Synthesis and reactivity of monothiooxamides and thiohydrazides of oxamic acids

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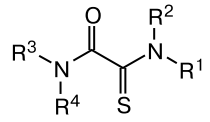
The review surveys the studies carried out at the Laboratory of Heterocyclic Compounds of the N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, dealing with the chemistry of monothiooxamides and thiohydrazides of oxamic acids. Convenient methods for the synthesis of the title compounds are described and the possibility of converting them into diverse products, including N- and S-containing heterocycles, is demonstrated.

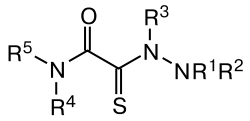
Key words: monothiooxamides, thiohydrazides of oxamic acids, amines, sulfur, chloroacetamides, heterocyclic compounds.

Introduction

Oxamic acid monothiooxamides (**A**) were first prepared in the late 19th century, while thiohydrazides (**B**) have been known since the second half of the 20th century.



A



B

However, intensive development of the chemistry of these derivatives started only in the 1990s. The interest in this class of compound is due to their unique complexing properties[1—4] and the possibility of synthesizing diverse compounds, including heterocyclic ones, based on them.[5,6] Monothiooxamide fragments have also been found in natural products.[7—10] Active studies of the biological properties of these compounds are currently in progress.[11—15]

The monothiooxamides and thiohydrazides of oxamic acids contain proximate amide and thioamide or thiohydrazide groups within one molecule. In some cases, this combination imparts unexpected properties to the molecules and allows one to go beyond the scope of the traditional synthetic potential stipulated by known properties of these groups.

The difference between the reactivities of amide and thioamide fragments provides the possibility of performing regioselective reactions with successive involvement of these groups; this opens up the route to a diversity of products. Study of the structures,[16—19] reactivities, and physicochemical properties of these polyfunctional compounds is of indubitable theoretical interest.

This review is the first attempt to describe systematically and analyze the published and authors´ data on the chemistry of monothiooxamides and thiohydrazides of oxamic acids.

1. Methods for the synthesis of oxamic acid monothiooxamides and thiohydrazides

Among the methods reported in the literature[20—22] and their numerous modifications, the method of monothiooxamide synthesis based on the reaction of substituted amides, mainly α-chloroacetamides, with elemental sulfur and amines is the most facile and convenient one.[23,24] However, this method, in which the reactants are mixed simultaneously and which is exceptionally simple from the preparative standpoint, still suffers from a serious drawback, in particular, it requires a long-term heating of the reaction mixture during which sulfur reacts with amines to give a complex mixture of products and this complicates the preparation and isolation of monothiooxamides. Thus we have shown that simultaneous addition of elemental sulfur and amine to chloroacetamide **1** results in α-aminoacetamides **2**, which further react with sulfur only on long-term heating giving monothiooxamides **3**, as a rule, in relatively low yields[24] (Scheme 1).

An exception to this rule is the reaction of quinoxalinone **4** with elemental sulfur (Scheme 2), after which thioxodihydroquinoxalinone **5** was isolated in a good yield.[25]

Our systematic research into S-functionalization of α-chloroacetamides has shown[26,27] that monothioox-

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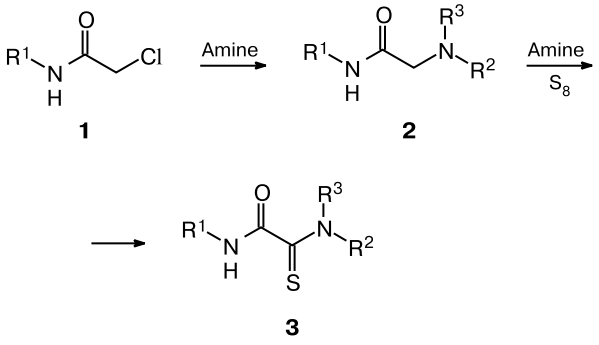
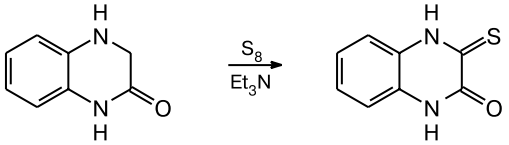
Scheme 1



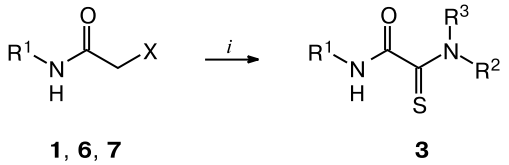
R^1 = Ar, R^2, R^3 = H, Ar

Scheme 2



amides can be prepared in high yields under mild conditions at room temperature by using a preliminarily prepared solution of elemental sulfur in amines. The effects of solvents, the nature of the α-substituents in amides, and the substituents in the "acetamide" and "amine" components on the course of the reaction carried out with a solution of elemental sulfur in amines was studied.[28] It was found[28] that S-functionalization of chloroacetamides **1** or their pyridinium derivatives **6** [29] proceeds most smoothly, while for other substituted acetamides, for example for **7**, the yields of monothiooxamides are markedly lower (Scheme 3).

