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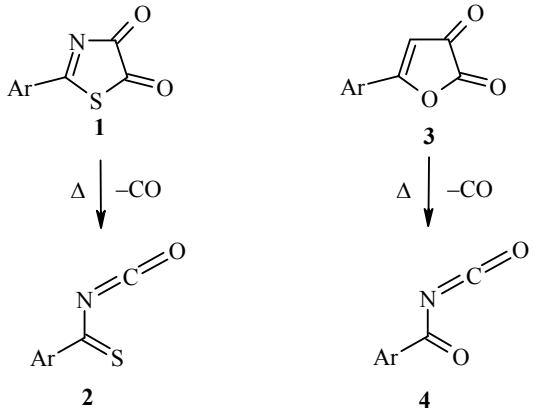
# SYNTHESIS AND CHEMICAL CHARACTERISTICS OF 2-SUBSTITUTED THIAZOLINE-4,5-DIONES. (REVIEW)

**D. D. Nekrasov and A. S. Obukhova**

*Methods for the production of 2-substituted thiazoline-4,5-diones and their thermolysis in the presence of nucleophiles and dienophiles are examined.*

**Keywords:** 2-arylthiazoline-4,5-diones, azaheterocycles, thioaroyl isocyanates, nucleophilic addition, cycloaddition.

In most chemical transformations 2-substituted thiazoline-4,5-diones **1** form intermediate thioacyl isocyanates **2**, which enter readily into nucleophilic addition and cycloaddition. Compounds **1** are heteroanalogs of 5-aryl-2,3-dihydrofuran-2,3-diones **3**, which form a different type of acylheterocumulenes, i.e., the aroylketenes **4**.




In spite of the structural similarity of compounds **1** and **3** their chemical characteristics have substantial differences arising from thermolysis, decyclization, and recyclization of both types of dioxoheterocycle [1]. Thiazolinediones **1** were first described in 1960 [2], and furandiones **3** were described in 1975 [3]. In contrast to the latter, however, there have not so far been any systematic data on compounds **1**. Diones of type **1** were only briefly mentioned in reviews during examination of the chemical transformations of acylheterocumulenes [5], azadienes [6], and cyanamides [7].

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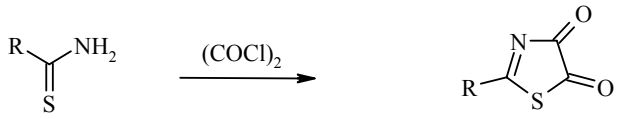
## 1. THE PRODUCTION OF THIAZOLINE-4,5-DIONES

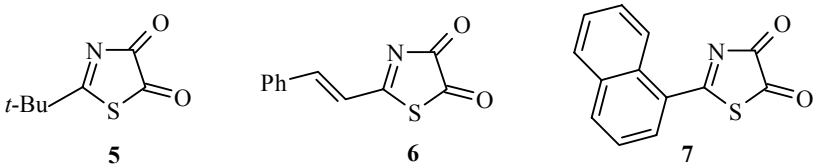
The first examples of thiazoline-4,5-diones were obtained with yields of 70-83% by the acylation of thioamides (mostly arylthioamides) with oxalyl chloride in absolute acetone at -20°C [2, 8-10].



**1**

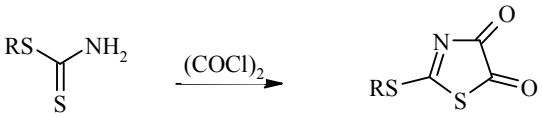
R = Ph, 4-$MeC_6H_4$ [2], 4-$ClC_6H_4$, 4-$O_2NC_6H_4$, 4-$MeOC_6H_4$, 4-$Me_2NC_6H_4$ [8], 2,4-$Me_2C_6H_3$, 2,4,6-$Me_3C_6H_2$ [9], $Ph_2CH$, Br, Ph(EtOCO)CH, $EtOCOCH_2$ [10]

The corresponding 2-*tert*-butyl-, 2-styryl-, and 2-(1-naphthyl)thiazoline-4,5-diones **5**-**7** were obtained by an analogous procedure from *tert*-butyl-, styryl-, and naphthylthioamides [8, 9].



**5** **6** **7**

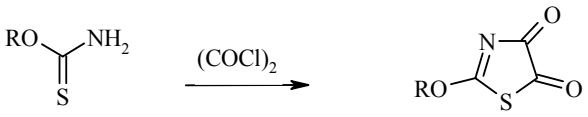
With oxalyl chloride in chloroform at -15 to -20°C the amides of dithiocarboxylic acids form 2-alkyl(aryl)mercaptothiazoline-4,5-diones **8** [11].



**8**

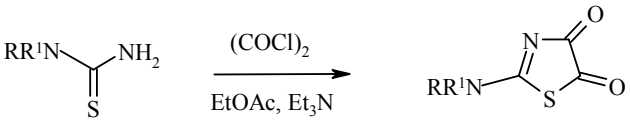
R (yield of **8**, %): Me (15), $PhCH_2$ (86), Ph (87)

During the production of 2-alkyl-, 2-phenyl-, and 2-cyclohexyloxythiazoline-4,5-diones **9** methylene chloride or dichloroethane was used instead of acetone. The reaction was conducted at 0°C [12].



R = Me, Et, Ph, *cyclo*-$C_6H_{11}$ **9**

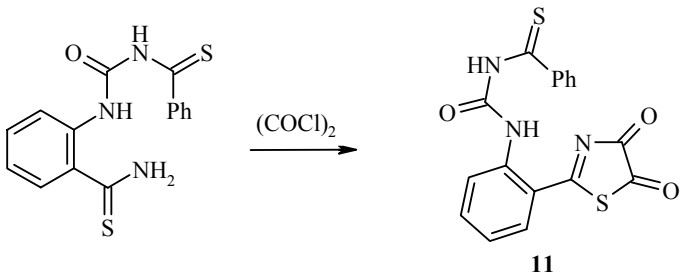
2-Amino-substituted thiazoline-4,5-diones **10** are obtained with good yields from N,N-disubstituted thioureas in anhydrous ethyl acetate in the presence of triethylamine at -30°C [12].



**10**

R, $R^1$ or $R+R^1$ (yield of 10, %): Me, Me (50); Et, Et (71); $(CH_2)_5$ (73); Me, Ph (73); Et, Ph (73); Bu, Ph (65); Ph, Ph (75)

Thiazoline-4,5-dione **11** was obtained with a yield of 69% from N-(2-thiocarbamoylphenyl)-N'-thiobenzoylurea under analogous conditions [13].

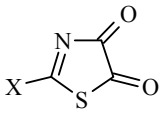



**11**

## 2. THE PHYSICAL CHARACTERISTICS OF THIAZOLINE-4,5-DIONES

The IR and UV spectra are the most informative for 2-substituted thiazoline-4,5-diones.

