FREE RADICAL INTRAMOLECULAR CYCLIZATION IN THE SYNTHESIS OF HETEROCYCLES (REVIEW)

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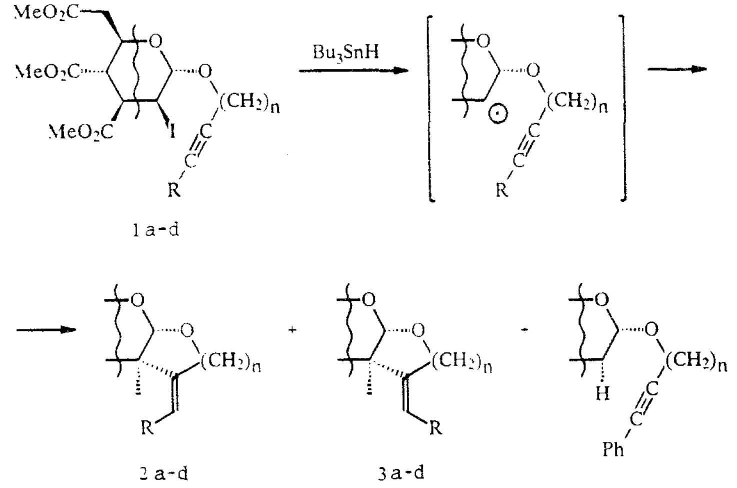
The possibilities of homolytic cyclization for the synthesis of heterocyclic compounds are examined. Factors affecting the yield and structure of the resulting products are discussed.

Intramolecular cyclization forms the basis of an enormous number of methods of obtaining heterocyclic compounds [1]. Increased interest has been observed recently in free radical cyclization, chiefly in the synthesis of bi- and polycyclic heterocycles [2, 3]. The advantages of this method include high regio- and stereoselectivity and the possibility of obtaining polyfunctional heterocycles. As a rule, carbon-centered radicals are generated in the first step from appropriate unsaturated halides (iodides, bromides, and more rarely chlorides). These are capable of adding intramolecularly at a multiple carbon–carbon bond. The results of using this and other recently published methods are considered in the present review.

1. Oxygen-Containing Heterocycles

The intramolecular addition of carbon-centered radicals at a carbon–carbon triple bond is a general method of free radical heterocyclization.

An intramolecular radical addition occurs when appropriate acetylenic iodides (1a-d) are reduced with tributylhydridotin [4]. Compounds with tetrahydrofuran (2a-c), (3a-c) or with tetrahydropyran (2d, 3d) rings are formed in high yield as a result.



1a-d

2a-d

3a-d

R = Ph (1a—3a, 1d—3d); R = n-Bu (1b—3b); R = t-Bu (1c—3c); n = 1 (1a–c, 2a–c, 3a–c);
n = 2 (1d–3d)

The cyclohexyl radicals generated in the first step add intramolecularly to the carbon–carbon multiple bond and the stereochemistry of the products is determined by the type of substituent at the triple bond (Table 1). Thus, a phenyl or n-alkyl

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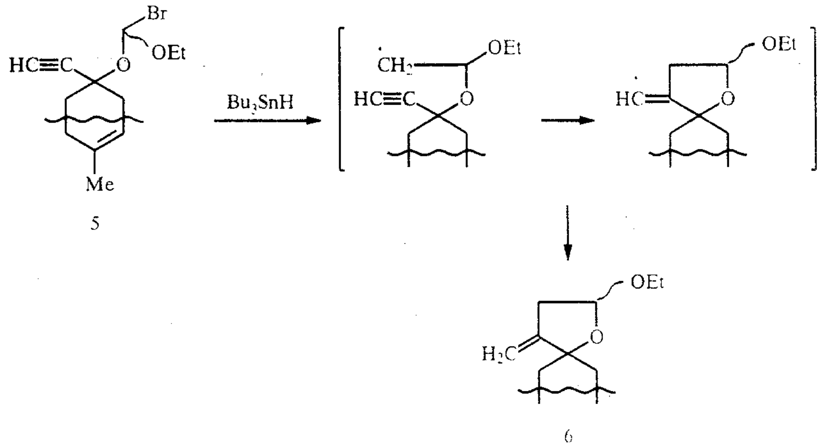
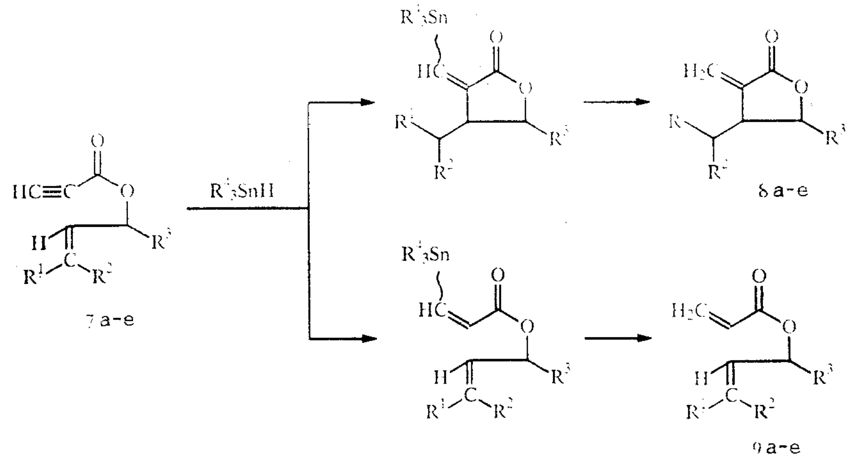
group causes the predominant formation of the endo forms of (2a), (2b), and (2d) while in the case of the bulky t-butyl group the exo isomer (3c) predominates. However, the structure of the substituent at the triple bond has practically no influence on the product yield. The intramolecular formation of five-membered rings proceeds significantly more readily than of six-membered rings which is indicated by the absence of hydrogenation products in the case of compounds (1a-c) and the high yield of linear ether on reducing substance (1d) [4].

It was shown in [5] that radical cyclization may be used successfully in the synthesis of spirocyclic lactones. In particular, reduction of an appropriate bromoacetaldehyde derivative (5) leads to the formation of the methylenic precursor of andirolactone (6).



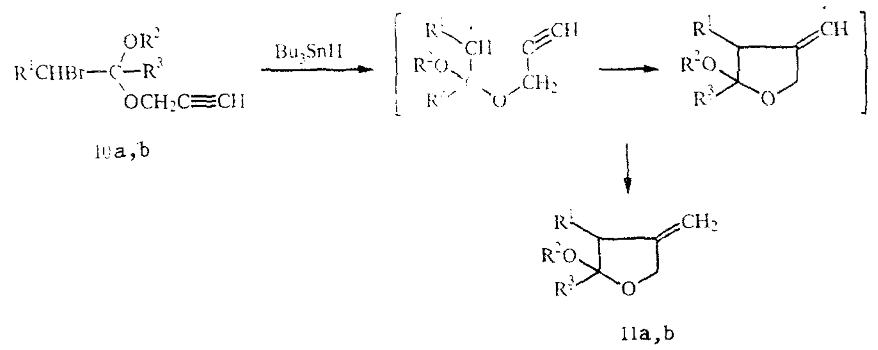
The α-methylene-γ-butyrolactones (8a-e) were obtained from appropriate alkenyl esters of propiolic acid (7a-e).