Scheme 3

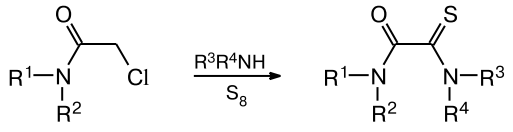


Reagents and conditions: *i*. Amine, S_8.

X = Cl (**1**); —N⁺ Cl⁻ (**6**);
—SPh, —$SCONH_2$, —$SSCH_2CONH_2$ (**7**)

The method is general; it allows one to synthesize a diversity of monothiooxamides containing aliphatic, aromatic, and heterocyclic substituents. By changing the "amine" and "acetamide" components, one can prepare monothiooxamide isomers (Scheme 4).

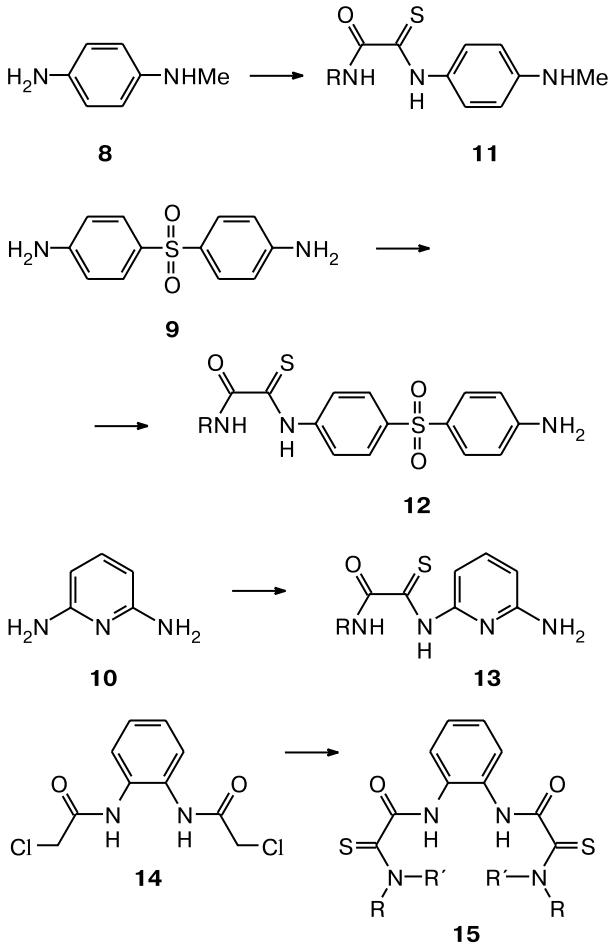
Scheme 4



R^1, R^2, R^3, R^4 = Alk, Ar, Het

When diamines **8—10** are used, the reaction involves only one amino group; hence, the amino group left in monothiooxamides **11—13** can be used for further transformations. Meanwhile, diamines can be readily converted into bis(chloroacetamides) **14** and then into the corresponding thiooxamides **15** (Scheme 5).[30]

Scheme 5

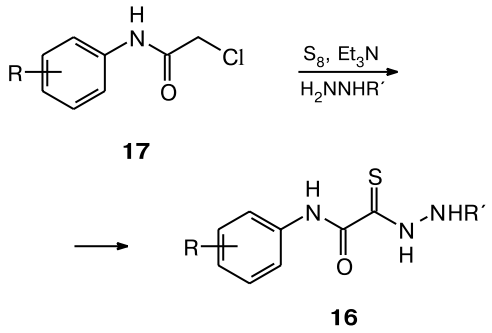


R, R´ = H, Ar

We have also shown[31] that oxamic acid thiohydrazides **16** are best prepared by the reaction of α-chloroacetamides

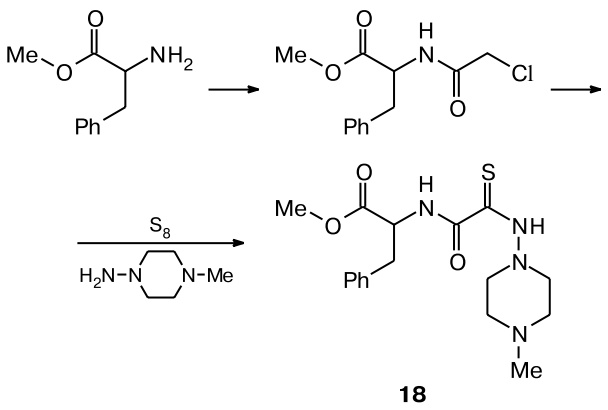
17 with elemental sulfur in the presence of hydrazines (Scheme 6); the product yields are 60—70%. Previously, this route has not been used to prepare thiohydrazides of oxamic acids.

Scheme 6

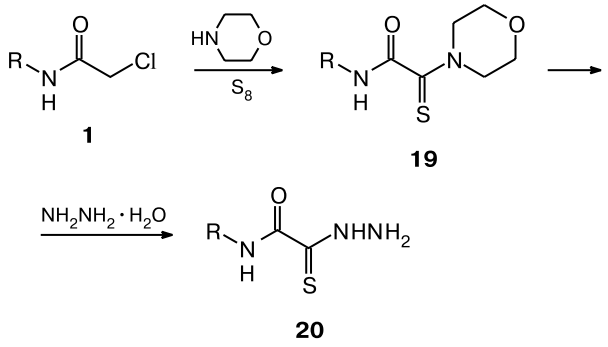


The method is versatile and, in particular, it permits the synthesis, of thiohydrazides (for example, **18**), containing an α-amino acid fragment (Scheme 7).