TABLE 1. The Spectral Characteristics of Some Thiazoline-4,5-diones

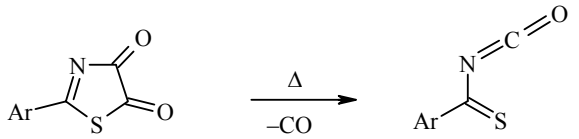


| Compound | X | IR spectrum, ν, $cm^{-1}$ (KBr) | UV spectrum, $\lambda_{max}$, nm (dioxane) | References |
|---|---|---|---|---|
| **1** | Ph | 1730, 1745 | 209, 304 | [8, 10] |
| | 4-$ClC_6H_4$ | 1726 | 310 | [8] |
| | 4-$MeOC_6H_4$ | 1722 | 350 | [8] |
| | 4-$Me_2NC_6H_4$ | 1694, 1722 | 450 | [8] |
| | 4-$O_2NC_6H_4$ | 1730 | 290 | [8] |
| **8** | MeS | — | 420 | [12] |
| **9** | MeO | 1725, 1785 | — | [12] |
| | EtO | 1725, 1785 | — | [12] |
| | PhO | 1725, 1785 | — | [12] |
| | *cyclo*-$C_6H_{11}O$ | 1725, 1785 | — | [12] |
| **10** | $Me_2N$ | 1705-1740 | — | [12] |
| | $Et_2N$ | 1705-1740 | — | [12] |
| | $(CH_2)_5N$ | 170-1740 | 325, 275 | [12] |
| | MePhN | 1705-1740 | — | [12] |
| | EtPhN | 1705-1740 | 325, 275 | [12] |
| | BuPhN | 1705-1740 | — | [12] |
| | $Ph_2N$ | — | 340 | [8] |

## 3. THE CHEMICAL CHARACTERISTICS OF THIAZOLINE-4,5-DIONES

### 3.1. Thermolysis with the Formation of Thioacyl Isocyanates

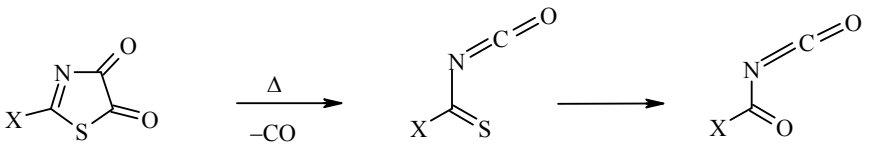
One of the characteristic properties of thiazoline-4,5-diones is the ability to eliminate carbon monoxide readily on heating to form the corresponding thioacyl isocyanates **2**. Thus, when boiled in toluene and also during vacuum sublimation or vacuum distillation the aryl-substituted compounds **1** are converted into the thioaroyl isocyanates **2**, which are unstable compounds that dimerize during storage [2, 8, 9].




**1** **2**

Ar = Ph, 4-$MeC_6H_4$ [2], 4-$ClC_6H_4$, 4-$O_2NC_6H_4$, 4-$MeOC_6H_4$, 4-$Me_2NC_6H_4$ [ 8], 2,4-$Me_2C_6H_3$, 2,4,6-$Me_3C_6H_2$ [9]

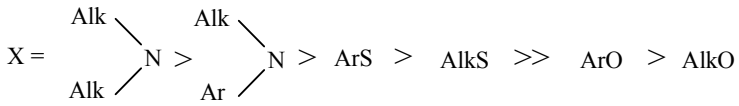
The most stable is thiobenzoyl isocyanate, a solution of which in toluene can be kept for up to two weeks [13]. When kept at room temperature the thioacyl isocyanates **12-14** obtained from the thiazolinediones **8-10** are susceptible to isomerization to the acyl isothiocyanates **15-17** [8, 11, 12].



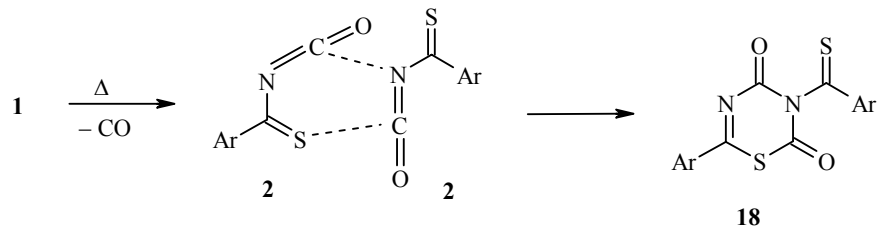

**8–10** **12–14** **15–17**

**8**, **12**, **15** X = RS; **9**, **13**, **16** X = RO; **10**, **14**, **17** X = $RR^1N$;
**8-10** R, $R^1$ see above (section 1)

The ability of the compounds **12-14** to isomerize depends of the substituent X and decreases in the following order:



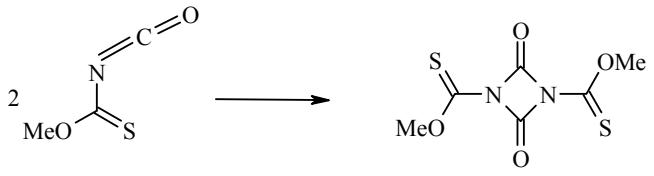

When the thiazolines **1** are heated in the range of 100-110°C without a solvent the obtained isocyanates **2** dimerize spontaneously to the substituted 1,3,5-thiadiazine-2,4-diones **18** [8].




**2** **2** **18**

Ar = Ph, 4-$ClC_6H_4$, 4-$MeOC_6H_4$, α-naphthyl, 4-$Me_2NC_6H_4$

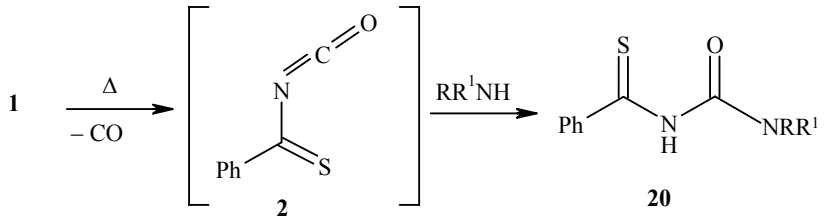
The dimerization of compounds **12-14** takes place similarly (without isomerization) except in the case of the 2-methoxythioacyl isocyanate **13** (X = MeO), which dimerizes with [2+2] cycloaddition to form 1,3-diazetidine-2,4-dione **19** [12].