$R^1 = H, R^2 = H, R^3 = H$ (7a, 8a, 9a); $R^1 = Me, R^2 = H, R^3 = H$ (7b, 8b, 9b); $R^1 = Me, R^2 = Me, R^3 = H$ (7c, 8c, 9c); $R^1 = H, R^2 + R^3 = -(CH_3)_2-$ (7d, 8d, 9d); $R^1 = Ph, R^2 = H, R^3 = H$ (7e, 8e, 9e); $R^4 = Bu, Ph$

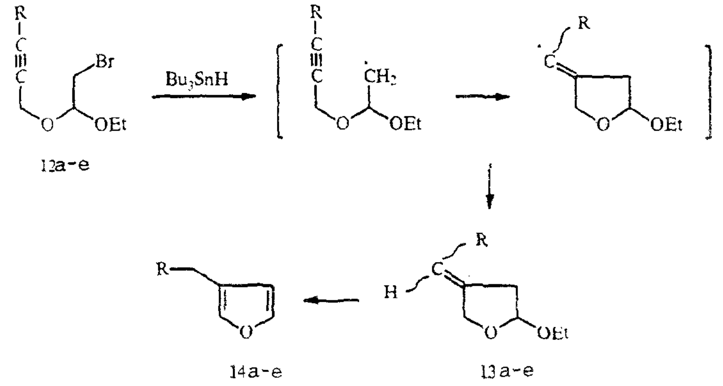
The addition of tin hydrides at the acetylenic bond in the first step occurs with the parallel formation of the corresponding cyclic and linear organotin compounds, the quantitative destination of which leads to the methylene–lactones (8a-e) and the acrylic acid esters (9a-e). It has been shown that the butylhydridotin was less reactive than the hydridophenyltin derivative but was preferred for the synthesis of lactones. The exception was the ester of cinnamyl alcohol (7e) which was cyclized by the action of $(C_6H_5)_3SnH$ with a higher selectivity.

The corresponding methylenetetrahydrofurans (11a, b) were obtained from the propargyl acetals of the bromoaldehydes (10a, b) by reduction [7]. Depending on the nature of the substituents the yields varied from 40 to 70%.



$R^1 + R^2 = (CH_2)_3$, $R^3 = H$ (10a, 11a); $R^1 + R^3 = (CH_2)_4$, $R^2 = Me$ (10b, 11b)

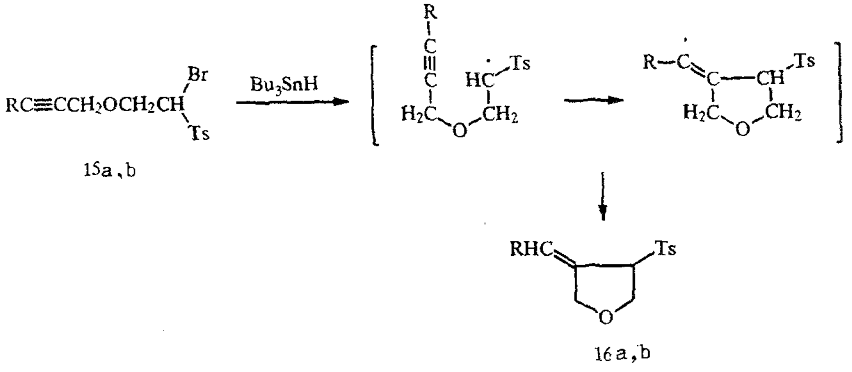
The cyclization of propargyl acetals of bromoacetaldehyde (12a-e) to the corresponding alkoxytetrahydrofurans (13a-e) proceeds in a mainly similar manner [8]. This served as the key stage in the synthesis of the 3-substituted furans (14a-e).



$R = n\text{-}C_6H_{13}$ (12a—14a); $R = n\text{-}Bu$ (12b—14b); $R = COOEt$ (12c—14c);
$R = Me_2C{=}CH{-}(CH_2)_2{-}CH_2$ (12d —14d);
$R = Me_2C{=}CH{-}(CH_2)_2{-}CMe{=}CH{-}(CH_2)_2CH_2$ (12e —14e)

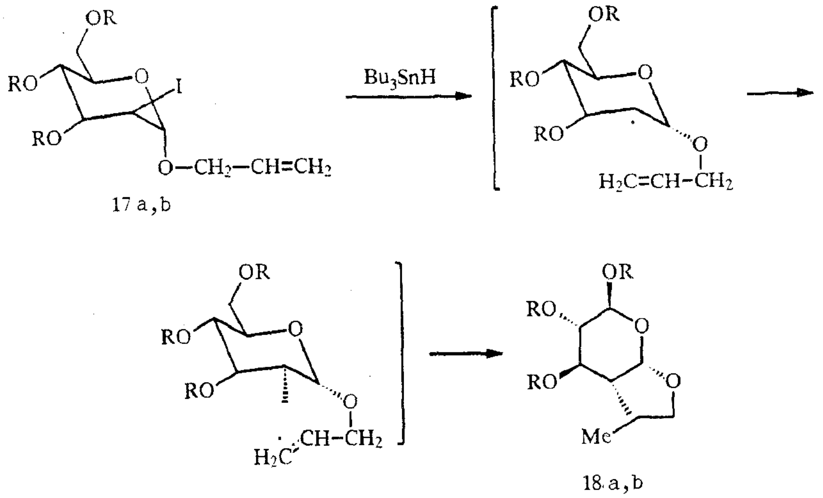
Reduction of the propargyl esters of the bromine-containing alcohols (15a, b) is accompanied by cyclization to the methylenetetrahydrofurans (16a, b) [9].

A phenyl residue at the triple bond increases the yield of the cyclic ether (16a) to 90%.



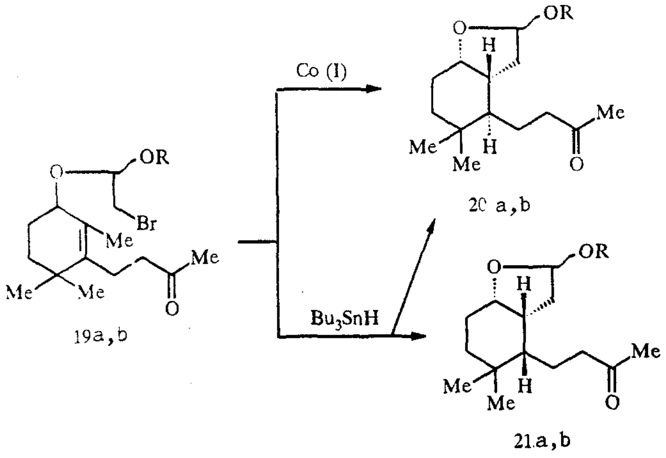
$R = Ph$ (15a, 16a); $R = C_5H_{11}$ (15b, 16b)

Intramolecular radical addition at a carbon–carbon double bond is fairly widespread. The cyclization of iodides containing allyloxy groups (17a, b) is based on this principle [10, 11]. The corresponding bicyclic compounds (18a, b) are obtained in 65-80% yield which indicates the lower reactivity of an ethylenic compared with an acetylenic bond in intramolecular radical cyclization (Table 1).



R = CH_3CO (17a, 18a); R = $Me_2SiC(CH_3)_3$ (17b, 18b)

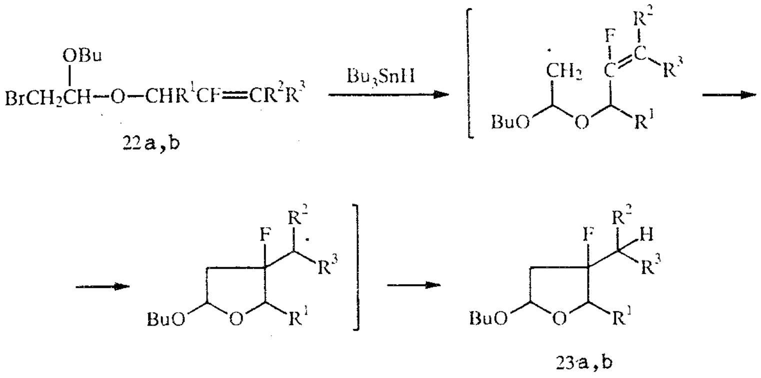
It has been shown [12] that the stereospecificity of radical cyclization depends significantly on the nature of the reducing agent. In the presence of complexes of univalent cobalt the compounds (19a, b) form only the isomers (20a, b) while reduction with tributylhydridotin leads to the parallel formation of (20a, b) and (21a, b).