Scheme 7



Scheme 8



By using readily available morpholine derivatives **19**,[31] one can prepare oxamic acid thiohydrazides **20** with an unsubstituted thiohydrazide fragment (Scheme 8).

Evidently, the higher yields of oxamic acid monothiooxamides and thiohydrazides obtained in the reactions with solutions of elemental sulfur and amines or hydrazines prepared beforehand compared to those observed upon simultaneous addition of the reagents is due to the decrease in the probability of the side alkylation of amines or hydrazines with chloroacetamides.

2. Reactivity of oxamic acid monothiooxamides and thiohydrazides

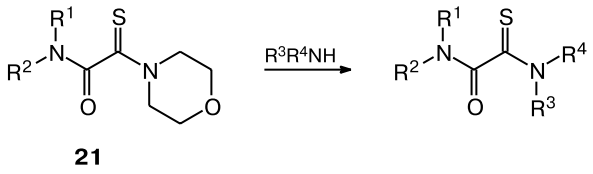
The chemical properties of monothiooxamides are determined by the difference between the reactivities of the thioamide and amide groups. Owing to the easy polarizability of the π-bond, the thiocarbonyl group reacts with nucleophiles and enters into electrophilic and radical reactions much more readily than the carbonyl group. The proximate location of the amide functions provides additional activation and allows conduction of the reactions under mild conditions. For example, thiobenzomorpholide does not react with amines or hydroxylamine even on long-term heating in ethanol or pyridine, whereas monothiooxamides readily undergo transamidation even at room temperature.[32] Presumably, the reactivity of these compounds seems to be largely dictated by steric rather than electronic effects of substituents.[33]

2.1. Reactions of monothiooxamides with nucleophiles resulting in the formation of "linear" products

The reactions of monothiooxamides with N-nucleophiles yield nitrogen-containing derivatives of oxalic acid; the use of reagents with two nucleophilic centers can be accompanied by the formation of heterocycles.

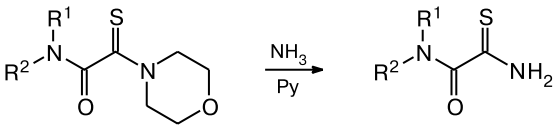
Most often, monothiooxamides react with primary and secondary amines under mild conditions, the reactions involving only the thioamide group.[32] Transamidation is widely used for the synthesis of new monothiooxamides from more readily available compounds (for example, from *N*(S)-morpholine derivatives **21**)[32] (Scheme 9).

Scheme 9



The process is usually carried out in the amine or DMF solvent. Compounds of type **22** with an unsubstituted thioamide group, which is readily transformed into heterocycles, are prepared by the reaction with ammonia in pyridine[34] (Scheme 10).

Scheme 10



21 **22**

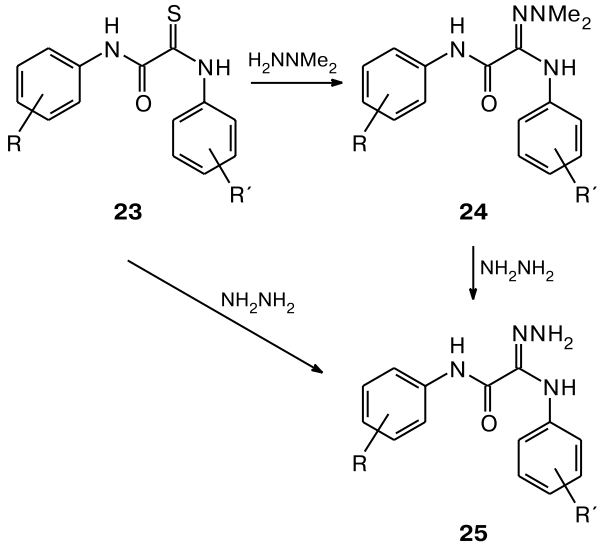
Monothiooxamides **23** smoothly react with hydrazines to give the corresponding amidrazones **24** and **25** [35] (Scheme 11).

In recent years, we have studied the reactions of monothiooxamides with hydroxylamines as this line of research proved promising in the synthesis of derivatives of various heterocycles, for example, 1,2,4-oxadiazoles,[36] furoxans, tetrazoles, 1,3,4-oxadiazoles, dihydroisoxazoles and isoxazoles,[37] and 1,2,4-triazoles.[38] Depending on the amount of hydroxylamine used, this reaction gives either amidoximes **26** or the products of further reduction **27** [39] (Scheme 12).

