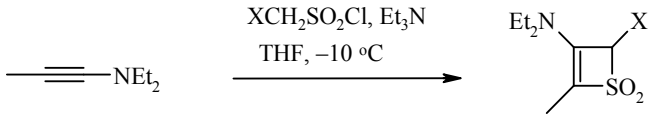
An interesting example of [2+2] cycloaddition of unsubstituted sulfene to enamines to construct a difficultly obtainable heterocyclic system is the synthesis of 3-ferrocenyl-3H-thiete and its 1,1-dioxide [12].



The authors of the patent [13] mention the possibility of using chloromethane- or bromomethanesulfonyl chloride as initial sulfonyl chloride for generation of the sulfene. The products here are 2-chloro- or 2-bromo-3-(dialkylamino)thietane 1,1-dioxides respectively (the yields are not given).

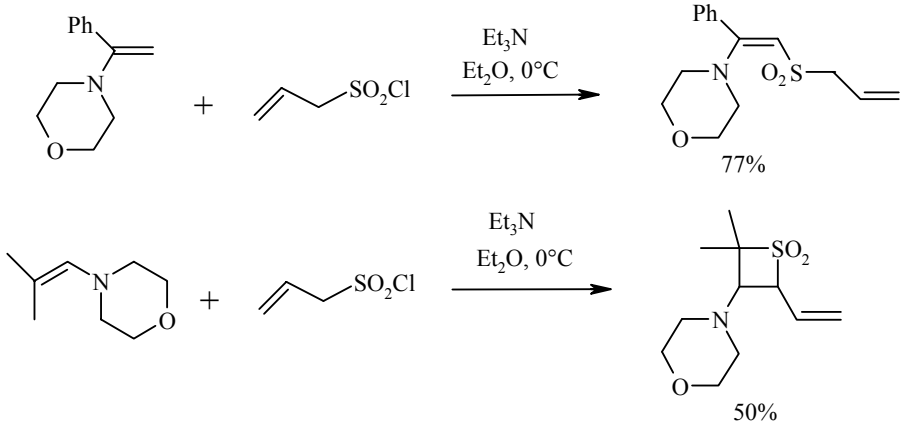


The use of ynamines instead of enamines gave 2-halogeno-3-dialkylamino-2H-thiete 1,1-dioxides.

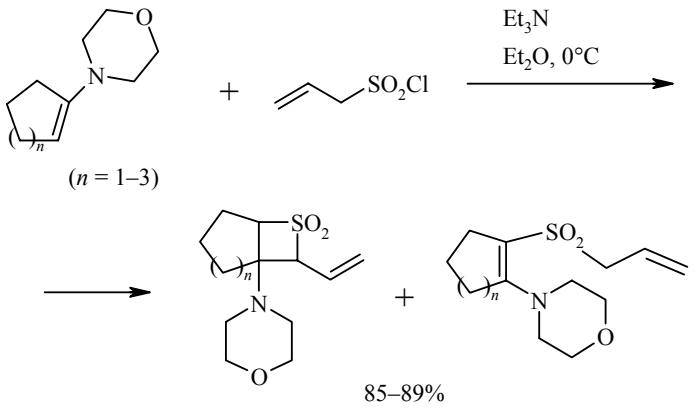


X = Cl, Br

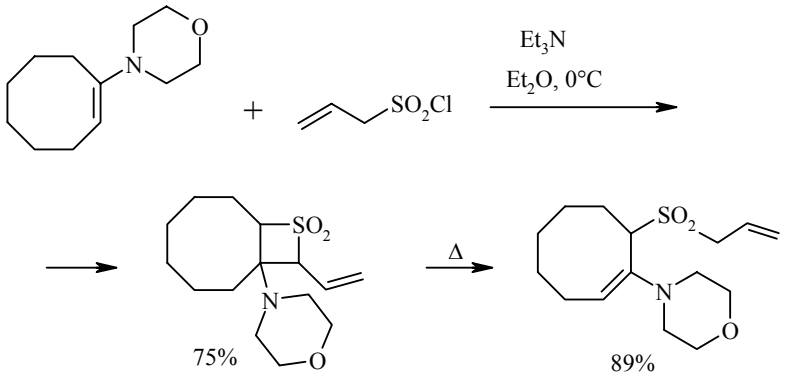
On the contrary, the transition to conjugated vinyl- or ethynylsulfenes, produced from allyl- or propargylsulfonyl chloride respectively, substantially changes the result of the reaction. Here, the expected 3-aminothietane 1,1-dioxides are often not formed at all, or their formation is complicated by a series of side processes. The addition of vinylsulfene to enamines with various structures was investigated in detail in [14]. In these cases acyclic allylsulfones – products from attack of the vinylsulfene as electrophile at the α-carbon atom of the enamine (a reaction similar to the C-alkylation or C-acylation of enamines without subsequent hydrolysis)– are formed in addition to the [2+2] cycloaddition products. The preferential formation of one or the other product depends on the structure of the enamine; it was shown that they are formed independently. The only product from the reaction of vinylsulfene with 4-(1-phenylvinyl)morpholine is an acyclic sulfone, whereas 4-(2-methyl-1-propenyl)morpholine only forms a 3-aminothietane derivative.



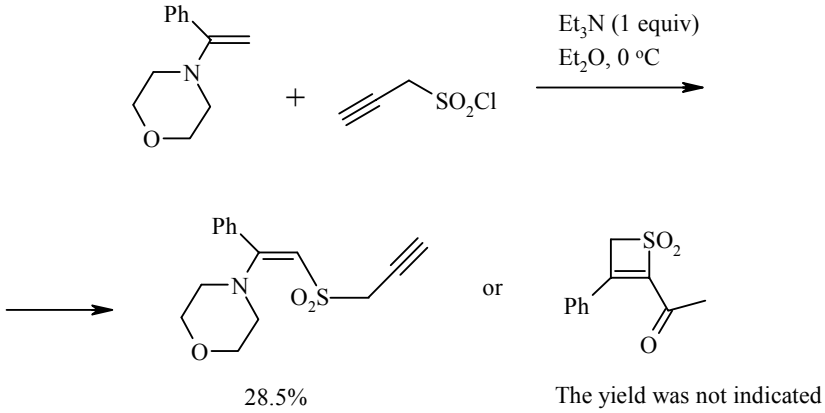
The last compound is thermally stable and does not undergo ring opening either under the reaction conditions or when heated with a base. Enamines obtained from cyclic ketones give a mixture of the two possible products.



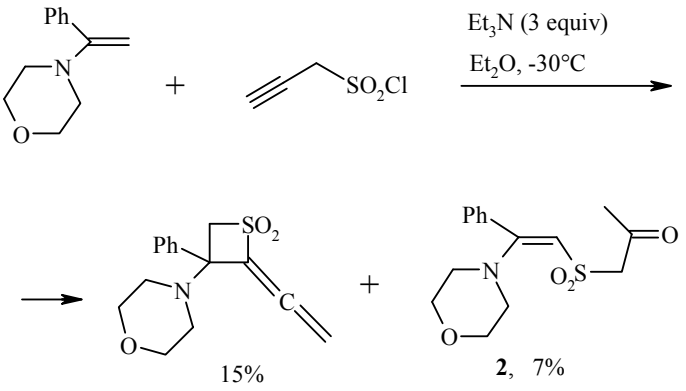
The only exception was 1-(morpholino)cyclooctene, which with the vinylsulfene first forms a [2+2] cycloaddition product. During recrystallization from ethanol this is transformed with a high yield into an exocyclic sulfone. (Migration of the double bond also occurs.)



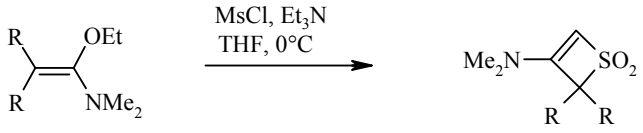
3-Aminothietane is also not formed when propargylsulfonyl chloride is brought into reaction with 4-(1-phenylvinyl)morpholine under the same conditions [15]. Either an acyclic propargyl sulfone (an analog of the product from the reaction of this enamine with the allylsulfonyl chloride–triethylamine system) or 2-acetyl-3-phenyl-4H-thiete 1,1-dioxide (a product from more complicated transformations) was isolated, depending on the treatment of the reaction mixture.