R = Me (19a, 20a, 21a); R = Et (19b, 20b, 21b)

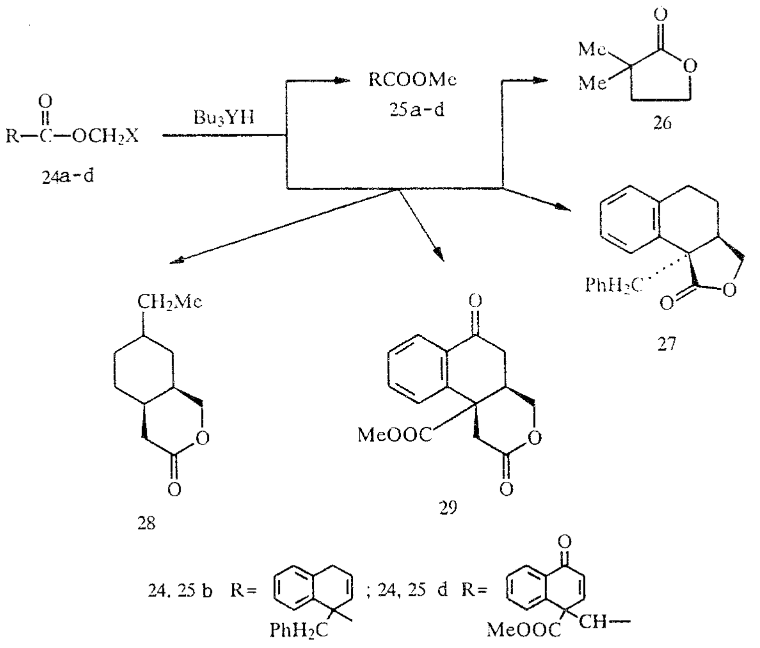
Probably the stereochemistry of reducing the bromides (19a, b) with tributylhydridotin is determined by 1,3-diaxial interactions in the intermediate cyclohexyl radicals but when using univalent cobalt the cyclization proceeds through an intermediate organocobalt compound.

It has been shown [13] that the radical cyclization of unsymmetrical acetals of bromoacetaldehyde (22a, b) occurs with the formation of the corresponding substituted tetrahydrofurans (23a, b). The presence of a fluorine atom at the carbon–carbon double bond increases the yield of cyclization products to 75-80%.



$R^1 = C_6H_{13}$, $R^2 = R^3 = H$ (22a, 23a); $R^1 = CH_3$, $R^2 = H$, $R^3 = C_6H_5CH_2$ (22b, 23b)

The formation of lactones by radical cyclization was considered in [14]. Reduction of the corresponding selenophenol derivatives (24a, b) or the iodomethyl esters (24c, d) with an equimolar quantity of tributylhydridotin or of tributylhydridogermanium leads to cyclic products (26)-(29).



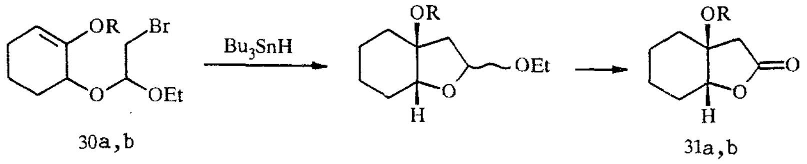
R = $(CH_3)_2CCH{=}CH_2$ (24a, 25a); X = SeC_6H_5 (24a,b);
R = $CH_3{-}CH_2{-}CH{-}CH{=}CH{-}CH{-}$ (24c, 25c); Y = Se, Ge; X = I (24c,d)

The heterocyclic compounds (26)-(29) formed are of significant interest and are available with difficulty by other methods (Table 2).

The formation of lactones is facilitated (Table 2) when the double bond is included in a carbocyclic fragment particularly if it is conjugated with an electron-accepting carbonyl group.

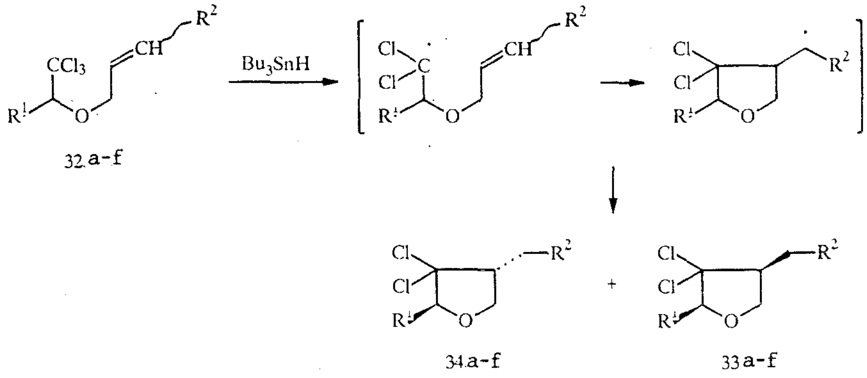
It was shown, using compound (24a) as an example, that at 80°C the rate constant (K_c) for the intramolecular cyclization of the intermediate acyloxymethyl radical $CH_2{=}CH{-}C(CH_3)_2{-}COO\dot{C}H_2$ was an order of magnitude less than the rate constant (K_H) for the fission of hydrogen from $(C_4H_9)_3SnH$, i.e., $K_c/K_H = 0.12$ mole/liter.

The cyclization of the bromoacetals (30a, b), which is proposed for the preparation of γ-butyrolactones (31a, b), is similar in many ways [15].



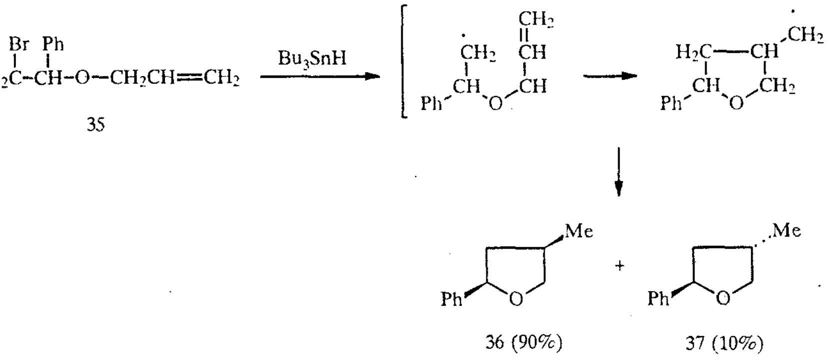
R = t-C_4H_9 (30a, 31a); R = CH_3 (30b, 31b)

The intramolecular addition is assisted, as a rule, by aromatic substituents located in the α position of the resulting heterocycle [16]. It is interesting that in this case cis isomers predominate in the reaction products (Table 3).



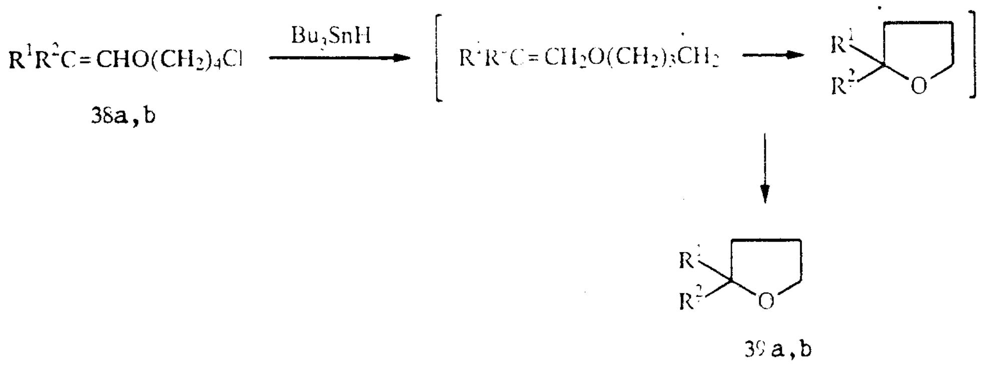
$R^1 = C_6H_5$ (32 a,f, 34a,f), $R^2 = C_6H_5$ (32a—34a); $R^1 = p$-ClC_6H_4, $R^2 = H$ (32c,d,e–34b,c,d,e);
$R^1 = C_5H_{11}CHC_2H_5$ (32c—34c); $R^1 = C_3H_7$ (32d–34d; $R^1 = CH_3COOCH_2$ (32e —34e),
$R^2 = CH_3$ (32f, 33f, 34f)

The reduction of α-bromomethylbenzyl allyl ether (35) is accompanied by cyclization and leads to the formation of a mixture of stereoisomeric 4-methyl-2-phenyltetrahydrofurans (36, 37) in which the cis form (36) predominates [16].