The above-mentioned products may be obtained *via* a one-pot procedure including the reaction of chloroacetamides **1** with sulfur and amine in pyridine followed by the addition of hydroxylamine hydrochloride without isolation of the intermediate monothiooxamide **19**.[39]

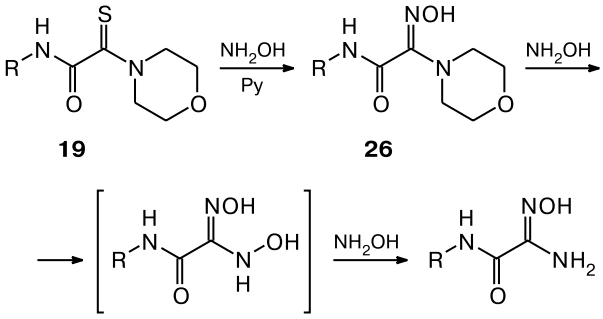
The reaction with *O*-methylhydroxylamine is not that unambiguous and is susceptible to the effect of substituents in the benzene ring (Scheme 13). The expected

Scheme 11



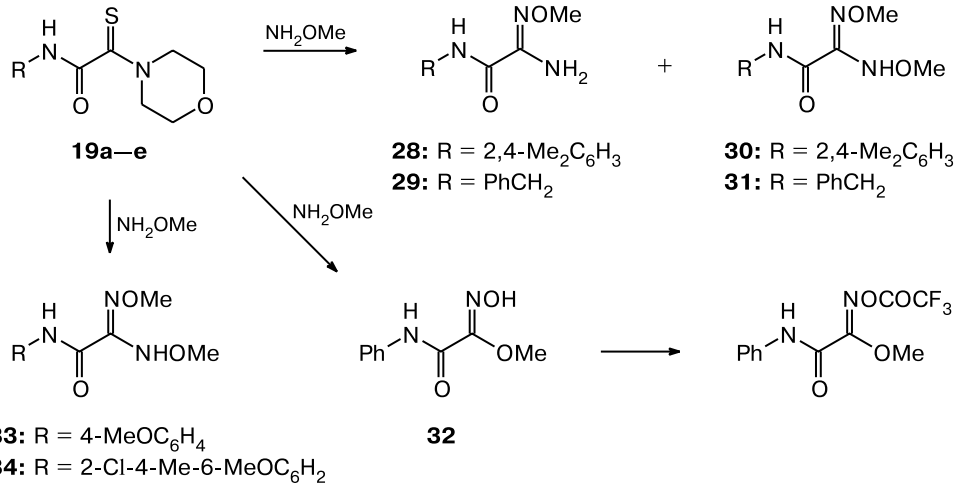
23 **24** **25**

Scheme 12



19 **26** **27**

Scheme 13



19a—e

28: R = 2,4-$Me_2C_6H_3$
29: R = $PhCH_2$

30: R = 2,4-$Me_2C_6H_3$
31: R = $PhCH_2$

33: R = 4-$MeOC_6H_4$
34: R = 2-Cl-4-Me-6-$MeOC_6H_2$

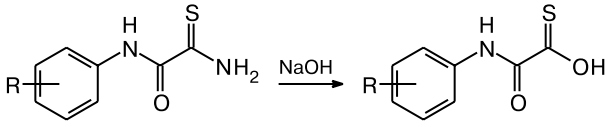
32

R = 2,4-$Me_2C_6H_3$ (**a**), $PhCH_2$ (**b**), Ph (**c**), 4-$MeOC_6H_4$ (**d**), 2-Cl-4-Me-6-$MeOC_6H_2$ (**e**)

N-methoxyamidines **28** or **29** are formed only in the case of monothiooxamides **19a** and **19b** (together with amidines **30** and **31**), whereas phenylmonothiooxamide **19c** affords methyl hydroxamate **32**. Ethoxyphenylmonothiooxamide **19d** and compound **19e**, containing a trisubstituted phenyl group, are converted into *N*,*N*′-bis(methoxyamidines) **33** and **34** [40,41] (Scheme 13).

In the presence of alkali, monothiooxamides **35** are saponified to thiooxanyl acids **36** [42] even at room temperature (Scheme 14).

Scheme 14

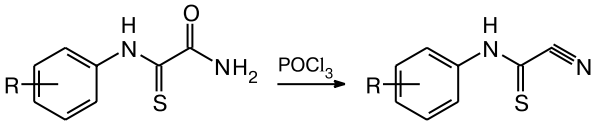


35 **36**

R = H, Alk, MeO, Hal

It is worthy of note that the reaction can involve not only selective modification of the more labile thioamide group, but also transformation of the amide group with retention of the thioamide fragment. For instance, on treatment with thionyl chloride, amide **37** is converted into nitrile **38** (Scheme 15)*.

Scheme 15



37 **38**

R = H, Alk, MeO, Hal

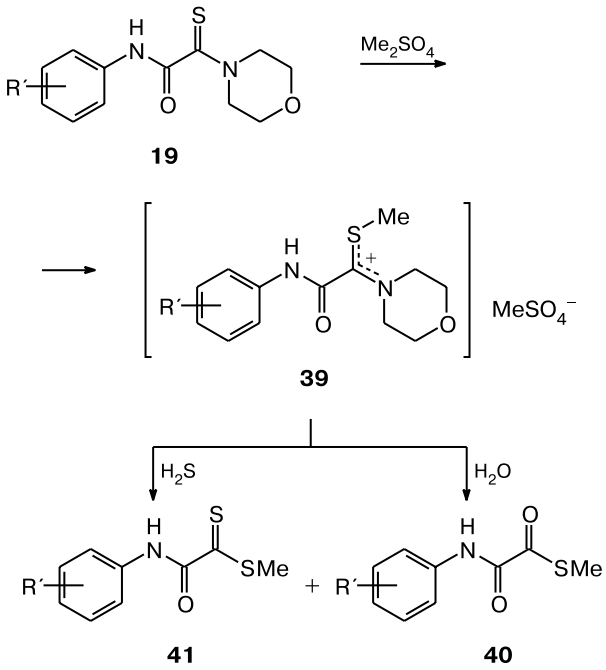
Monothiooxamides **19** are alkylated with dimethyl sulfate giving rise to salts **39**, which are easily hydrolyzed to give S-alkyl thiooxalates **40** and react with hydrogen sulfide to give dithiooxalates **41** [27] (Scheme 16).