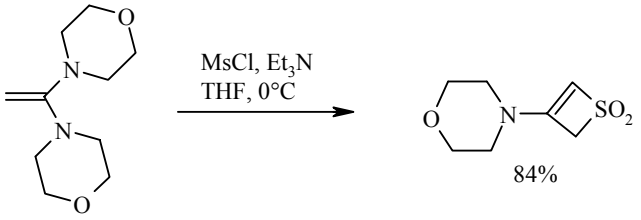
With the use of 3 equiv of triethylamine and stronger cooling it was possible to obtain a small yield of a 3-aminothietane derivative containing a vinylene substituent instead of the expected ethynyl at position 2 and also the sulfone **2** as side product.



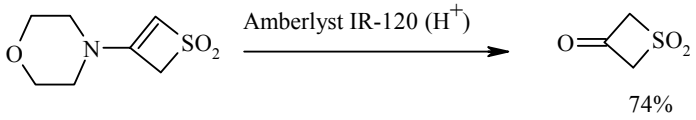
The formation of compound **2** was observed in the first experiment.
With sulfene enediamines and 1-alkoxyenamines form 3-dialkylaminothiete 1,1-dioxides [16].



R = H, 34%; R = Me, 62%



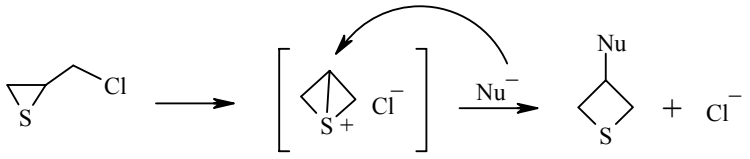
Acid hydrolysis of the last compound was used for the synthesis of 3-oxothietane 1,1-dioxide.



2. Thiirane–Thietane Rearrangement

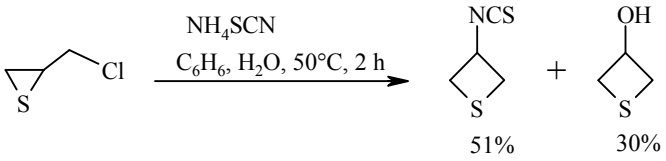
The thiirane–thietane rearrangement occurs during the reaction of (α-halogenoalkyl)thiiranes with certain nucleophiles that secure effective solvation of the cations (water, lower alcohols). The general scheme of the rearrangement can be represented as follows [17].

The intermediately formed 1-thioniacyclobutane is attacked by the nucleophile at the carbon atom at position 3 with opening of the diagonal bond. Conversion of the nucleophile NuH into the active anionic form (Nu^-) requires the presence of at least one equivalent of the base.

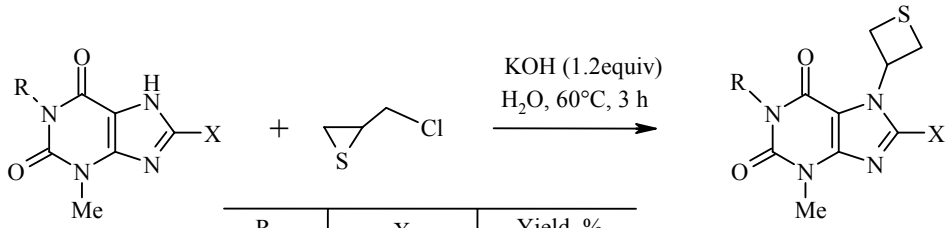


The most often used substrates in this reaction are phenols [18, 19] and carboxylic acids [20], which give 3-aryloxy- and 3-acyloxythietanes respectively as products. Thietan-3-ol can be obtained from (chloromethyl)thiirane by the action of sodium carbonate in aqueous ethanol [21].

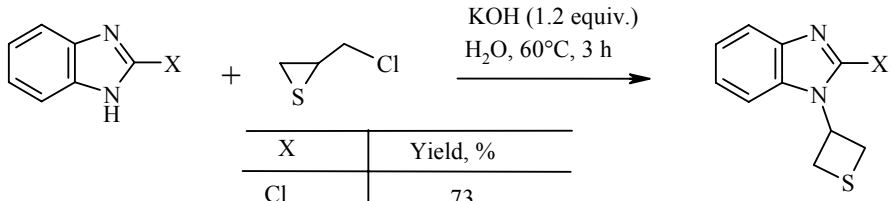
Although it was claimed in [22] that the reaction of (chloromethyl)thiirane with ammonium thiocyanate leads to the formation of thietan-3-yl isothiocyanate we were unable to reproduce these results.



Alkylation at the nitrogen atom by (chloromethyl)thiirane in an aqueous medium, which takes place with a thiirane-thietane rearrangement, is known for two types of heterocyclic systems–xanthines and benzimidazoles [23].

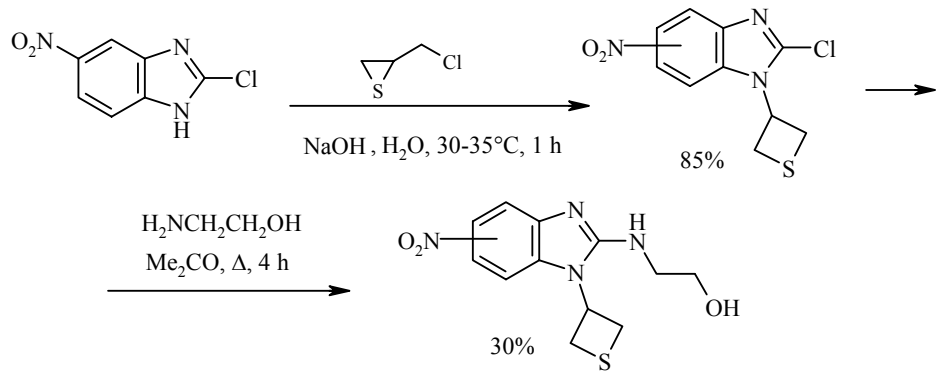


R	X	Yield, %
Me	Cl	31
Me	Br	44
Me	BnNH	36
Me	PhNH	33
H	Cl	28

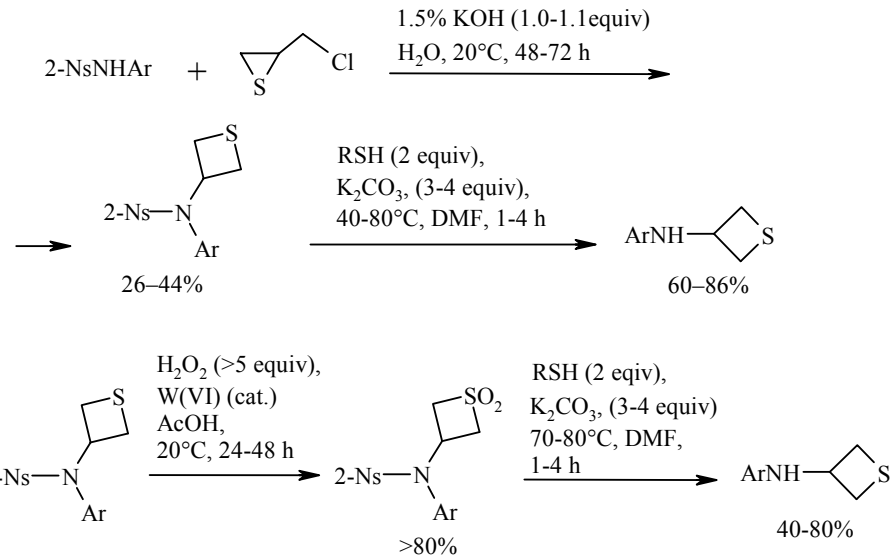


X	Yield, %
Cl	73
SO_2Me	41
SO_2Et	39

A derivative of 1-(3-thietanyl)benzimidazole in the form of a mixture of isomers was patented as a compound exhibiting anti-inflammatory activity [24].