This stereochemical result of the cyclization is determined evidently by the conjugation of the unpaired electron with the aromatic substituent in the β position.

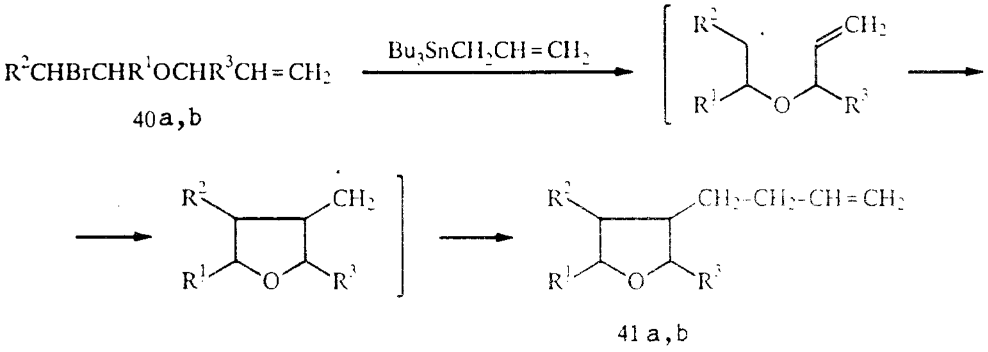
The 2,2-disubstituted tetrahydrofurans (39a, b) were obtained on reducing the 4-chlorobutyl vinyl ethers (38a, b) [17].



$R^1 = CN, R^2 = C_2H_5OOC$ (38a, 39a); $R^1 = CH_3CO, R^2 = CH_3OOC$ (38b, 39b)

Satisfactory yields were obtained only when the substituents had a clearly marked electron-accepting character.

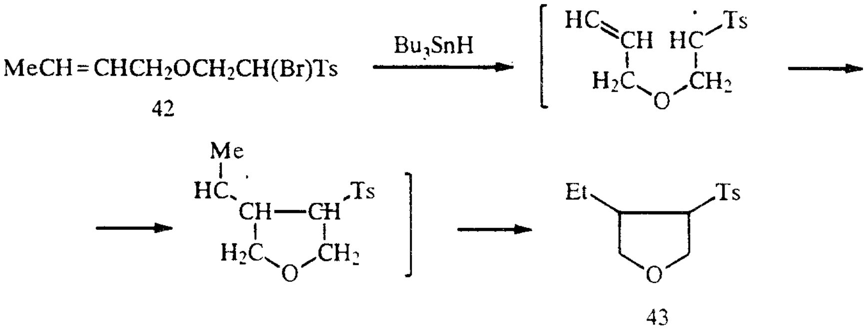
An interesting variant of the cyclization of the terminal olefins (40a, b) with homolytic substitution at the tin atom was proposed in [9].



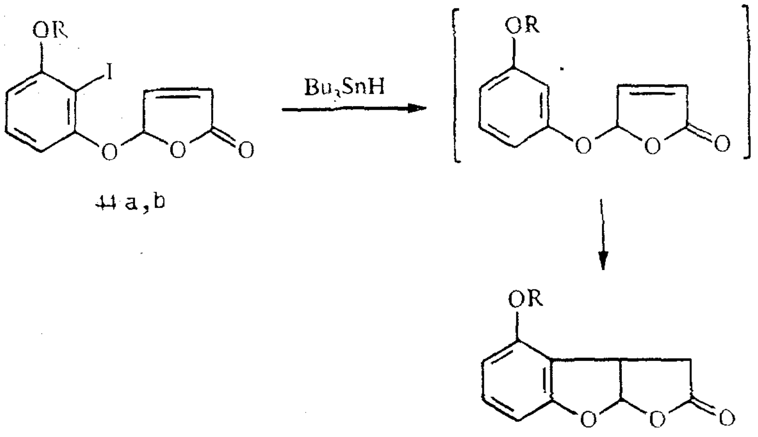
$R^1 + R^2 = (CH_2)_4, R^3 = H$ (40a, 41a); $R^1 = R^2 = H, R^3 = CH_3$ (40b, 41b)

The yield of the alkenyltetrahydrofurans (41a, b) did not exceed 50%; however, it was noted that these compounds are available with difficulty by other methods.

The selective formation of tetrahydrofuran derivatives (43) was recorded in the reductive cyclization of the allyl β-bromoethyl ethers (42) [18].



It has been shown [19] that the reduction of appropriate haloaromatic compounds (44a, b) serves as a convenient method of obtaining the substituted benzofurans (45a, b), the yields of which reached 80%.

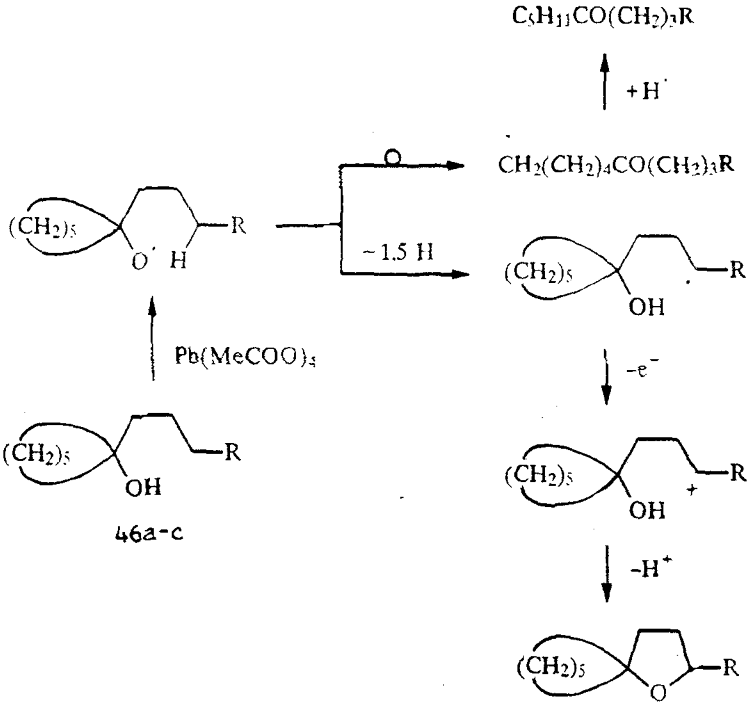


44a,b

45a,b

R = CH_3 (44a, 45a); R = CH_3OCH_2 (44b, 45b)

Intramolecular rearrangements of free radicals in systems providing one-electron transfers may also lead to the formation of oxygen-containing heterocycles. It was shown in several studies [20-23] that the oxidation of hydroxyl-containing compounds with lead tetraacetate may be accompanied by the formation of the corresponding oxygen-containing heterocycles (47a-c). A typical example of such a cyclization is the oxidation of 1-alkylcyclohexanols (46a-c) with side chains containing more than three carbon atoms [22].



46a-c

47a-c

R = CH_3 (46a, 47a); R = C_3H_7 (46b, 47b); R = C_5H_{11} (46c, 47c)

Tetrahydrofuran derivatives are not formed from 1-alkylcyclopentanols since in this case only a β-fission of the intermediate oxygen-centered radicals takes place.

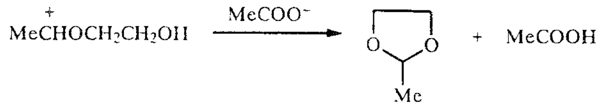
There is definite interest in the oxidative cyclization of 2-ethoxyethanol to 2-methyl-1,3-dioxolane in the synthesis of cyclic acetals [23]. Evidently the reaction includes rearrangement of the 2-ethoxyethyloxyl radicals with a 1,5-migration of a hydrogen atom and subsequent one-electron oxidation and cyclization (see top of following page).