**13** **19**

### 3.2. Thermolysis in the Presence of N-Nucleophiles

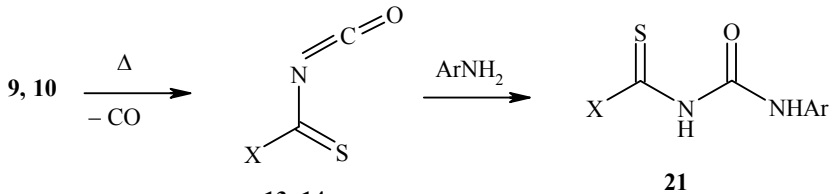
The transformations of thiazolinediones involving nucleophiles have been studied most. As mentioned above, they take place through the formation of intermediate isocyanates, which react with the N- or C-nucleophile *in situ*. Thus, when the thiazolinedione **1** (Ar = Ph) is heated with various primary or secondary amines the obtained thiobenzoyl isocyanate **2** enters into addition with these nucleophiles, leading to almost quantitative yields of the ureas **20** [13].



**2** **20**

R = H, $R^1$ = Bu, *cyclo*-$C_6H_{11}$, Ph, 4-$MeOC_6H_4$, 2-$O_2NC_6H_4$, 2,4-$(O_2N)_2C_6H_3$, 2-$NCC_6H_4$, 4-$H_2NC_6H_4$, 2-pyridyl, 3-Ph-1,2,4-thiadiazol-5-yl, PhCO, PhCONH, PhCH=N; R+$R^1$ = $(CH_2)_5$; R = $R^1$ = Ph

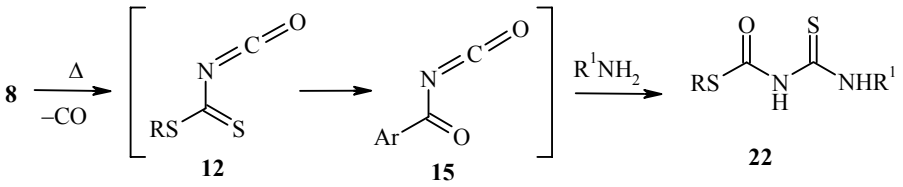
With the isocyanates **13** and **14**, obtained from the thiazolinediones **9** and **10**, aromatic amines form thioacylureas **21** [13].



**13, 14** **21**

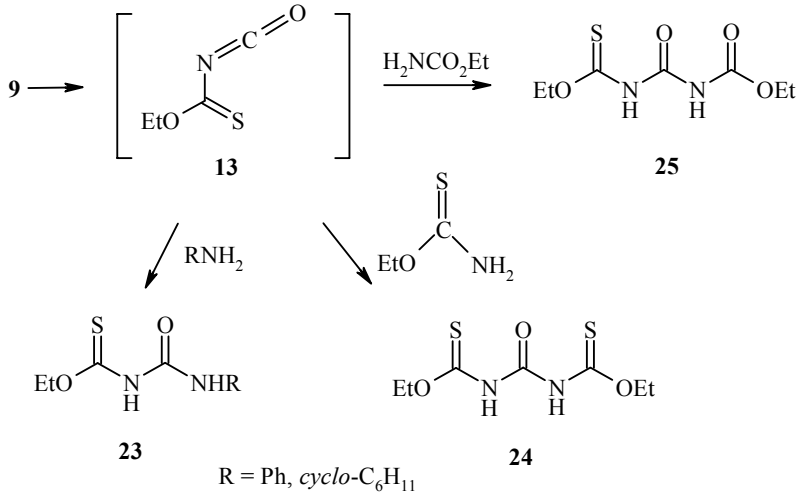
**21** Ar = Ph, 4-$O_2NC_6H_4$; X, see **13**, **14** (section 3.1)

Thermolysis of the thiazolinediones **8** in the presence of aniline and some of its 4-substituted derivatives leads not to ureas but to thioureas **22** on account of the ease of rearrangement of the intermediate mercaptothiocarbonyl isocyanates **12** to the mercaptocarbonyl isothiocyanates **15** [11].



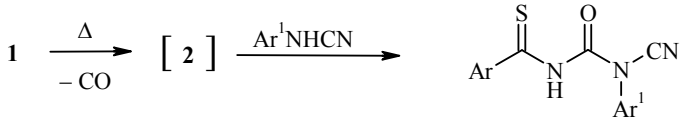
$R^1$ = Ph, 4-$MeOC_6H_4$, 4-$O_2NC_6H_4$, 4-$NCC_6H_4$

The ethoxycarbonyl isocyanate **13**, formed from the dione **9**, reacts with aniline, cyclohexylamine, thiopropionamide, and ethyl carbamate with the formation of the corresponding ureas **23**-**25** [14].



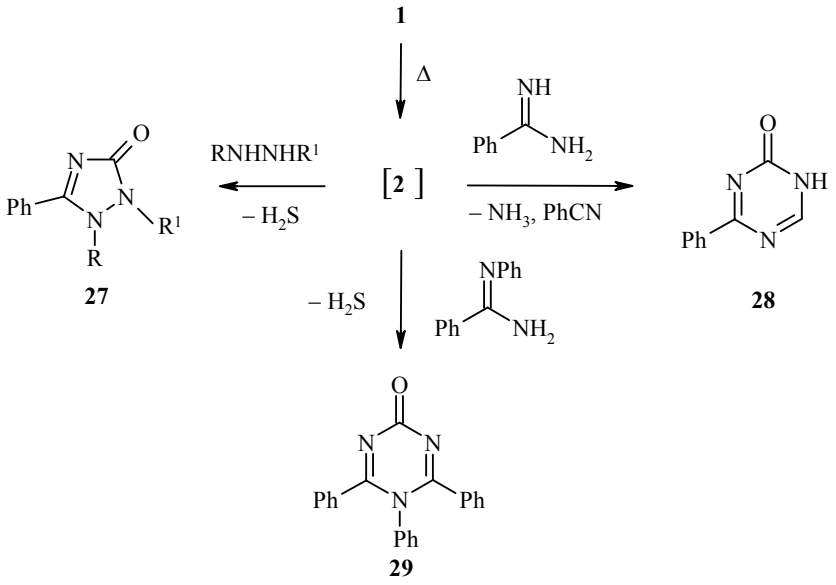
R = Ph, *cyclo*-$C_6H_{11}$

It is interesting to note that arylcyanamides react with thioaroyl isocyanates not through the cyano group, but through the amino group to form compounds **26** [15].