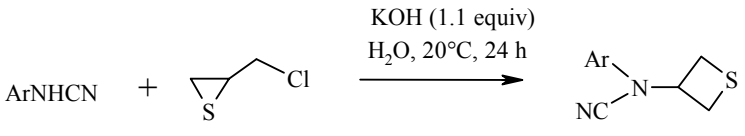
Sulfamides also undergo similar alkylation with a thiirane–thietane rearrangement, and in the case of N-arylsulfamides the yields of the products–N-(thietan-3-yl)sulfamides–amount to 50-60%. 3-(Arylamino)-thietanes and 3-(arylamino)thietane 1,1-dioxides were obtained by the alkylation of 2-nitrobenzenesulfanilides with (chloromethyl)thiirane followed by removal of the 2-nitrobenzenesulfonyl group [25].



(2-Ns = 2-nitrobenzenesulfonyl); Ar = Ph, 4-$MeOC_6H_4$; 2-*i*-PrC_6H_4, 4-ClC_6H_4; 1-naphthyl, 3-$O_2NC_6H_4$; RSH = PhSH, $HSCH_2CO_2H$, 2-$HSC_6H_4CO_2H$

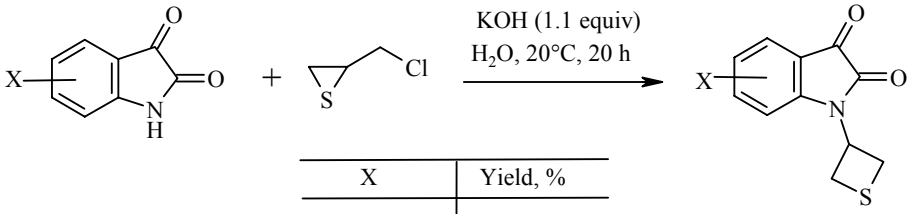
The method cannot be used for anilines containing a strong electron-withdrawing substituent (e.g., Ar = 4-$O_2NC_6H_4$) conjugated with the reaction center on account of the extremely low yields at the alkylation stage (<2%).

The reaction of arylcyanamides with (chloromethyl)thiirane in dilute aqueous alkali leads to the formation of N-aryl-N-(thietan-3-yl)cyanamides with yields of 40-70% [26].



Ar	Yield, %
Ph	72
4-$MeOC_6H_4$	59
1-Naphtyl	38

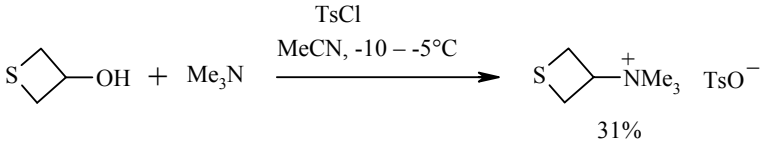
During alkylation with (chloromethyl)- or (bromomethyl)thiirane in a dilute aqueous solution of alkali isatins unsubstituted at the nitrogen atom (with the exception of 5-nitroisatin, which does not enter into the reaction) form N-(thietan-3-yl)isatins with low yields [26].



X	Yield, %
H	24
5-Br	22
5-MeO	34
5,6-(OCH_2O)	24

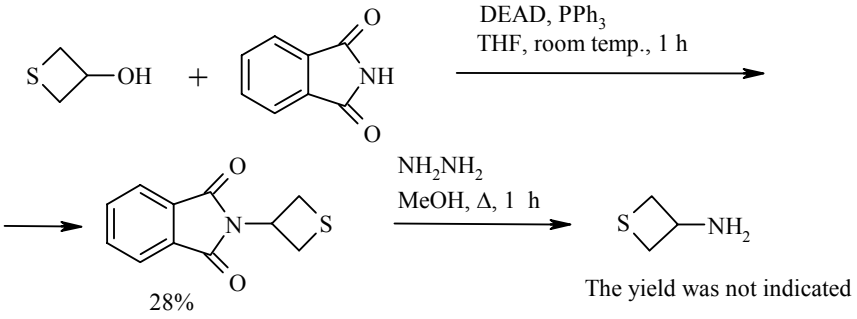
3. Nucleophilic Substitution at the C-3 Atom of Thietane

Nucleophilic substitution is unknown for thietanes with a sulfide atom containing a good leaving group at position 3. For instance, 3-thietanyl tosylate was not described and was not isolated during an attempt to synthesize it by the standard procedure. It is known, however, that a trimethyl(thietan-3-yl)ammonium salt is formed with a moderate yield in the reaction of a mixture of 3-thietanol and an excess of triethylamine with tosyl chloride under anhydrous conditions at reduced temperature [6].

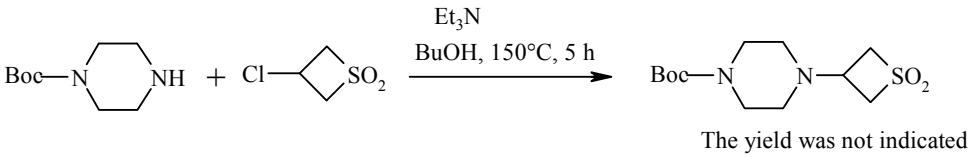


The reaction clearly takes place through the intermediate formation of 3-thietanyl tosylate, but no attempts were made to establish the type of substitution mechanism (S_N1 or S_N2). The instability of thietanes with a leaving group at position 3 may be due to anchimeric assistance afforded by the sulfur atom in the transannular position. A consequence of this is the exceptionally easy reaction of these compounds with external nucleophiles, in this case with triethylamine. Conversely, thietanes containing poor leaving groups, such as a chlorine atom, at position 3 are stable.

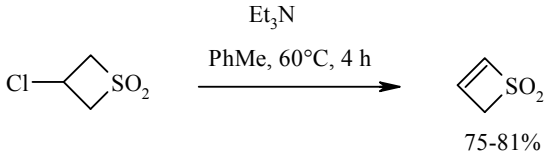
The authors of the patent [27] report the possibility of synthesizing unsubstituted 3-aminothietane by a two-stage scheme, the first stage of which is a Mitsonobu reaction with thietan-3-ol and phthalimide.



The reaction of 3-substituted thietane 1,1-dioxides (the substituent is the leaving group) with amines is well known for dialkylamines and also formally represents nucleophilic substitution, e.g., see [28].



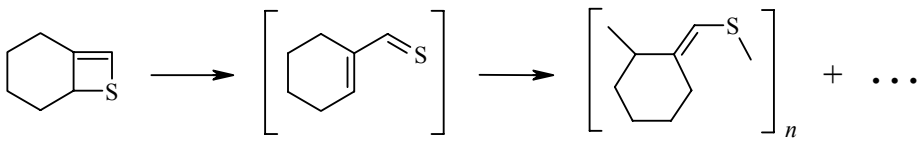
In this case, however, elimination–addition must be considered a possible and more likely alternative to the S_N2 mechanism, since the dehydrohalogenation of 3-chlorothietane 1,1-dioxide takes place readily and with high yields under the action of tertiary amines [29, 30].



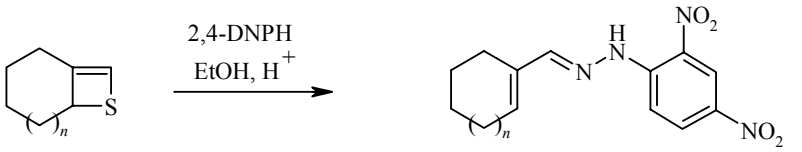
Unlike the corresponding sulfide, 1,1-dioxothietan-3-yl tosylate is stable [30].

4. Michael Addition

The use of thietes as Michael acceptors with enamines in the role of nucleophiles has not been described. This is due to the extremely low stability of thietes and the ease of their involvement in various side reactions. These mainly include electrocyclic opening of the ring with the formation of mixtures of low-molecular compounds and polymers containing vinyl sulfide fragments [6].