The yield of 2-methyl-1,3-dioxolane did not exceed 30%; however, in the case of other cellosolves and more efficient oxidizing agents it would be expected that the method proposed is of preparative interest.

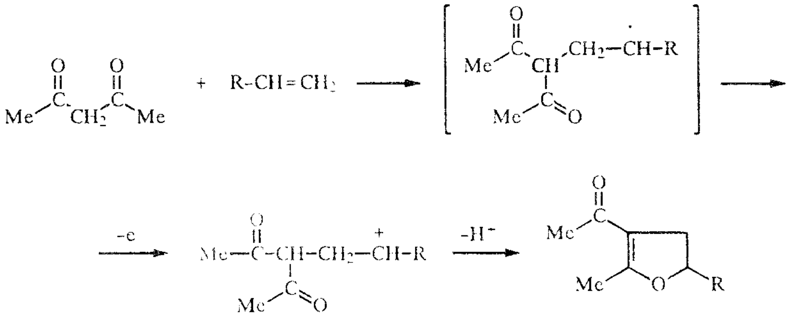
$$2\,EtOCH_2CH_2OH \xrightarrow{Pb(MeCOO)_4} 2\,EtOCH_2CH_2\dot{O} + 2\,MeCOOH + Pb(MeCOO)_2$$

$$EtOCH_2CH_2\dot{O} \longrightarrow Me\dot{C}HOCH_2CH_2OH$$

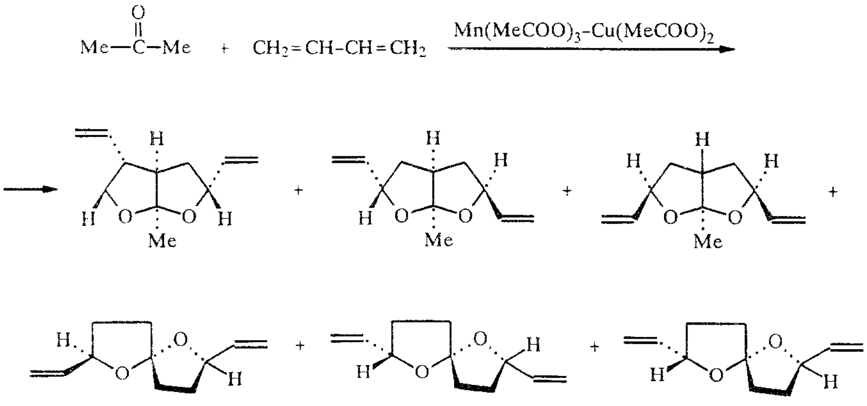
$$2\,Me\dot{C}HOCH_2CH_2OH \xrightarrow{Pb(MeCOO)_4} 2\,Me\overset{+}{C}HOCH_2CH_2OH + Pb(MeCOO)_2 + 2\,MeCOO^-$$



It is possible to form mono- and spirocyclic tetrahydrofurans by cyclization of the intermediate carbocations in the oxidative addition of carbonyl compounds to olefins and dienes [24, 25]. Acetylacetone reacts with terminal olefins under the action of Mn(III) or Co(III) forming tetrahydrofuran derivatives [24].

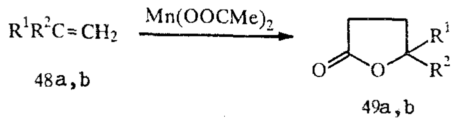


Under analogous conditions acetone reacts with butadiene forming a mixture of isomeric dioxaspirononanes [25].



The multistage reaction mechanism proposed includes the intermediate formation and cyclization of allyl carbocation with the carbonyl group in the γ position.

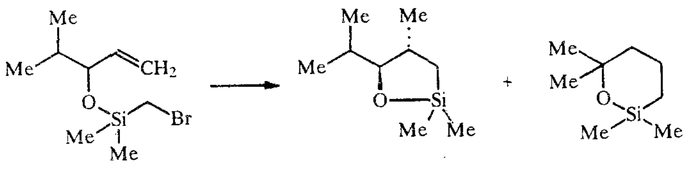
The one-electron oxidation of gem-disubstituted ethylenes (48a, b) by manganese complexes in acetic acid solution also leads to the γ-butyrolactones (49a, b). However, in this case the yield of the products (49a, b) did not exceed 60% [26].



$R^1 = R^2 = C_6H_5$ (48a, 49a); $R^1 = p\text{-}CH_3C_6H_4$ (48b, 49b); $R^2 = C_6H_5$ (48b, 49b)

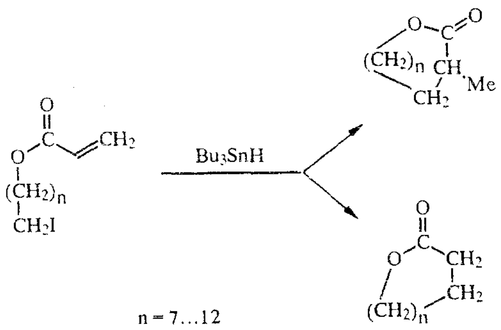
Probably a change of conditions and the type of catalyst may increase significantly the yield of the desired lactones.

Cases are known of forming a ring containing a silicon atom from the corresponding dimethylbromomethylallyloxysilane [27].



The six-membered siloxanes are the main cyclization products (yield over 80%).

Recently [28, 29] a homolytic cyclization has been proposed for the synthesis of macrocyclic lactones. The reduction of the acrylate esters of the appropriate iodohydrins enables macrocycles to be synthesized in one step.



This method is the most acceptable for obtaining lactones with a ring size greater than 10. The yield of the desired compounds reached 90%.

TABLE 1. Radical Cyclization of Iodine-Containing Ethers [4] [Bu_3SnH/azoisobutyronitrile (AIBN), benzene, 80°C, 24 h]

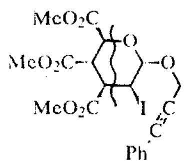
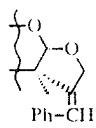
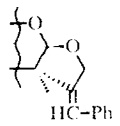
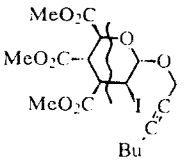
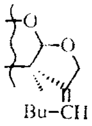
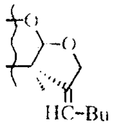
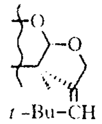
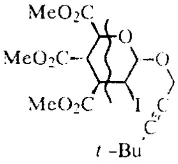
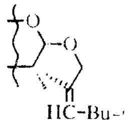
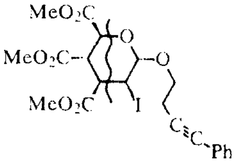
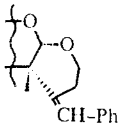
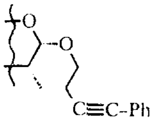
Reactant	Yield, %	Reaction products	Selectivity, %
 1 a	85	 2 a	74
		 3 a	26

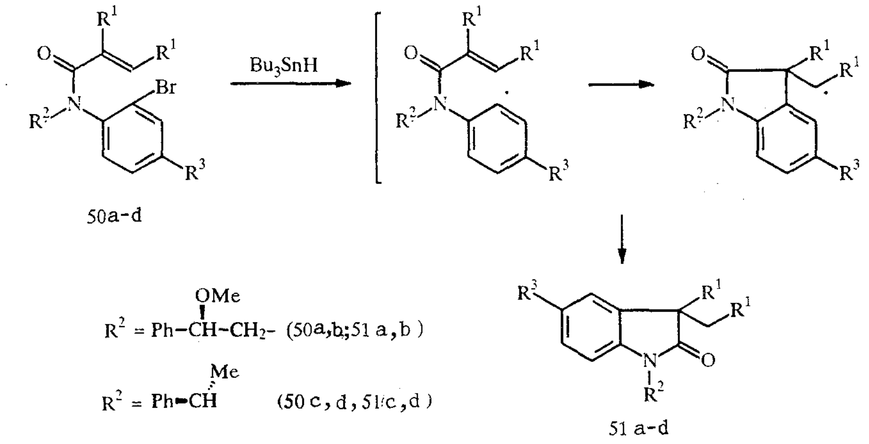
TABLE 1 (continued)

 1b	90	 2b	71
		 3b	29
		 2c	36
 1c	91		
		 3c	64
		 2d	38
 1d	79	 3d	31
		 4	31

2. Nitrogen- and Sulfur-Containing Heterocycles

In several cases intramolecular radical addition leads to the formation of nitrogen- or sulfur-containing heterocycles.