2.2. Reactions of monothiooxamides resulting in nitrogen-containing heterocycles

Transamidation on treatment with diamines can be accompanied by a reaction involving the thiocarbonyl group, which can serve as a route to various heterocyclic compounds, in particular, dihydroimidazoles (**42**, $n = 1$) and tetrahydropyrimidines (**42**, $n = 2$)[32] (Scheme 17).

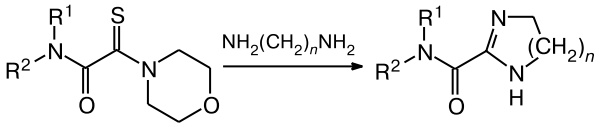
* Unpublished results.

Scheme 16



19 **39** **41** **40**

Scheme 17

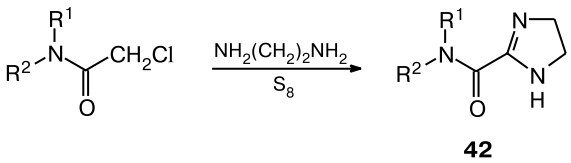


21 **42**

n = 1, 2

A one-step method for the synthesis of dihydroimidazoles by the reaction of chloroacetamide with a preliminarily prepared solution of elemental sulfur in ethylenediamine without isolation of morpholide **21** has been proposed.[32] Dihydroimidazoles are formed in high yields (Scheme 18).

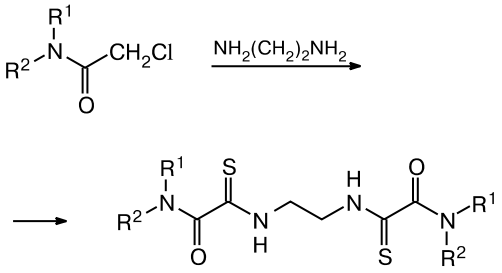
Scheme 18



42

The reaction outcome was found to depend on the order of mixing the reactants. If a solution of elemental sulfur in ethylenediamine is added to chloroacetamide, compound **43** is produced (Scheme 19).

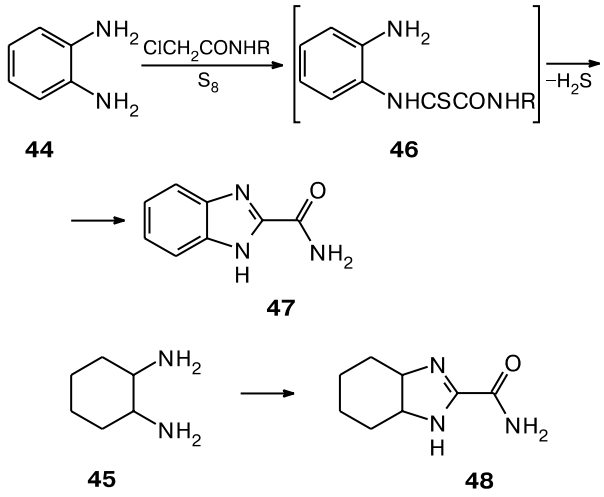
Scheme 19



43

The reactions of aromatic (**44**) and alicyclic (**45**) diamines with chloroacetamides proceed in a similar way. After the formation of monothiooxamide (of type **46**) at one amino group, further cyclization involving the thioamide fragment and the other amino group takes place at room temperature yielding amide **47** or **48**, respectively[27,30] (Scheme 20).

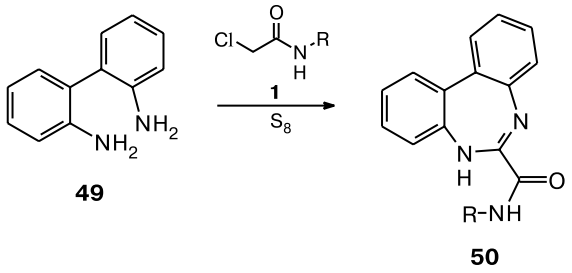
Scheme 20



44 **46** **47** **45** **48**

The reaction of 2,2′-diaminobiphenyl (**49**) with chloroacetamides is a route to diazepines **50** [43] (Scheme 21).