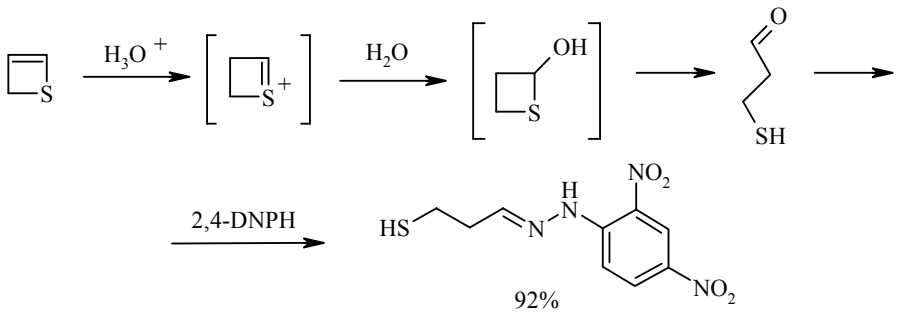


Unsubstituted thiete decomposes fully at room temperature in less than 24 h, and some of its analogs are even less stable. Thietes react with 2,4-dinitrophenylhydrazine in the presence of acids, giving not the products from addition at the double bond but hydrazones, formed from the corresponding unsaturated thioaldehydes (n = 1, 2) [6].

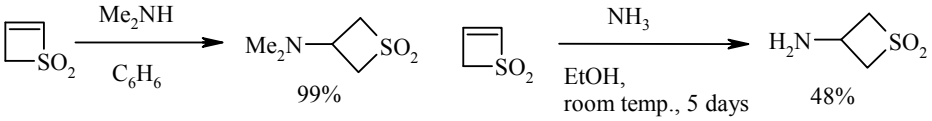


(n = 1, 2) The yields were not indicated

Unsubstituted thiete here gives a high yield of a derivative of 3-mercaptopropionaldehyde [6].

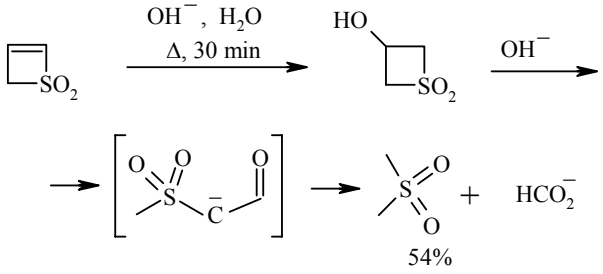


On the other hand, thiete 1,1-dioxides add secondary amines readily and with high yields and, not so readily, ammonia (reaction with primary amines was not described) [31].

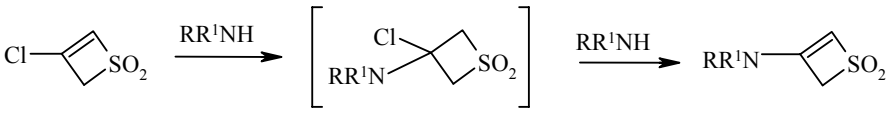


However, the reduction of the 3-aminothietane 1,1-dioxides obtained in this way with lithium aluminum hydride is an unreliable method; the yield at this stage probably depends strongly on the nature of the substituents in the thietane ring and for the various derivatives lies in the range of 23-85% [6, 31].

Thiete 1,1-dioxide adds H_2S, ethanol, and thiophenol similarly in the presence of bases, but the reaction with a solution of barium hydroxide is accompanied by decomposition [31].



The reaction of secondary amines with 3-chlorothiete 1,1-dioxide is Michael addition followed by elimination of a molecule of HCl [32]. The reaction gives yields from moderate to high.

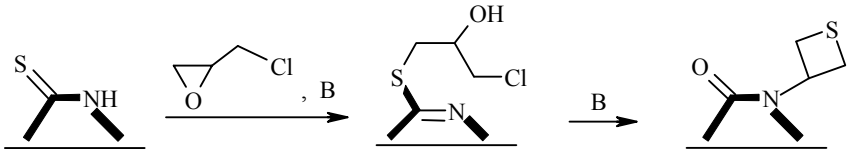


R	R^1	Yield, %
Me	Me	66
Et	Et	95
$-(CH_2)_5-$		67
$-(CH_2)_2O(CH_2)_2-$		59
Me	Ph	56

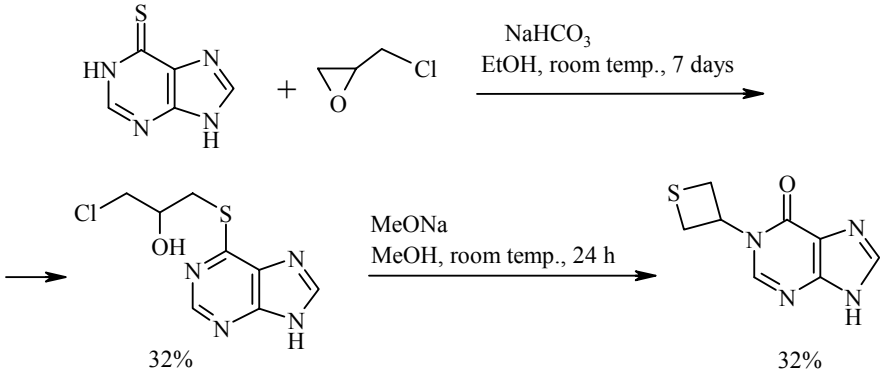
Imidazole (in the presence of triethylamine), alcoholates, and thiolates can be used as nucleophiles in this reaction.

5. Rearrangements of Compounds Containing a Thioamide Fragment

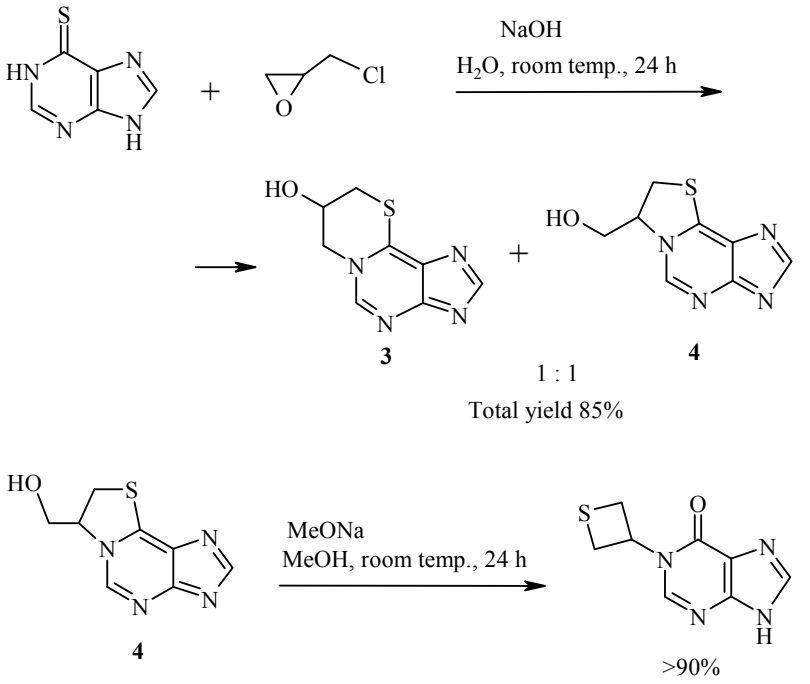
During the alkylation of certain thiolactams with epichlorohydrin in the presence of a base the products from opening of the oxirane ring are formed. Under the action of stronger bases they then rearrange to the corresponding N-(thietan-3-yl)lactams. The general scheme of the rearrangement can be represented in the following way (the thioamide fragment must enter into the composition of the heterocyclic system):



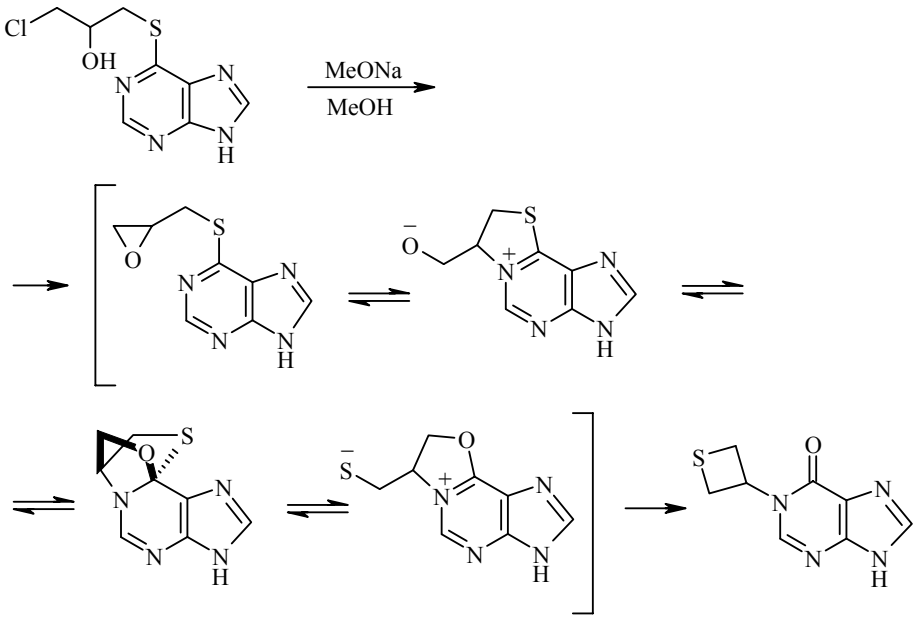
This reaction was discovered during the alkylation of 6-mercaptopurine [33].