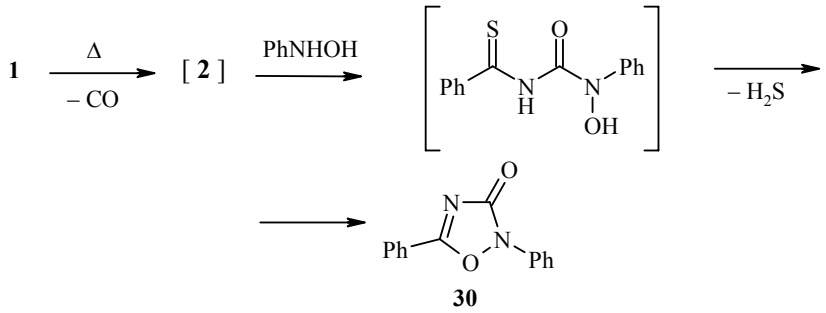
Ar = Ph, 4-$MeC_6H_4$, $Ar^1$ = 2-$ClC_6H_4$

N,N'-Substituted hydrazines form 1,2,4-triazolones **27** when heated with thiazolinedione **1** (Ar = Ph). In the case of benzamidine and N-phenylbenzamidine the 1,3,5-triazinones **28** and **29** respectively were obtained [13]. Nucleophilic addition to the isocyanate **2** is accompanied by the release of $H_2S$ and cyclization in the case of hydrazines and N-phenylbenzamide and by the release of $H_2S$ and benzonitrile in the case of benzamidine.

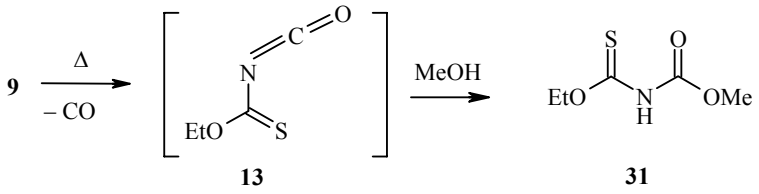


**27** R, $R^1$ = H, H; H, Ph; Ph, H; Ph, Ph

The addition of phenylhydroxylamine to the isocyanate **2** is also accompanied by the formation of the cyclic product **30** with the release of $H_2S$ [16].

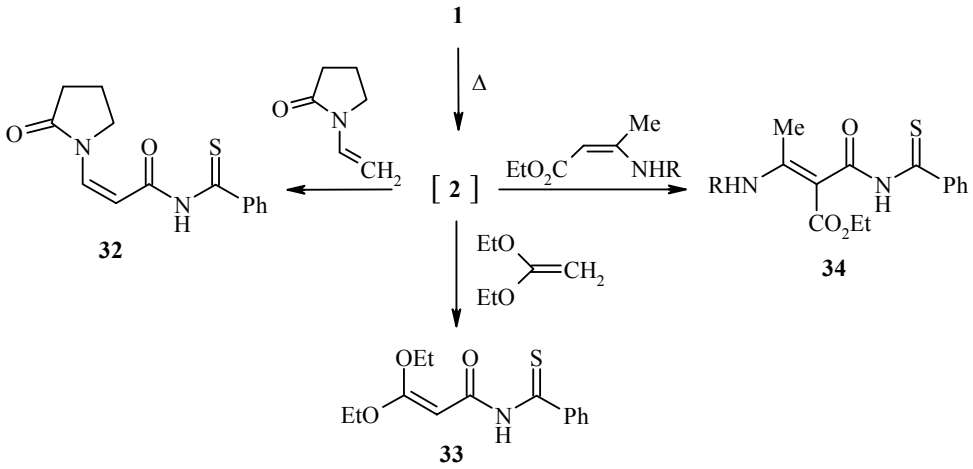


When boiled in methanol the ethoxy-substituted dione **9** only gives the nucleophilic addition product **31** with a quantitative yield [14].



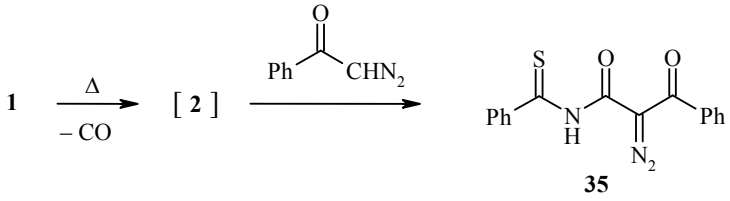
### 3.3. Thermolysis in the Presence of C-Nucleophiles

Addition products **32-34** are also formed during the thermolysis of thiazolinedione **1** (Ar = Ph) in the presence of the C-nucleophiles indicated in the scheme below [17].

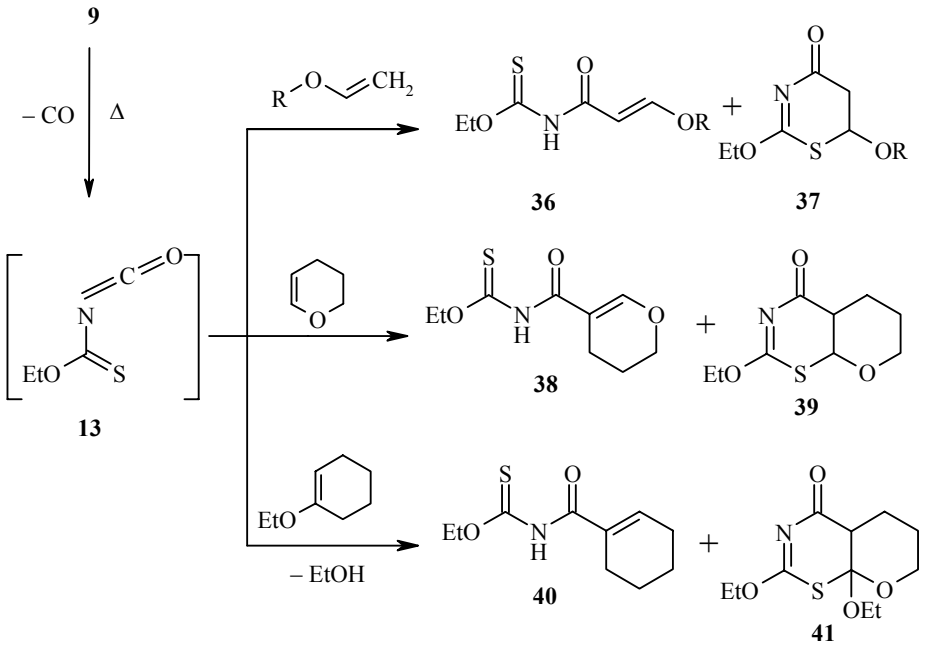


**34** R = H, Me, *cyclo*-$C_6H_{11}$

It is interesting that diazoacetophenone plays the role of C-nucleophile during the reaction with the isocyanate **2** (Ar = Ph) and reacts with the formation of compound **35** [16].

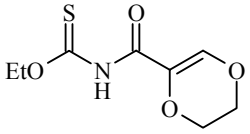


Ethyl and butyl vinyl ethers, 3,4-dihydropyran, and 1-ethoxycyclohexene react with ethoxydithiocarbonyl isocyanate **13** simultaneously as CH nucleophiles and dienophiles with the formation of compounds **36**-**41** [18].