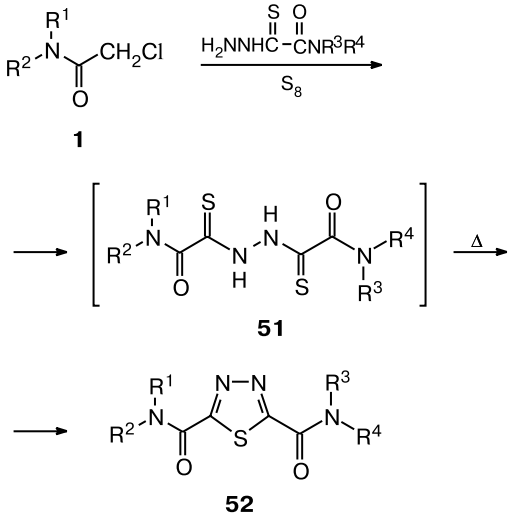
Scheme 21



49 **50**

The process occurs vigorously: indeed, it is even impossible to isolate the intermediate bis(monothiooxamides) **51**, because they rapidly cyclize to give bis(carbamoyl)-1,3,4-thiadiazoles **52** [35] (Scheme 22).

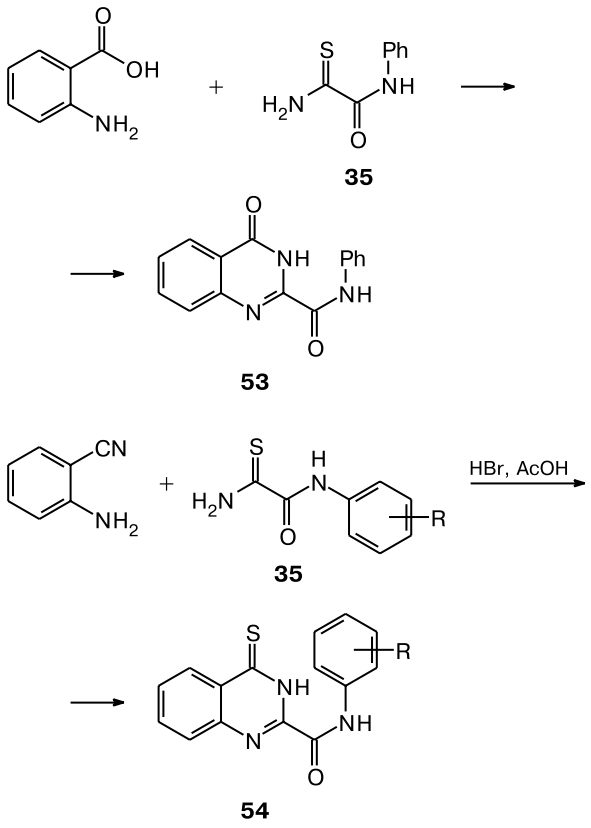
Scheme 22



1 **51** **52**

Given below are examples of effective use of monothiooxamide fragments and neighboring functional groups for constructing fused heterocyclic systems. Thus the re-

Scheme 23

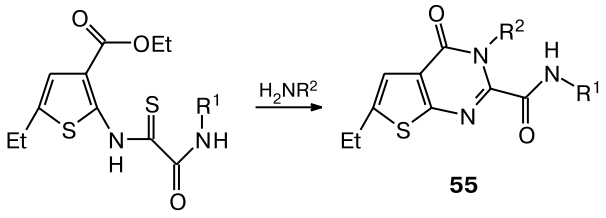


35 **53** **35** **54**

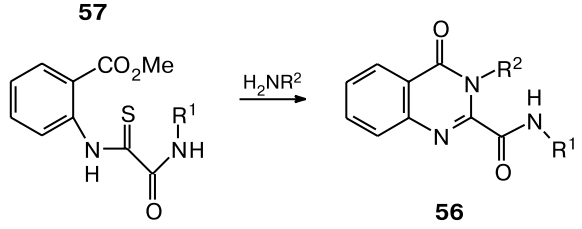
action of monothiooxamide **35** with anthranilic acid or *o*-aminobenzonitrile has resulted in the synthesis of quinazolines **53** and **54** [44] (Scheme 23).

A convenient route to thienopyrimidines **55** and quinazolines **56** containing various substituents at the nitrogen atom of the dihydropyrimidine ring[44] has been developed. The method makes use of accessible monothiooxamides **57** and **58** (Scheme 24).

Scheme 24



57 **55**

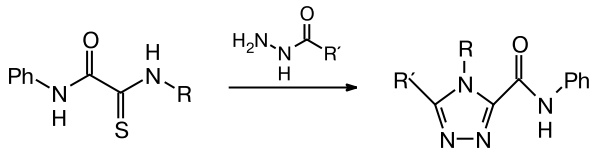


58 **56**

R^1 = H, Alk, Ar; R^2 = Alk, Ar, NH_2, NHR^1

Refluxing of monothiooxamides with hydrazides in pyridine provides a one-step synthesis of 3-carbamoyl-1,2,4-triazoles[38] (Scheme 25).

Scheme 25



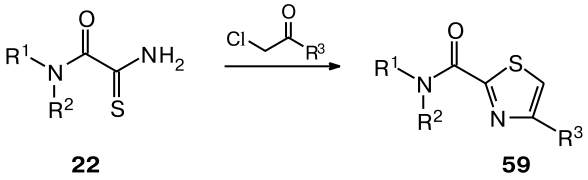
2.3. Synthesis of sulfur-containing heterocyclic compounds from oxamic acid monothiooxamides and thiohydrazides

2.3.1. Preparation of heterocycles from monothiooxamides

Thioamide groups in monothiooxamides are able to undergo heterocyclization with retention of the sulfur atom, which gives rise to sulfur-containing heterocyclic amides.