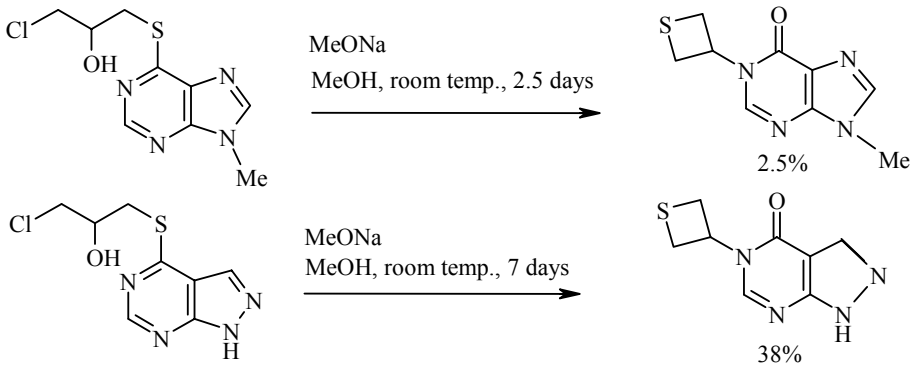
Further investigations showed that two compounds are formed during the alkylation of 6-mercaptopurine in the presence of aqueous alkali, and only one of them is converted into the desired product when treated with sodium methoxide [34]. This can explain the low yield of the thietane derivative.



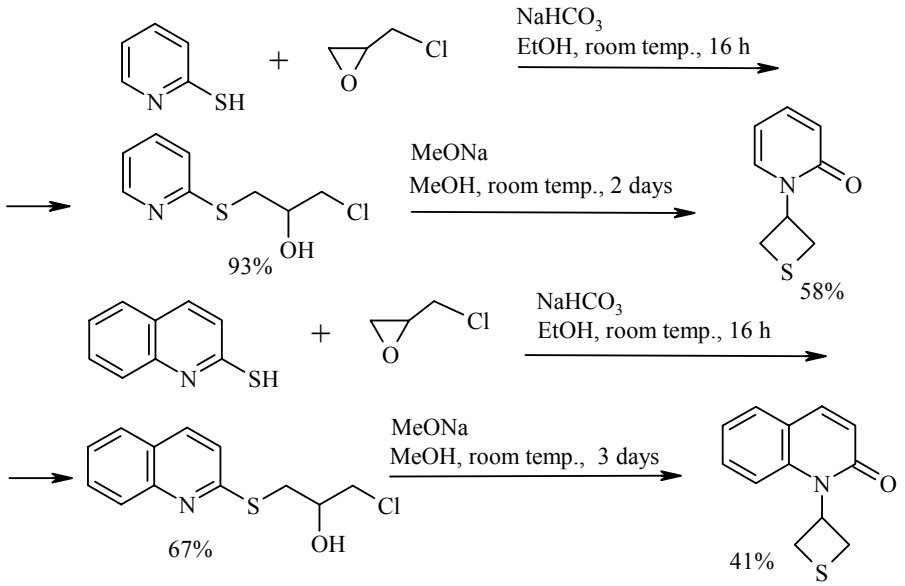
The mechanism of formation of compound **4** is not entirely clear; it is presumably formed during opening of the intermediately formed oxirane ring against the Krasuskii rule. The authors [33] propose the following possible transformation mechanism.



The introduction of a methyl substituent at position 9 in the 6-mercaptopurine molecule reduces the nucleophilicity of the N-1 atom of the purine system and as a result greatly reduces the yield of the thietane derivative. On the other hand, the transition to the pyrazolo[3,4-*b*]pyrimidine system has practically no effect on the yield of the desired compound [34].

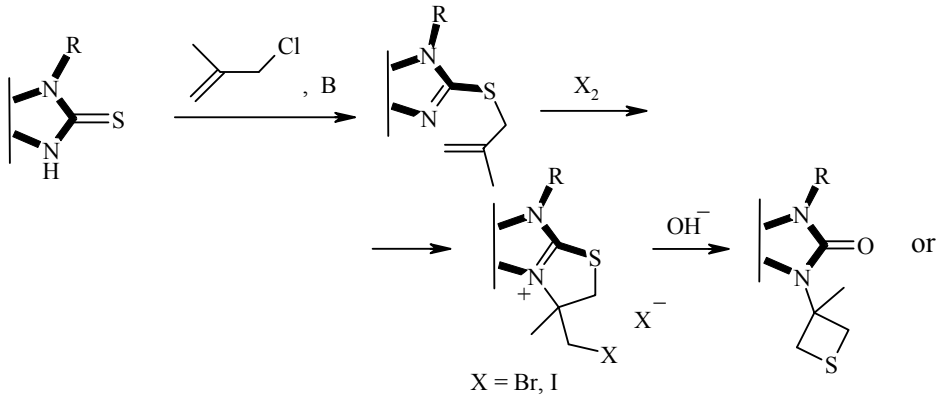


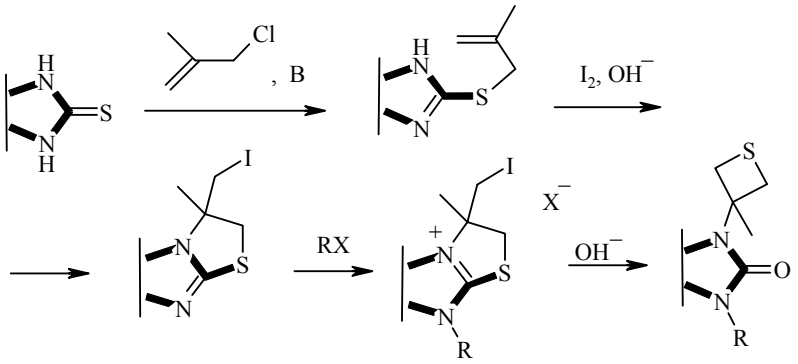
1-(Thietan-3-yl)-2(1H)-pyridone and 1-(thietan-3-yl)-2(1H)-quinolone are formed similarly and with higher yields [34].



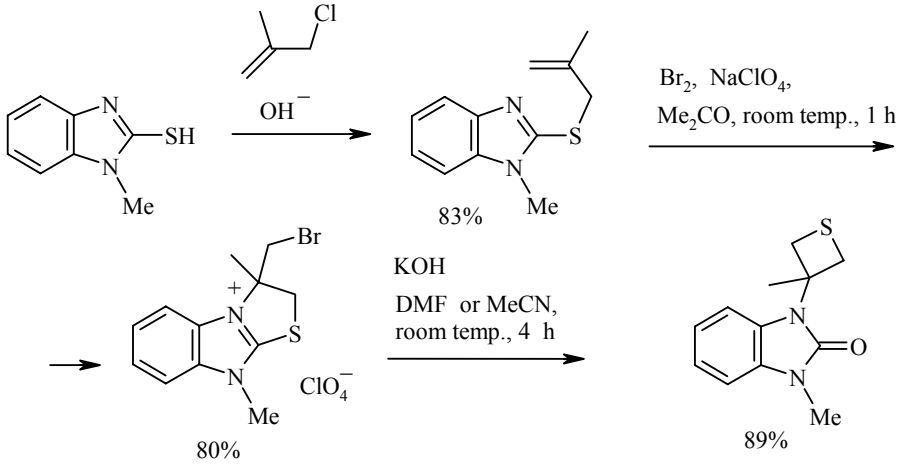
Attempts to use 2-mercaptobenzoxazole, 2-mercaptopyrimidine, and 2-mercaptoimidazole as substrates did not lead to derivatives of thietane, while 4-mercaptopyrimidine gave pyrimidin-4(3H)-one as the main product (44%) and only 5% of the target compound. A series of experiments on the use of acyclic thioamides in this reaction also ended in failure; unfortunately, the authors did not give the details of these experiments [34].