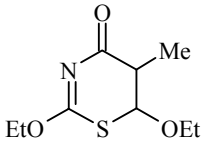


**36, 37** R = Et, Bu

In this reaction 2,3-dihydro-1,4-dioxin only forms the C-addition product **42**, while 1-ethoxy-2-methylethylene forms the [4+2] cycloaddition product **43** [18].

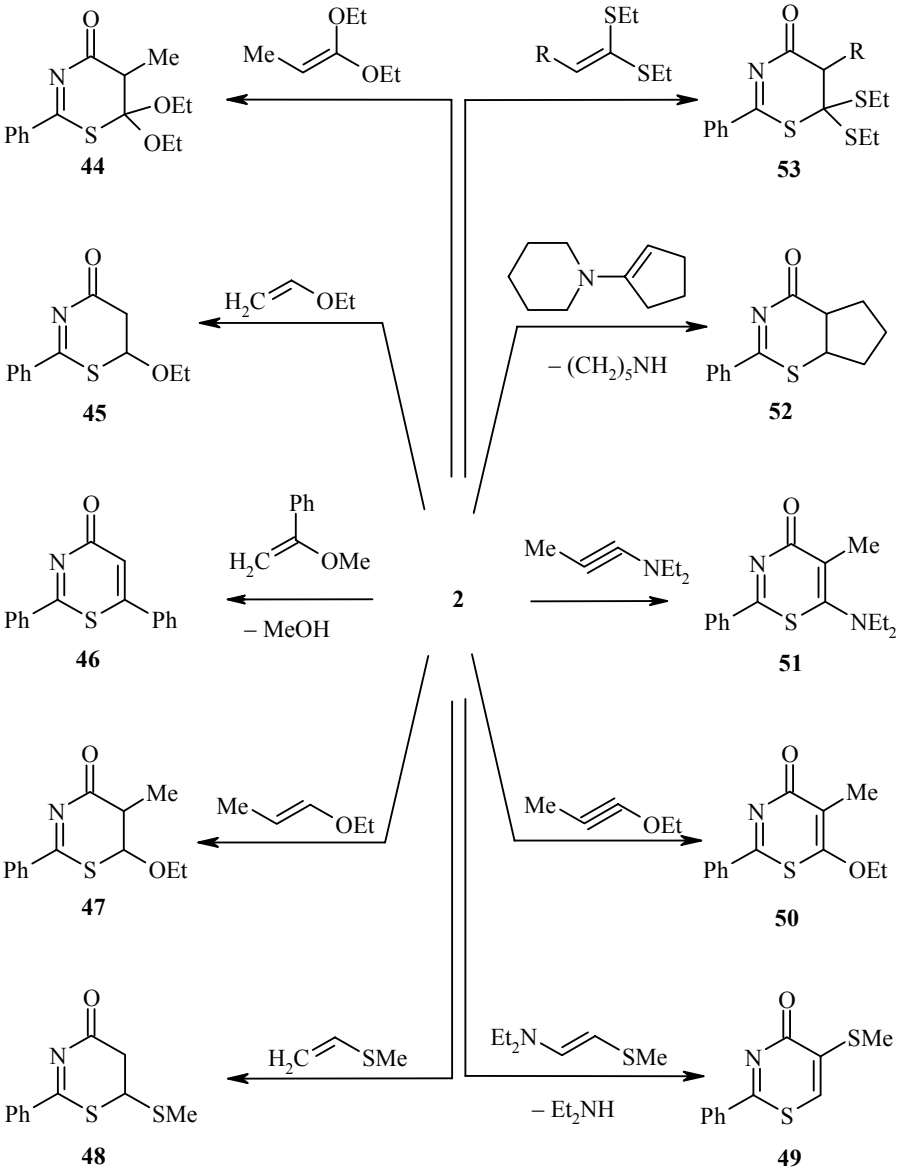


**42**



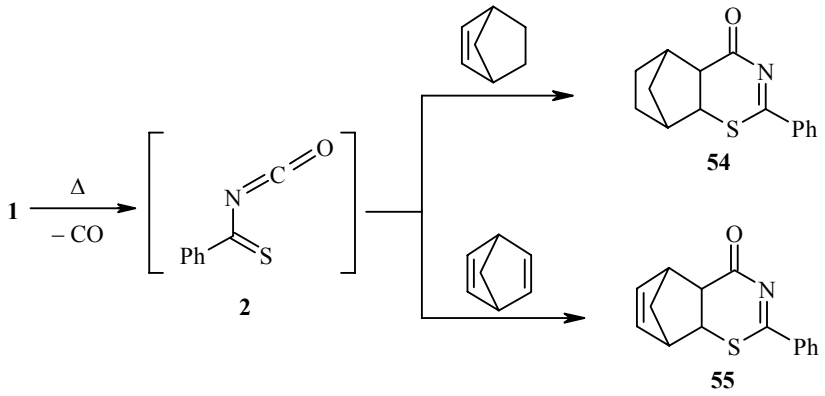
**43**

Numerous examples of the similar reaction of thiobenzoyl isocyanate **2** with vinyl ethers and sulfides and also with substituted acetylenes, leading to the cyclic products **44-53**, are described in [17, 18].

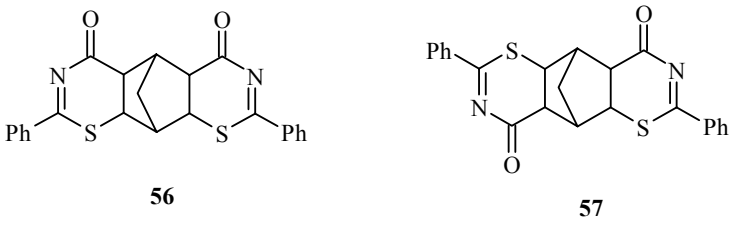



**53** R = H, Me

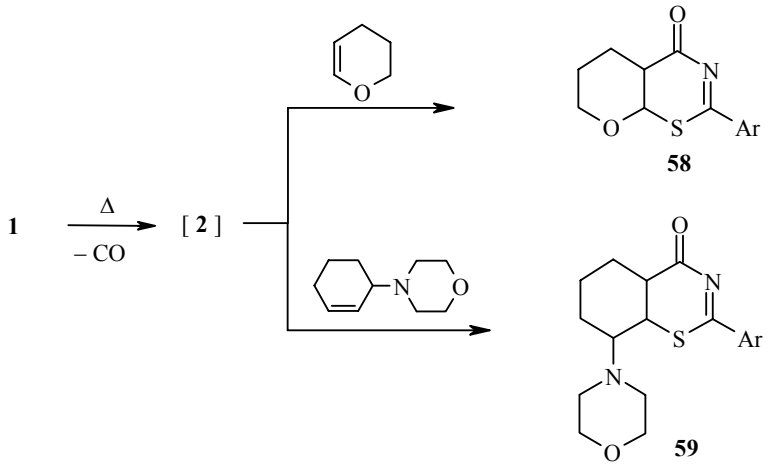
Norbornene and norbornadiene form the cycloadducts **54** [9, 20] and **55** [20] respectively.