For example, the reaction of monothiooxamides **22** with α-haloketones furnishes thiazoles **59** [27] (Scheme 26).

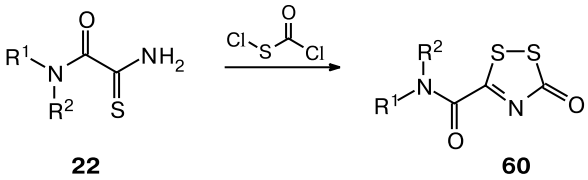
Scheme 26



22 **59**

Heating of monothiooxamides **22** with chlorocarbonylsulfenyl chloride in toluene affords 3-oxo-3*H*-1,2,4-dithiazoles **60** [34] (Scheme 27).

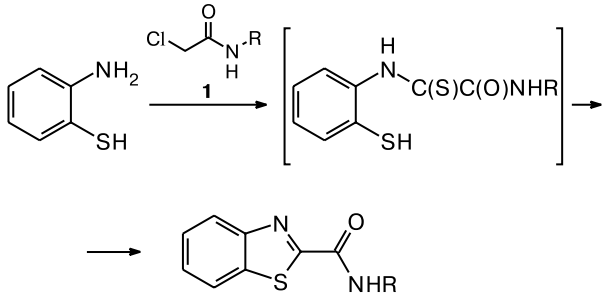
Scheme 27



22 **60**

The reaction of chloroacetamides **1** with *o*-mercaptoaniline gives benzothiazoles **61** [45] (Scheme 28).

Scheme 28

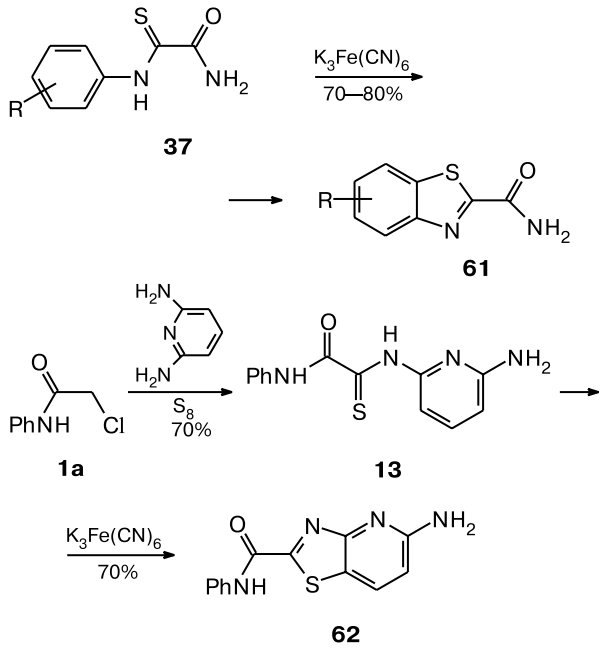


61

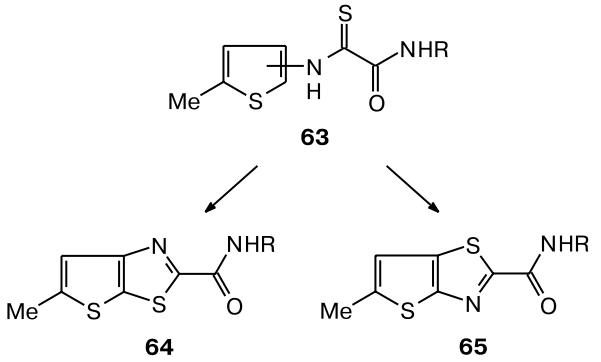
A method for the synthesis of 2-carbamoylbenzothiazoles **61**, **62** by oxidation of monothiooxamides with aromatic (**37**) or heterocyclic substituents (**13**) has been proposed (Scheme 29).[42]

Note that 2-carbamoylbenzothiazoles are key structures in the synthesis of a natural product luciferin and its analogs.[42,46] The step of preparation of benzothiazoles from monothiooxamides is also involved in the synthesis of 1,2-dihetarylethenes, which are currently under active research as photochromic systems for optoelectronics.[47—49]

Scheme 29



Scheme 30

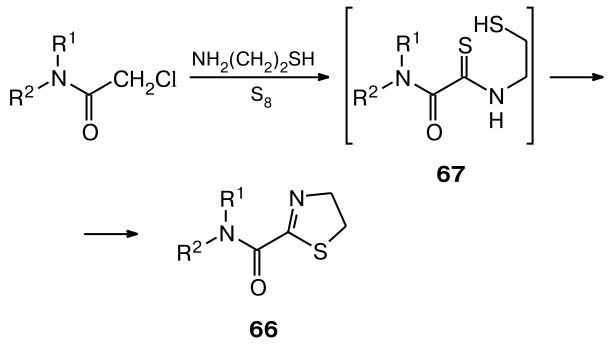


R = Alk, Ar

The method we discuss is general and suitable for preparing compounds with different structures. For example, cyclization of monothiooxamides of the thiophene series **63** in the presence of $K_3Fe(CN)_6$ smoothly proceeds at room temperature to give 2-carbamoylthieno[3,2-*d*]- (**64**) or [2,3-*d*]thiazoles (**65**)[50,51] (Scheme 30).