The related rearrangement of 2-methallylsulfamoylazoles, taking place during halogenation and subsequent treatment with a base, was reported [35]. The general scheme of the transformation can be represented as follows:

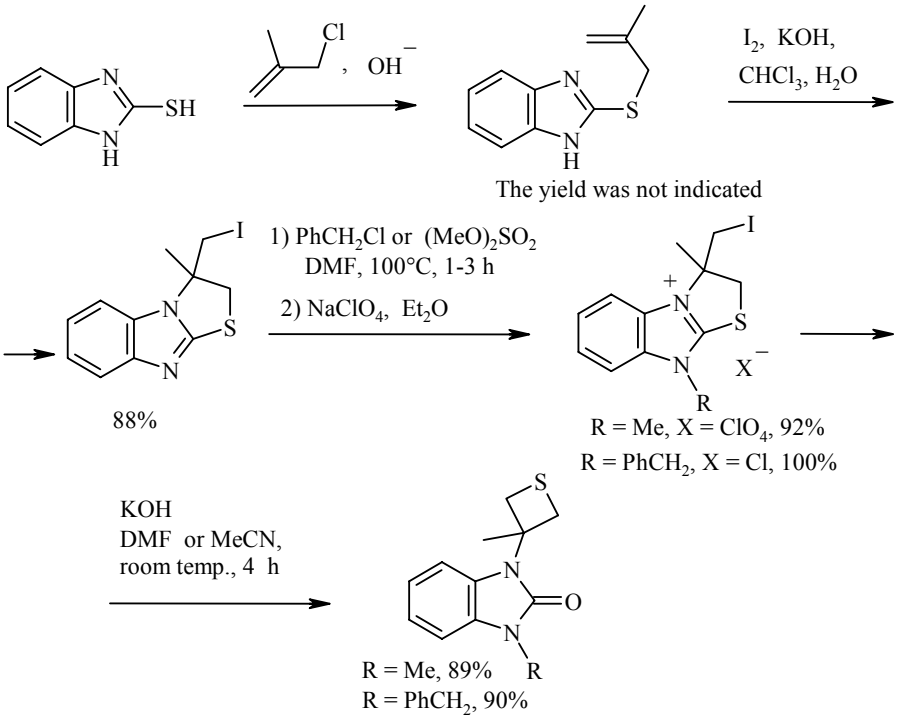




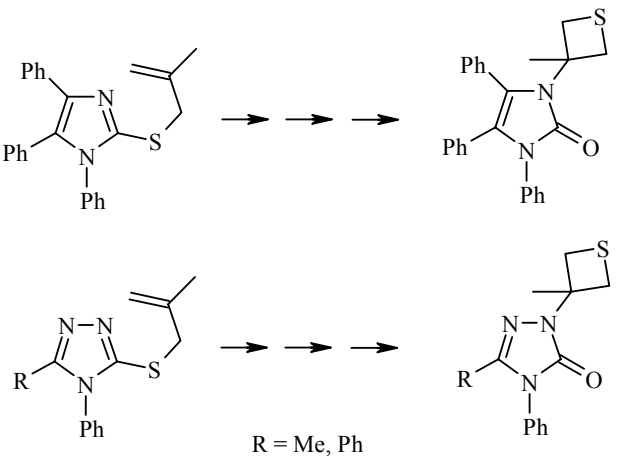
The first variant can be illustrated by the following example:



In the other variant 2,3-dihydroimidazo[2,1-*b*][1,3]thiazoles were brought into alkylation at the nitrogen atom; in this case the use of bromine instead of iodine leads to a large reduction in the yield or to the formation of other products.

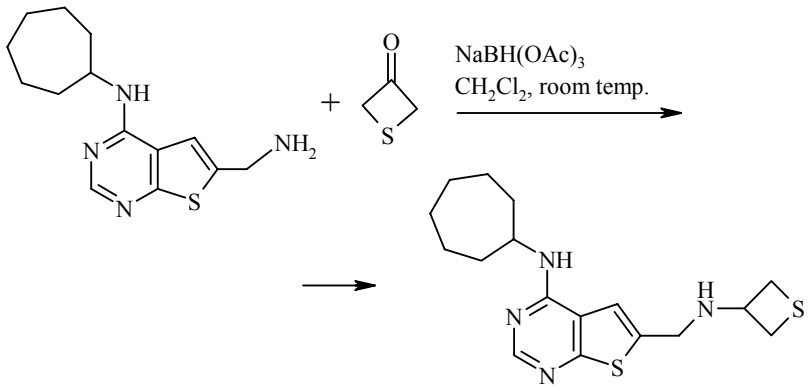


N-(3-Methylthietan-3-yl)-substituted imidazoles and 1,2,4-triazoles can be obtained similarly (and also with high yields) [35].

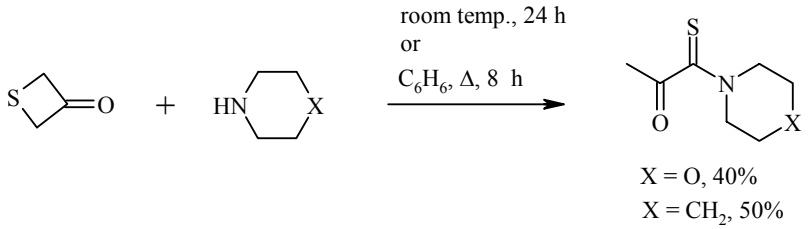


6. Reductive Amination

A single successful synthesis of a derivative of 3-(alkylamino)thietane by reductive amination of thietan-3-one, produced *in situ* by oxidation of thietan-3-ol with pyridinium chlorochromate, is known [36]. The yield was not given.

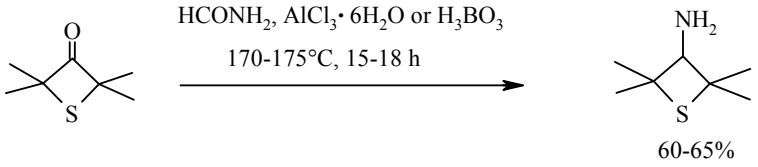


Secondary amines do not form enamines in reaction with thietan-3-one. Instead the thioamides of pyruvic acid are formed with yields in the order of 40-50% [37].



Mercaptoacetone was detected as side product of this reaction.

The authors of the patent [38] report the successful reductive amination of sterically hindered 2,2,4,4-tetramethylthietan-3-one by the action of formamide in the presence of an acidic catalyst.

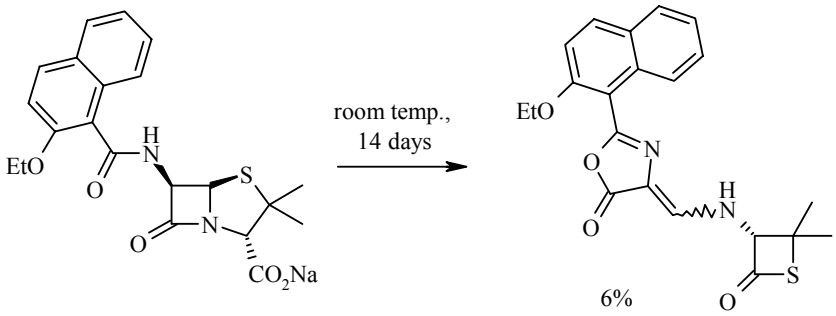


The absence of other data on reactions of thietan-3-one or its analogs with alkylamines, arylamines, or ammonia leads to the conclusion that the possibility of synthesizing derivatives of 3-aminothietane with various sets of substituents by reductive amination is extremely doubtful.