With a twofold excess of norbornadiene the isomers **56** and **57** are obtained [20].



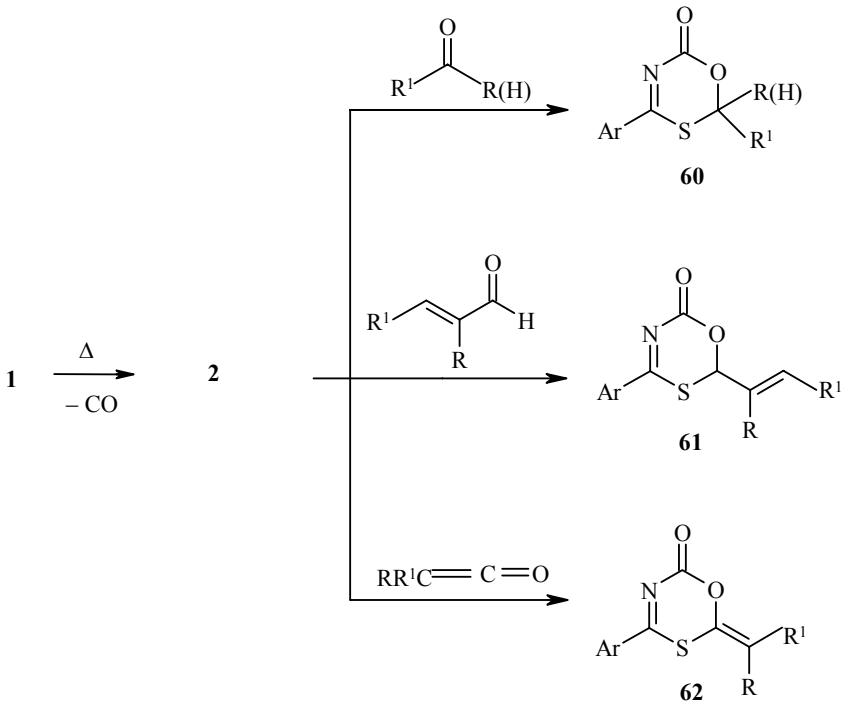
Dihydropyran and morpholinocyclohexene enter similarly into [4+2] cycloaddition with thioaroyl isocyanates **2** with the formation of thiazinones **58** [14] and **59** [9] respectively.



Ar = Ph, 4-$ClC_6H_4$, 4-$MeOC_6H_4$

### 3.4. Thermolysis in the Presence of Heterodienophiles (Cycloaddition at C=O, C=N, and C≡N Bonds)

During the thermolysis of arylthiazolinediones **1** in the presence of carbonyl compounds (aldehydes or ketones) the C=O group of the neighbors acts as dienophile in reaction with the obtained isocyanate **2**. The products are 1,3,5-oxathiazinones **60** [21]. Unsaturated aldehydes [22, 23] and also ketenes [24] react with isocyanates **2** similarly at the C=O bond, leading to 1,3,5-oxathiazinones **61** and **62** respectively.

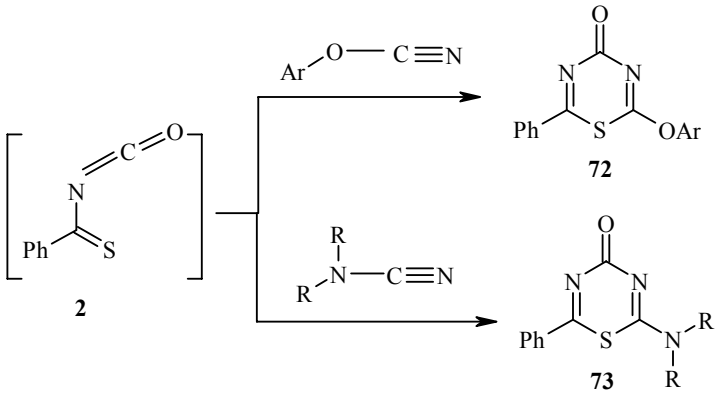


**60** R = H, $R^1$ = Et, Ph, 4-$MeOC_6H_4$, 4-$Me_2NC_6H_4$, 4-$O_2NC_6H_4$; R = Me; $R^1$ = Me, Ph; R = Ph, $R^1$ = Ph, PhCO; **61** R = H, Me, $R^1$ = H, Ph; **62** R = 4-$MeC_6H_4$, 4-$MeOC_6H_4$, $R^1$ = Me, Ph

The Diels–Alder heteroreaction of thioaroyl isocyanates **2** at the C=O bond of azomethines [9, 12, 25-28], ketene imines [24], azines [28], isocyanates [29], and 4,5-dihydro-1,3-thiazoles [30] leads to the formation of 1,3,5-thiadiazinones **63-71** (Scheme 1).

In the case of benzaldazines the products **66** are obtained if the isocyanate–azine ratio is 1:1, while compound **65** is obtained if a twofold excess of the isocyanate is used.