Derivatives of indole have been obtained [30] on reducing ortho-haloacrylanilides with tributylhydridotin.



50a-d

51 a-d

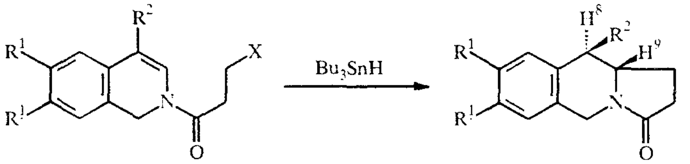
R^2 = Ph–CH(OMe)–CH$_2$– (50a,b;51 a,b)

R^2 = Ph–CH(Me) (50 c, d, 51c, d)

R^1 – H (50a,c, 51a,c); R^1 – CH_3 (50b,d, 51b,d). R^3 – H (50ab, 51a,b); R^3 – CH_3O (50c,d 51c,d)

Methyl groups at the carbon–carbon double bond and methoxyl groups in the aromatic nucleus increase the yield of the desired products (Table 4).

The benzo[f]indolizidines (53a-e) are obtained on reducing 2-(2-halopropionyl)-1,2-dihydroisoquinolines (52a-e) under standard conditions [31].



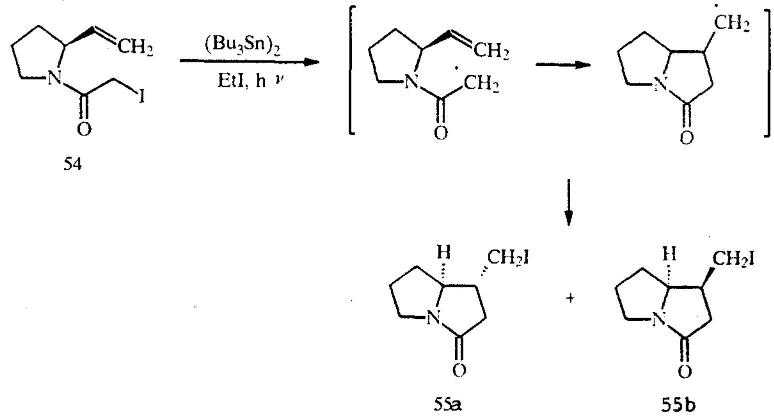
52 a-e

53 a-e

X – I (52a,c,d,e), X – Br (52b); R^1 = H (52a–d, 53a–d); R^1 = CH_3O (52e,53e); R^2 = H (52a,b,e, 53a,b,e); R^2 = CN (52c, 53c); R^2 = CO_2CH_3 (52d,53d)

The yield of product was reduced on replacing the iodine by a bromine atom while electron-accepting groups in the meta-position to nitrogen stimulated cyclization (Table 5).

The homolytic cyclization of iodoacetic acid amides has been used in the synthesis of pyrrolizidines [32, 33]. The generation of radicals by photodecomposition with $[(C_4H_9)_3Sn]_2$ in ethyl iodide enabled a mixture of the stereoisomers of (55a, b) to be obtained in 50-60% yield.

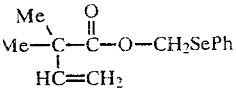
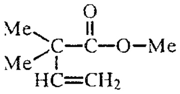
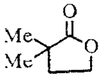
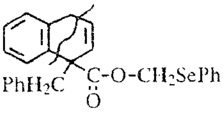
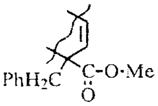
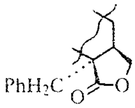
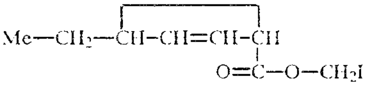
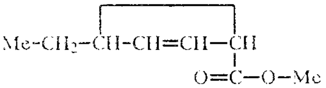
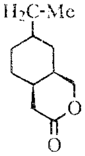
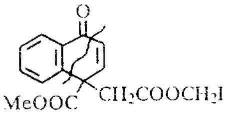
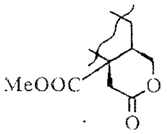


54

55a

55b

TABLE 2. Radical Cyclization of Derivatives of Selenophenol and Iodomethyl Esters [14] [Bu_3SnH (X = SeC_6H_5)/AIBN, Bu_3GeH (X = I)/AIBN, benzene, 80°C, 4 h]

Reactant	Yield, %	Reaction products	Selectivity, %
 $\mathrm{Me{-}\underset{HC=CH_2}{\overset{Me}{C}}{-}\overset{O}{\overset{\Vert}{C}}{-}O{-}CH_2SePh}$ 24 a	95	 $\mathrm{Me_2\underset{HC=CH_2}{C}{-}\overset{O}{\overset{\Vert}{C}}{-}O{-}Me}$ 25a	66
		 2b	34
 24b	80	 25b	18
		 27	82
 $\mathrm{Me{-}CH_2{-}CH{-}CH{=}CH{-}\underset{O=C-O-CH_2I}{CH}}$ 24c	85	 $\mathrm{Me{-}CH_2{-}CH{-}CH{=}CH{-}\underset{O=C-O-Me}{CH}}$ 25 c	95
		 28	5
 24d	90	 25d	8
		 29	92

It is interesting that the traditional reduction of compound (54) with tributylhydridotin to the target product did not occur in practice.

Tin hydrides proved to be effective when reducing the iodomethyl-lactams (56a-f) [34]. In this case the bicyclic products (58a-f) were formed in extremely high yield (50-95%) in addition to the methyl derivatives (57a-f) (Table 6).

TABLE 3. Radical Cyclization of 2,2,2-Trichloroethylallyl Ethers [16] (Bu_3SnH/AIBN, benzene, 80°C, 1 h)

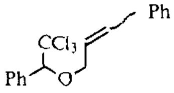
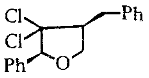
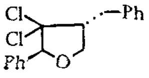
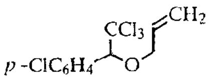
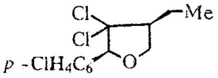
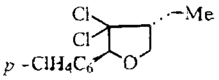
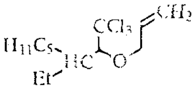
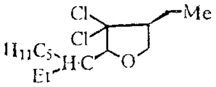
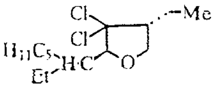
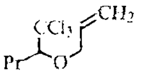
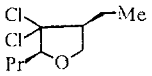
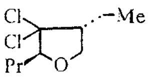
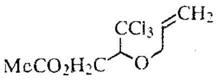
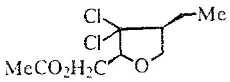
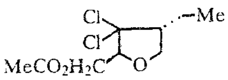
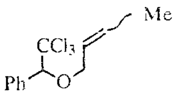
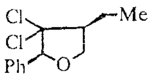
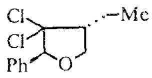
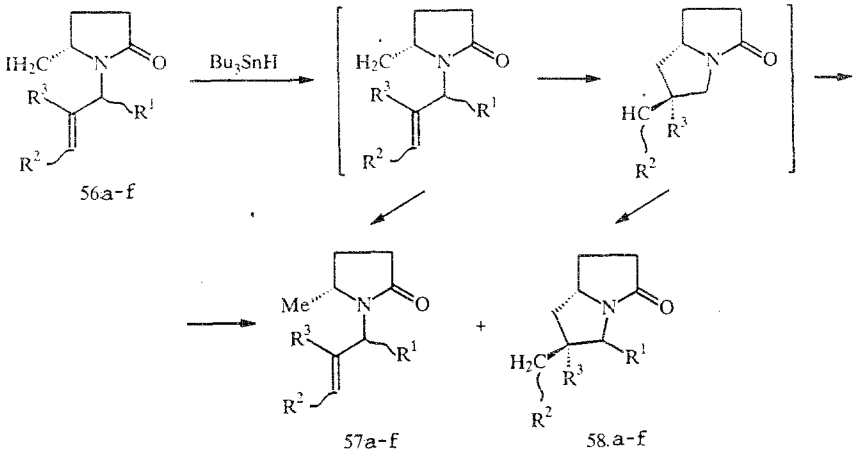
Reactant	Yield, %	Products	Selectivity, %
 32a	79	 33a	92
		 34a	8
 32b	82	 33b	91
		 34b	9
 32c	80	 33c	90
		 34c	10
 32d	72	 33d	86
		 34d	14