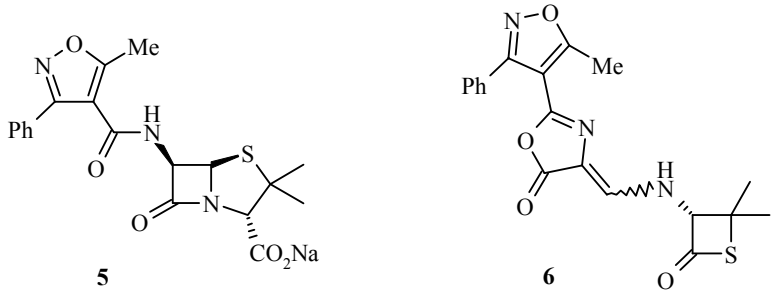
7. Derivatives of 3-Aminothietan-2-one

Interest in compounds of this group was raised by their discovery among the products from the decomposition of penicillins and also by the possibility of using them as acylating reagents for the insertion of a penicillamine fragment into a molecule.

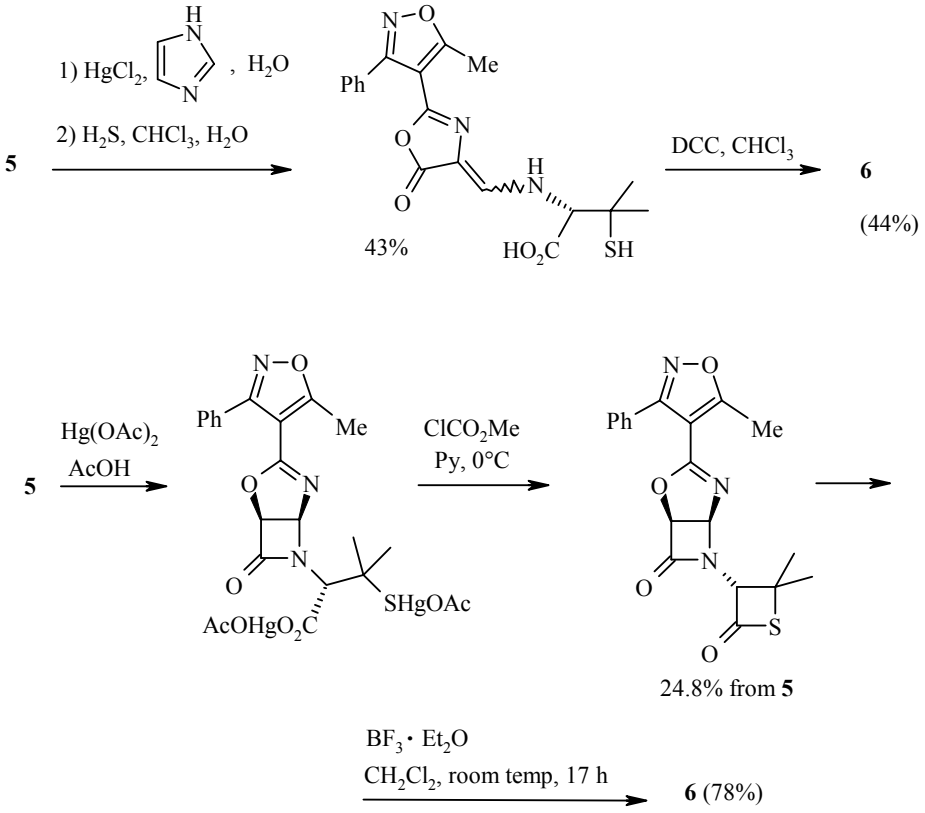
Storage of an aqueous solution of the sodium salt of the antibiotic naphcillin led to the separation of a precipitate, from which a derivative of 3-amino-4,4-dimethylthietan-2-one was isolated [39].



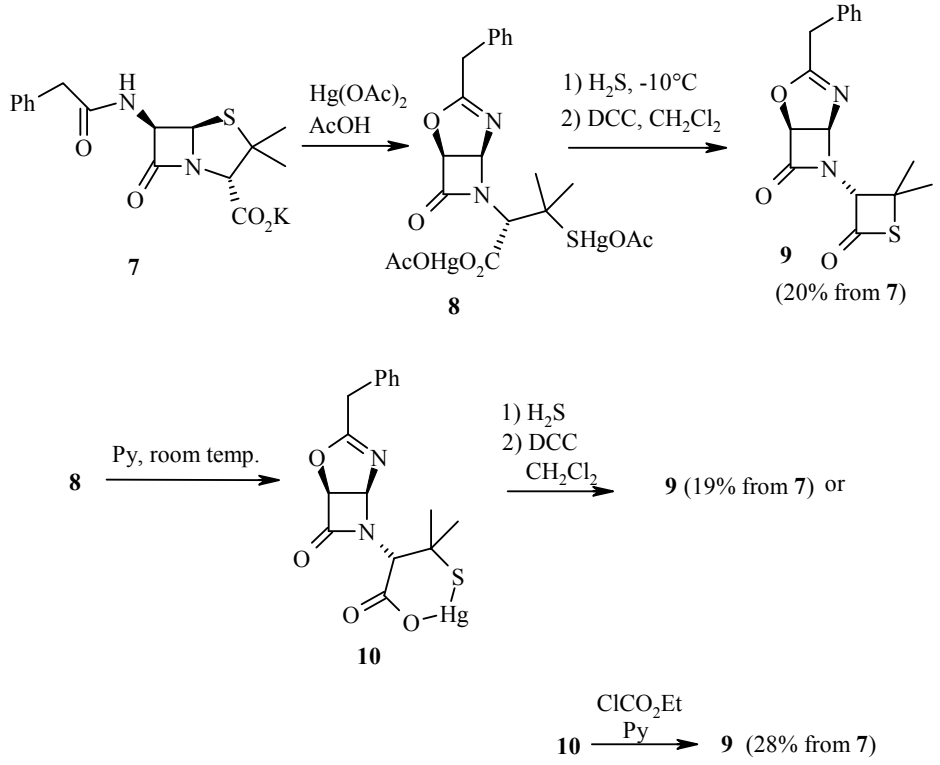
A very small amount (0.006%) of compound **6**, contained in the product as impurity, was obtained from the sodium salt of oxacillin (**5**).



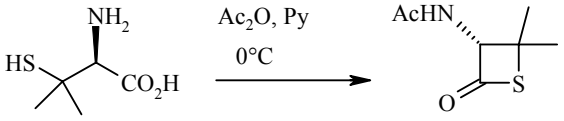
The structure of **6** was confirmed by the direction of its synthesis from compound **5** [39].



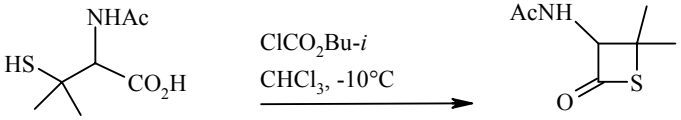
Similar derivatives were also obtained from benzylpenicillin (**7**) [40].



D-Penicillamine [41] and N-acetyl-DL-penicillamine [42] undergo cyclization to derivatives of 3-aminothietan-2-one under the influence of various dehydrating agents. In the reaction with optically active amino acids the configuration is retained.

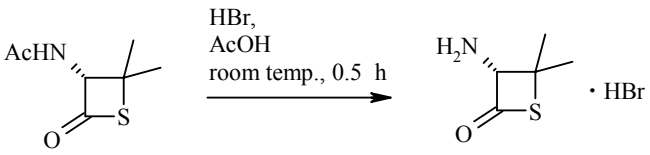


31–35%



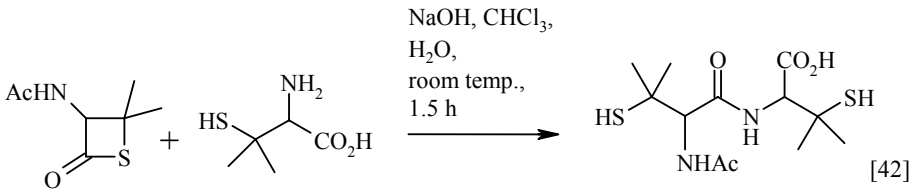
14%

The acetyl group can be removed by acid hydrolysis, which takes place without ring opening or racemization [41].