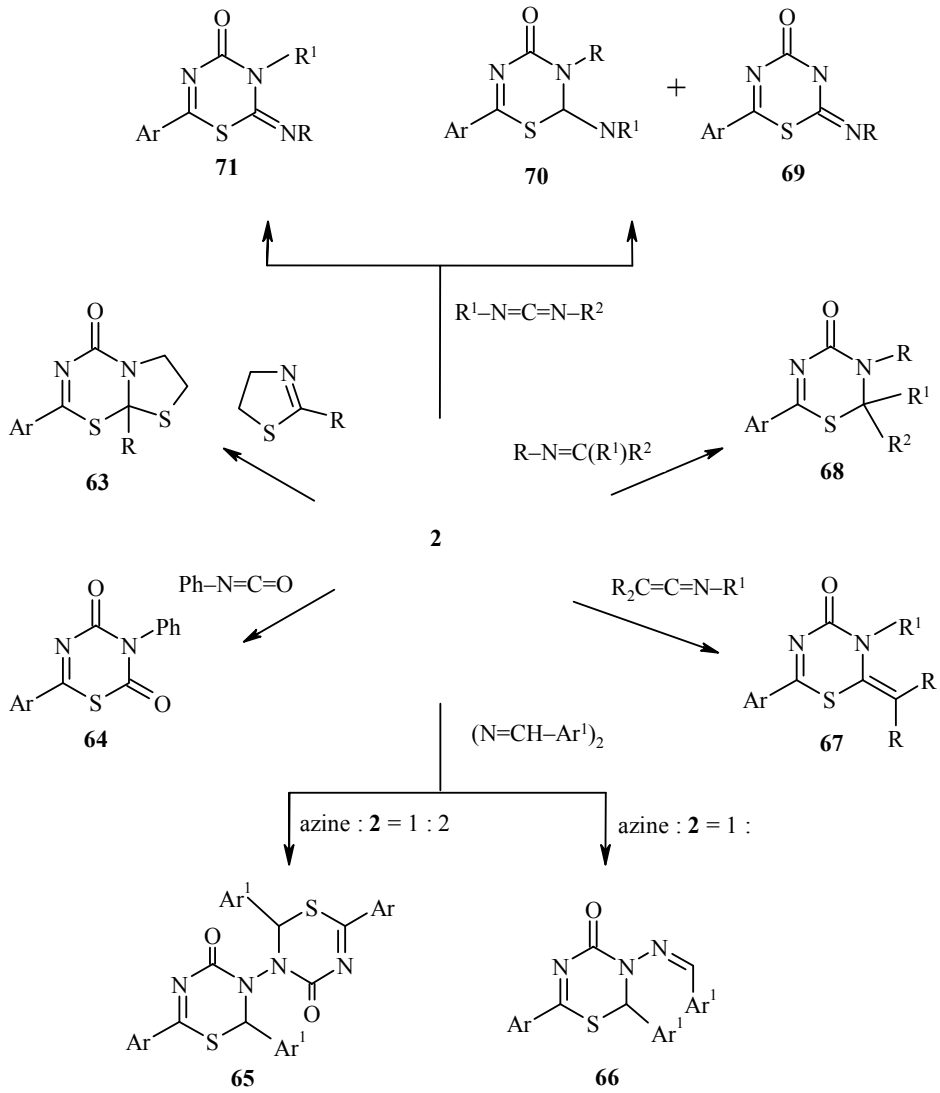
If there are strong electron-donating groups in the reagent the C≡N bond takes part in cycloaddition with the thioaroyl isocyanates. The esters of cyanic acid and disubstituted cyanamides were used in the reaction. The corresponding 1,3,5-thiadiazinones **72** and **73** were obtained with yields of 67-83% [31].



**72** Ar = Ph, 4-$MeOC_6H_4$, 4-$O_2NC_6H_4$; **73** R = Me, Ph

It should be noted that, unlike dialkylcyanamides, arylcyanamides behave like N-nucleophiles in this reaction (see section 3.2).

Scheme 1

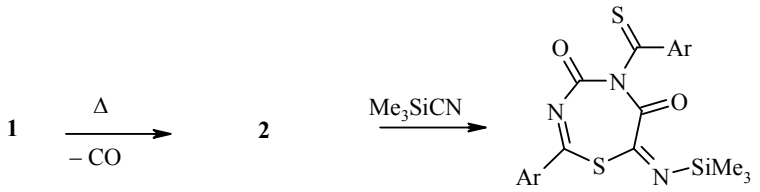


**2**, **63-71** Ar = Ph, 4-$ClC_6H_4$, 4-$MeOC_6H_4$, 4-$Me_2NC_6H_4$, α-naphthyl; **63** R = H, Me; **65**, **66** $Ar^1$ = Ph, 4-$MeC_6H_4$, 4-$ClC_6H_4$; **67** R = Me, Ph; $R^1$ = Me, 4-$MeC_6H_4$, 4-$MeOC_6H_4$; **68** R = Ph, 4-$MeOC_6H_4$, *cyclo*-$C_6H_{11}$, $R^1$ = H, Me, Ph, 4-$MeOC_6H_4$, 4-$O_2NC_6H_4$, $R^2$ = H, Me, Ph; **69**, **70** R = *cyclo*-$C_6H_{11}$, $R^1$ = Ph; **71** R = $R^1$ = *cyclo*-$C_6H_{11}$

The C≡N bond of thiocyanates, di(alkoxycarbonyl)cyanamides, and halogen cyanides does not enter into cycloaddition with the isocyanate **2** [31].

### 3.5. Other Reactions

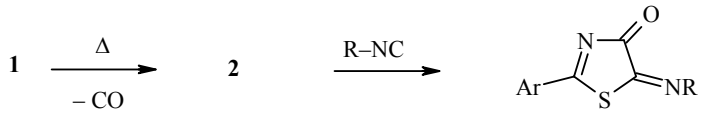
During the thermolysis of diones of type **1** in the presence of trimethylsilyl cyanide products **74**, containing two molecules of the intermediate isocyanate **2** and a molecule of the nitrile, are formed [32].



**74**

Ar = Ph, 4-$ClC_6H_4$, 4-$MeOC_6H_4$

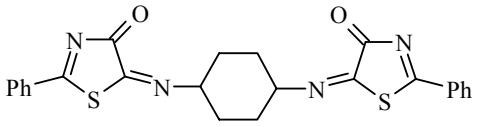
In reactions with thiazolinediones **1** isonitriles form 2-aryl-5-iminothiazolin-4-ones **75** [19, 29, 33].



**75**

Ar = R = Ph; Ar = Ph, R = 4-$MeOC_6H_4$, *cyclo*-$C_6H_4$;
Ar = α-$C_{10}H_7$, R = 4-$MeOC_6H_4$, *cyclo*-$C_6H_4$

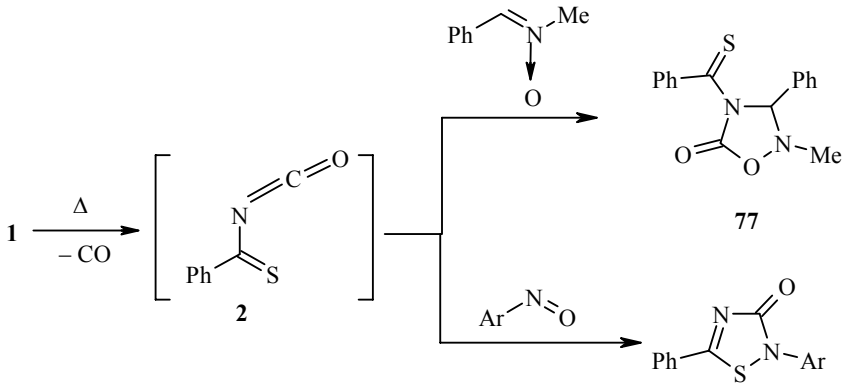
In the case of 1,4-cyclohexanediisonitrile the two C=N groups participate in the analogous reaction with the dione **1**, leading to the product **76** [34].



**76**

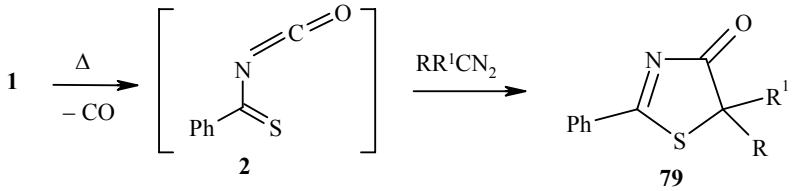
Each isonitrile group reacts with the intermediate isocyanate **2** by a mechanism of the [1+4] cycloaddition type.