TABLE 3 (continued)

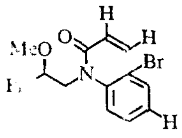
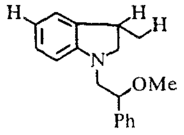
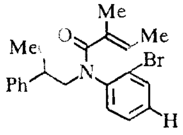
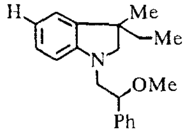
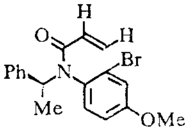
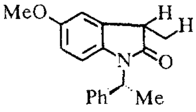
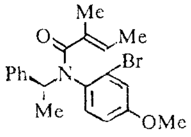
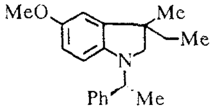
Reactant	Yield, %	Products	Selectivity, %
 32e	74	 33e	87
		 34e	13
 32f	86	 33f	90
		 34f	10



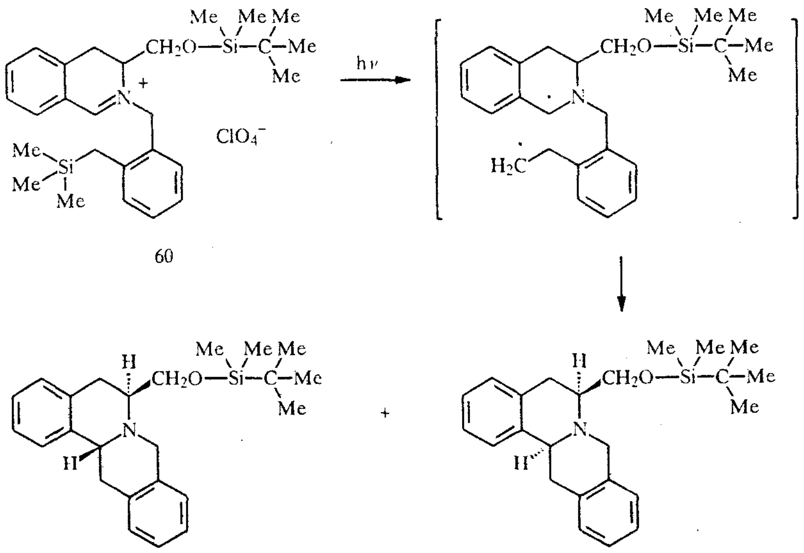
$R^1 = H$ (56a–d, 57a-d, 58a–d); $R^1 = Me$ (56d, 57d, 58d); $R^1 = (CH_2)_2CH_3$ (56f, 57f, 58f); $R^2 = H$ (56a,d,e,f, 57a,d,e,f, 58a,d,e,f); $R^2 = Ph$ (56b, 57b, 58b); $R^2 = Me$ (56c, 57c, 58c); $R^3 = H$ (56a-c,e,f, 57a-c,e,f, 58a-c,e,f); $R^3 = Me$ (56d 57d,58d)

Cyclization was facilitated by the presence of a methyl group at the double bond in the position β to the heteroatom.

TABLE 4. Radical Cyclization of ortho-Haloacrylanilides [30] (Bu_3SnH/AIBN, toluene, 0.5 h, 111°C)

Reactant	Product	Yield,%
 50a	 51a	83
 50b	 51b	79
 50c	 51c	64
 50d	 51d	59

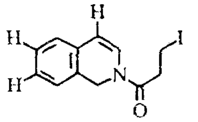
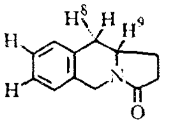
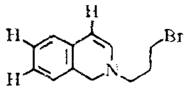
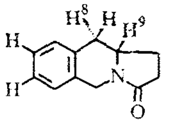
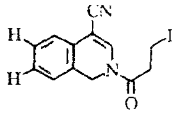
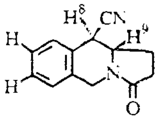
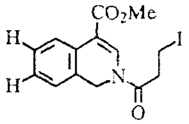
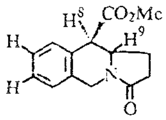
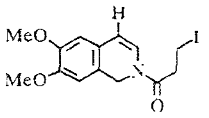
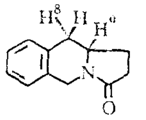
An example of the photocyclization of an N-silylbenzylhydroisoquinolinium salt (60) has been described [35].

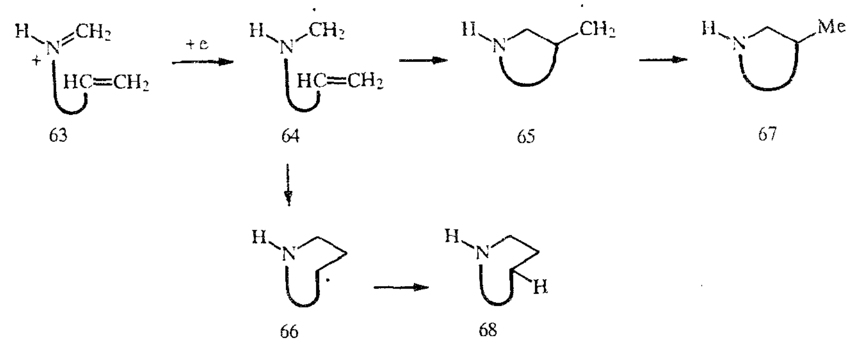


The stereoisomers (61a) and (61b) are evidently formed by the intramolecular recombination of intermediate biradicals.

There are data [36] on the synthesis of pyrrolidines and piperidines by the one-electron oxidation of the corresponding salts (63).

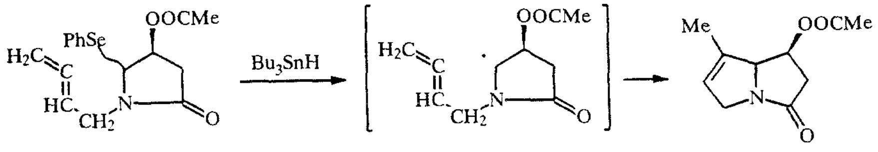
TABLE 5. Radical Cyclization of 2-(2-Halopropionyl)-1,2-dihydroisoquinolines [31] (Bu_3SnH/AIBN, benzene, 80°C, 3 h)

Reactant	Product	Yield, %
 52a	 53a	62
 52b	 53b	45
 52c	 53c	81
 52d	 53d	73
 52e	 53e	62



It is probable that α-aminomethyl radicals (64) are formed in the first step. These add intramolecularly at the multiple bond and lead to the products (67) and (68) through the intermediate radicals (65) and (66).