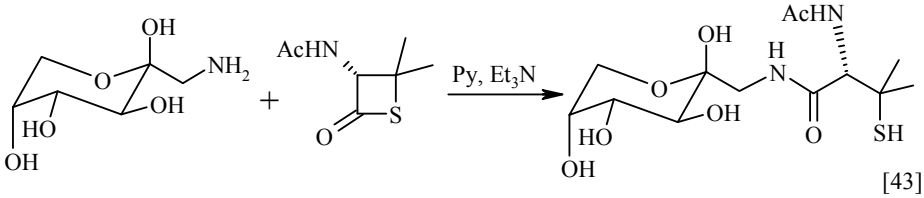
87%

The ring of 3-aminothietan-2-ones is readily opened by the action of amines, and for this reason they have found use as alkylating reagents.



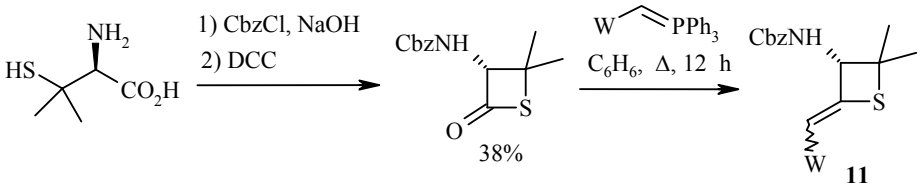
[42]

70%



[43]

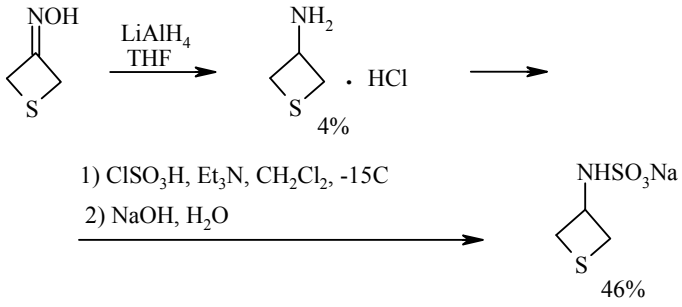
In addition, 3-aminothietan-2-ones protected at the nitrogen atom can be brought into the Wittig reaction with stable phosphorus ylides, whereas this reaction is unknown for the five-membered analogs dihydro-2(3H)-oxothiophenes [44].



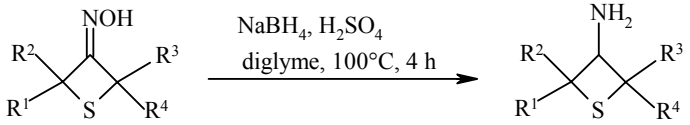
W	Yield, %	
	Z-**11**	*E*-**11**
CO_2Et	70	20
CN	64	
COMe	47	

8. Other Methods

The use of thietan-3-one as starting compound is common to several other synthetic approaches to derivatives of 3-aminothietane described in the literature. The reduction of thietan-3-one oxime to unsubstituted 3-aminothietane with a very low yield was reported; the obtained amine was then used for the synthesis of a derivative of sulfamic acid in the course of searches for harmless analogs of the synthetic flavor additive cyclamate [45].

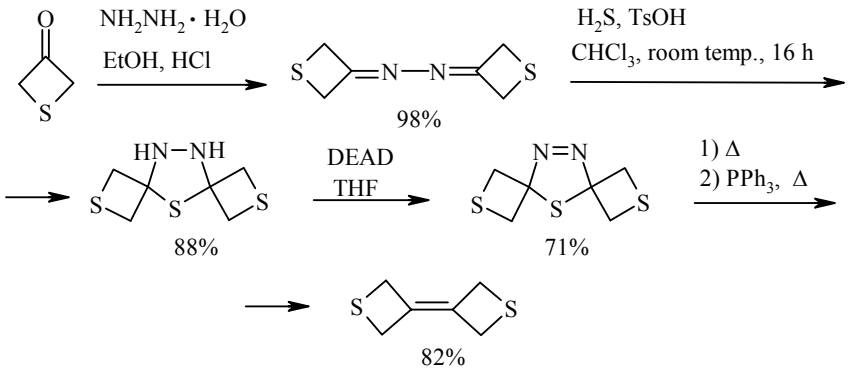


The possibility of reducing 3-thietanone oximes with a wide range of substituents at positions 2 and 4 with yields (~90%) by the action of diborane in diglyme in the presence of an excess of sulfuric acid at elevated temperature was reported in the patent [46].

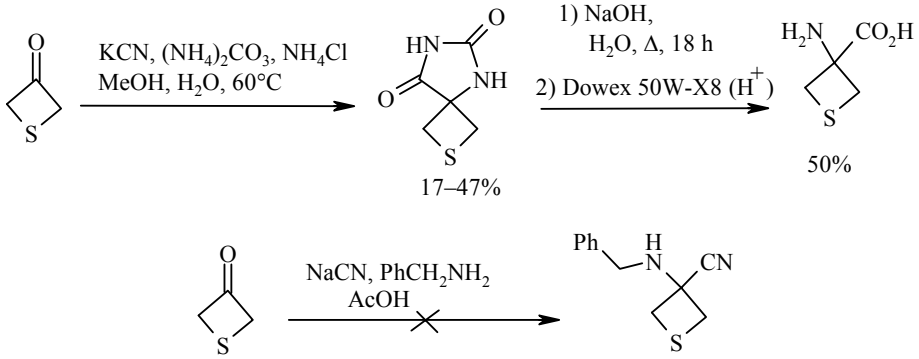


R^1–R^4 = H, alkyl, aryl

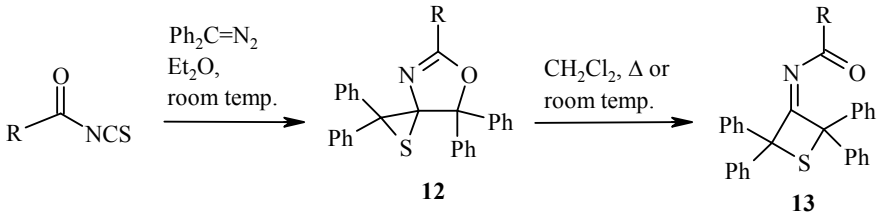
Derivatives of thietane with the nitrogen atom at position 3 were intermediate compounds in the synthesis of 3,3′-bithietanylidene [47].



3-Aminothietane-3-carboxylic acid was obtained in two stages with a low overall yield from thietan-3-one through hydantoin (a modified Bucherer–Bergs synthesis) followed by hydrolysis, since the Strecker synthesis does not lead to the desired result for this ketone [48].



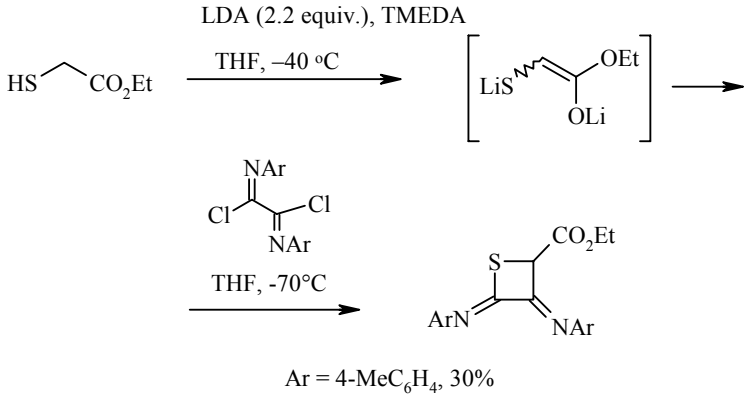
Isolated examples of the synthesis of 3-iminothietanes are known. In the reaction of acyl thiocyanates with 2 equiv of diphenyldiazomethane the intermediately formed spirocyclic compound is unstable and undergoes rearrangement to 3-acylimino-2,2,4,4-tetraphenylthietane spontaneously or with gentle heating [49].



12 **13**

R	Yield, %	
	12	**13**
CCl_3	37	83
CO_2Me	60	72
Ph	67	92
OEt	51	96
t-Bu	73	70

The dianion produced by the action of 2 equiv of a strong base on thioglycolic ester enters into a cyclization reaction with a 1,2-dielectrophile that is unusual for 1,2-dianions. The role of 1,2-dielectrophile is played by bis(imidoylchloride), and the reaction leads to the formation of a derivative of 2,3-diiminothietane [50].



Ar = 4-MeC_6H_4, 30%

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