C-Phenyl-N-methyl nitrone enters into [2+3] cycloaddition with phenylthiazolinedione with the formation of the 1,2,4-oxadiazolone **77**. In the reaction with nitrosobenzene 1,2,4-thiadiazolone **78** is formed [16].



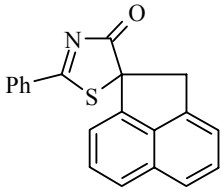
**78** Ar = Ph, 4-$ClC_6H_4$

The thermolysis of the diones **1** in the presence of diazoalkanes leads to derivatives of 4-thiazolone **79** [16].



**79** R = H, Me, Ph; $R^1$ = Ph; R + $R^1$ = 2,2'-biphenylylene

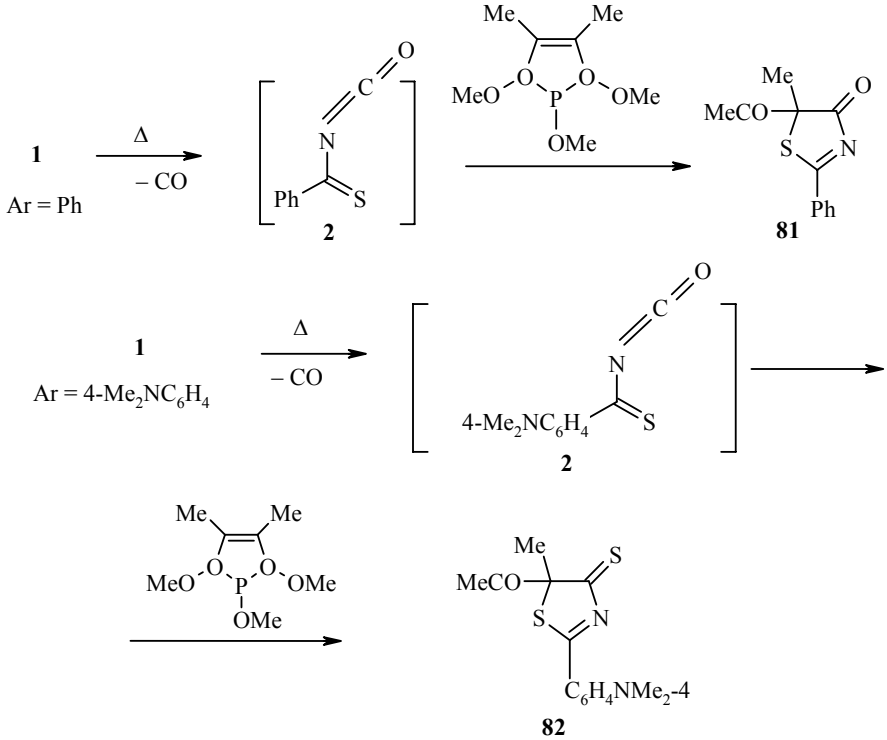
In the case of phenylthiazolinedione **1** and diazoacenaphthene the spiro compound **80** is formed [35].



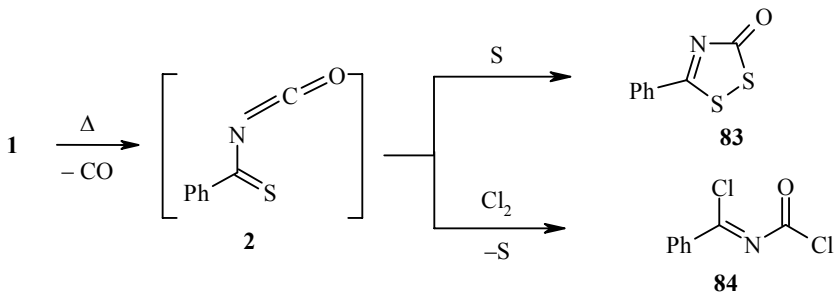
**80**

It was shown above that diazoacetophenone under the same conditions reacts as a C-nucleophile (see section 3.3).

The reaction of thiazolinediones **1** with 2,2,2-trimethoxy-4,5-dimethyl-2,2-dihydro-1,3,2-dioxaphospholine is affected by substituents at the second position of the thiazoline ring. The regioisomer **81** is formed with a phenyl substituent, and **82** with 4-dimethylaminophenyl [36].

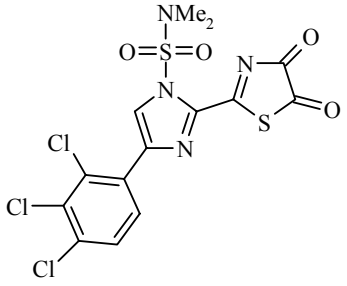


With sulfur [31] and chlorine [37] the thiazolinedione **1** (Ar = Ph) gives compounds **83** and **84** respectively.

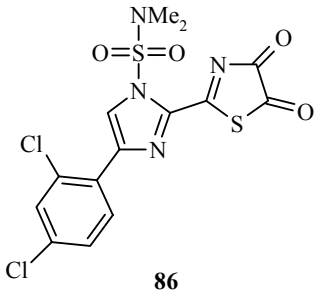



### 3.6. Some Aspects of the Practical Application of Thiazoline-4,5-diones

2-Substituted thiazoline-4,5-diones have been used as starting reagents in the synthesis of: a) 2-imino-4-thio(oxo)-5-polycyclovinylazolines, which are inhibitors of kinase P13 [38]; b) sulfur-containing amides of carboxylic acids, having hypotensive activity [39]; c) phenylalkylaminoalkoxyheteryl compounds with an antiischemic effect [40]. In addition to this, thiazolinediones themselves exhibit biological activity. For example, compounds **85** and **86** are fungicides [41].

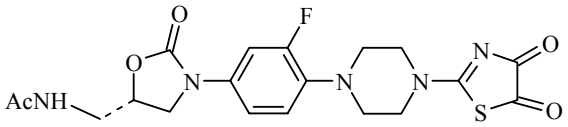


**85**



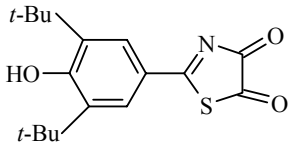
**86**

Bactericidal activity was discovered in the thiazoline-4,5-dione **87** [42].



**87**

Compound **88** exhibits anticonvulsive activity [43].



**88**

Thus, the published data on the properties of 2-substituted thiazoline-4,5-diones indicate that they may find use as initial reagents in the synthesis of heterocyclic compounds, including some that are biologically active [44-47].

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