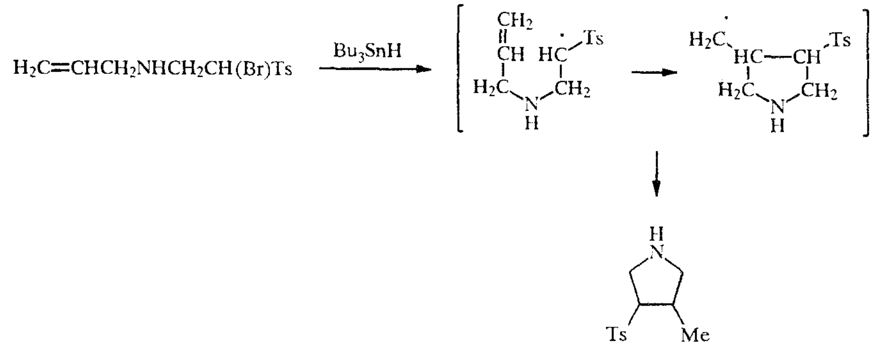
The synthesis of pyrrolizidine bases, including a radical cyclization stage, has been reported [37].



69

The bicyclic intermediate (69) is formed in 60% yield. Replacement of the allene fragment by ethylene leads to a saturated bicyclic structure.

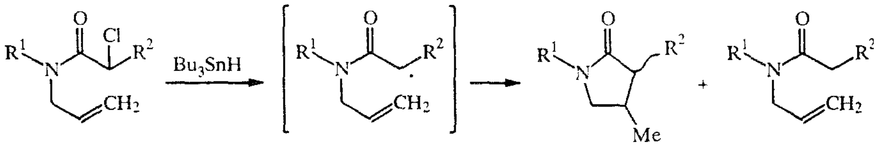
The radical cyclization to form the pyrrolidine derivative (70) proceeds stereoselectively [35].



70

The yield of compound (70) reached 90%; it was a mixture of cis and trans isomers with a predominance of the latter.

It has been shown that radical cyclization is an intermediate step in the synthesis of γ-butyrolactams from the corresponding N-allyl-α-chloroacetamides [38].



71a,b

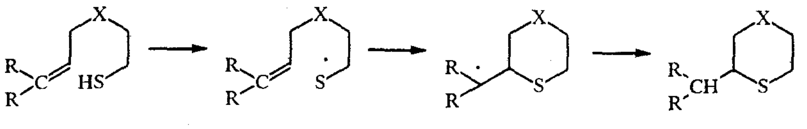
72a,b

73a,b

$R^1 = CH_3, R^2 = SCH_3$ (71a, 72a, 73a); $R^1 = CH_2P, R^2 = H$ (71b, 72b, 73b)

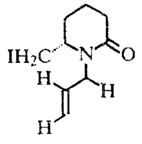
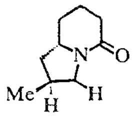
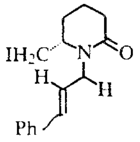
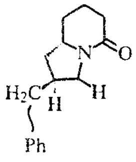
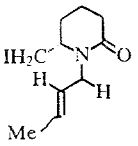
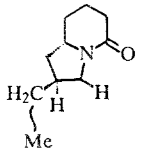
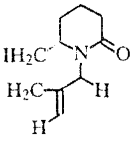
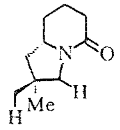
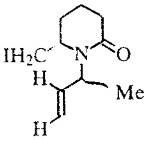
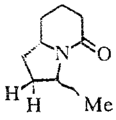
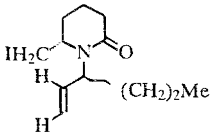
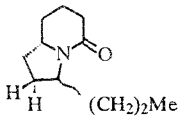
The yield of linear reduction products did not exceed 20% in the case of alkylthio derivatives ($R^2 = CH_3S$) and increased to 40% when $R^2 = H$. This is evidently connected with the fact that the intermediate radical with two captodative substituents ($R^2 = CH_3S$) is incapable of splitting out hydrogen from tributylhydridotin and the intramolecular addition is practically the only route for its decomposition.

Intramolecular homolytic addition with the formation of sulfur-containing heterocycles has been described in detail in a review [3]. The generation of sulfur-centered radicals by the fission of an S−H bond and intramolecular addition of them at a multiple bond is as normal.



$X = O, S; R = H, C_nH_{2n+1}, Cl$

TABLE 6. Radical Cyclization of N-Allyl-5-iodomethyl-2-pyrrolidinones [34] (Bu_3SnH/AIBN, benzene, 80°C)

Reactant	Time, h	Product*	Yield, %
 56a	4	 58a	70
 56b	3	 58b	72
 56c	12	 58c	52
 56d	12	 58d	96
 56e	12	 58e	54
 56 f	12	 58 f	58

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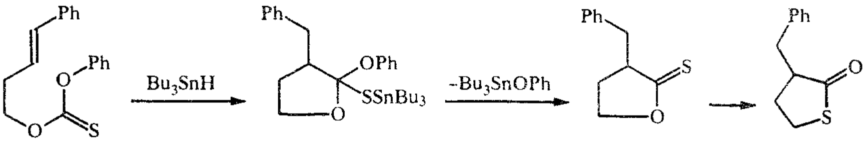
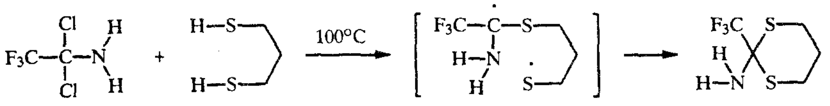
*In each case the by-products were the corresponding compounds (57a-f).

An intramolecular addition is also the intermediate step in the reductive cyclization of thiocarbonic acid esters [2].



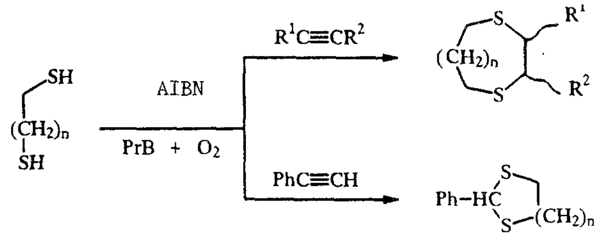
The intermediate organotin compounds decompose readily, which explains the high yield of lactones and thiolactones.

An interesting example of cyclization is the thermolysis of a mixture of suitable α,α-dichloro-β,β,β-trifluoroethylamines and 1,3-propanedithiol [39].



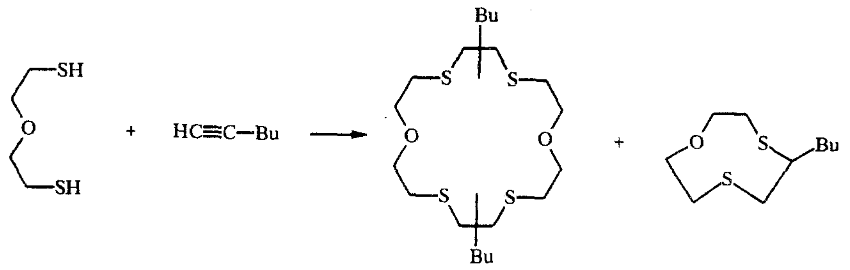
The enhanced stability of the carbon-centered radicals is explained by a captodative effect. The yield of the corresponding 1,3-dithianes from the intramolecular recombination reached 80%.

It has been shown [40, 41] that the homolytic addition of dithiols to an alkyne is a general method for the synthesis of dithiacycloalkanes. On initiation with tripropylborane in a medium of oxygen, or by the decomposition of AIBN, heterocyclization occurs by a type of 1,2-addition with alkylacetylenes, and phenylacetylene reacts only by a type of 1,1-addition [40]. The cis isomers were the main products from a series of 2,3-disubstituted 1,4-dithianes.



R^1, R^2 = alkyl CH_2Cl, CH_2OH, n = 1, 2

This principle has been used in the synthesis of macroheterocycles [41]. Intermolecular homolytic macrocyclization of 3-oxa-1,3-pentanediol with 1-hexyne gave 18- and 9-membered thia-crown ethers.



This is the first example of the use of intermolecular homolytic heterocyclization for the synthesis of thia-crown ethers.

Several variants of the intramolecular free radical cyclization leading to a polyfunctional heterocycle have been described up to the present. This is an important and interesting area of the chemistry of heterocyclic compounds and is widely applied in fine organic synthesis.

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