With mercaptoethanolamine introduced in the reaction with chloroacetamides, this approach serves as a smooth one-step route to difficultly accessible dihydrothiazoles[45] **66**; in some cases, it is possible to isolate the intermediate product **67** (Scheme 31).

Scheme 31



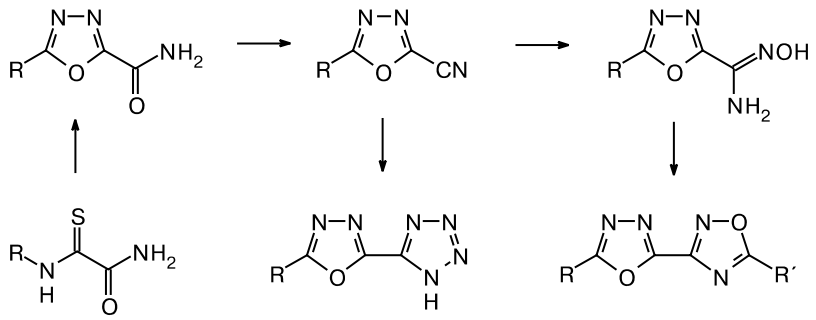
Due to the different reactivities of the thioamide and amide functions, they can be modified one after another to give mixed bisheterocycles.[52] Examples of such transformations are shown in Scheme 32, 33.

2.3.2. Preparation of heterocycles from oxamic acid thiohydrazides

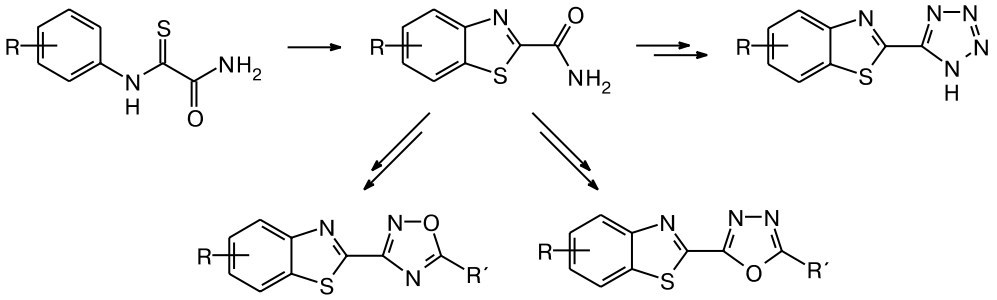
Methods for transforming the thiohydrazide fragment of oxamic acids into a 1,3,4-thiadiazole ring have now been thoroughly developed and the possibility of constructing other heterocyclic compounds has been demonstrated.

Heterocyclization of oxamic acid thiohydrazides on treatment with acid anhydrides or chlorides is the most widely used route to carbamoyl-1,3,4-thiadiazoles.[31] The synthesis of 2-haloalkyl-5-carbamoyl-1,3,4-thiadiazoles

Scheme 32

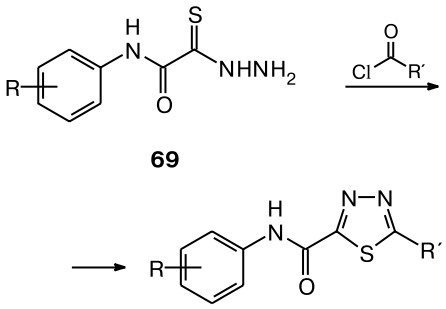


Scheme 33



68 by the reaction of oxamic acid thiohydrazides **69** with haloacyl chlorides under mild conditions has been described[27,31] (Scheme 34).

Scheme 34

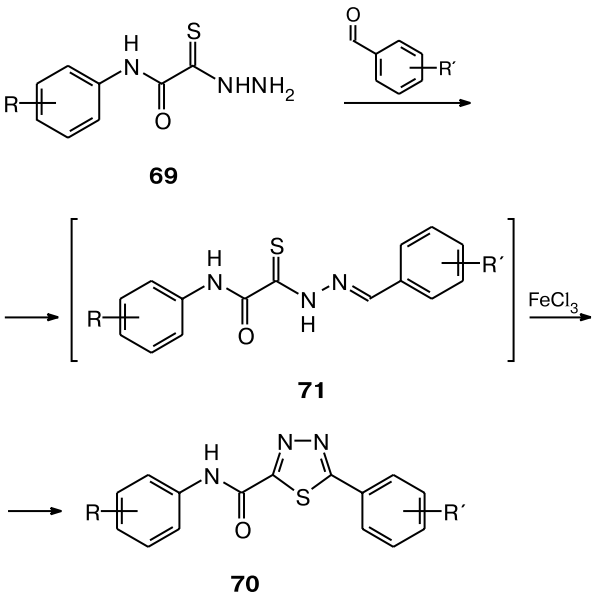


69

68

R′ = CH_2Cl, $CHCl_2$, CCl_3, CF_3

Scheme 35



69

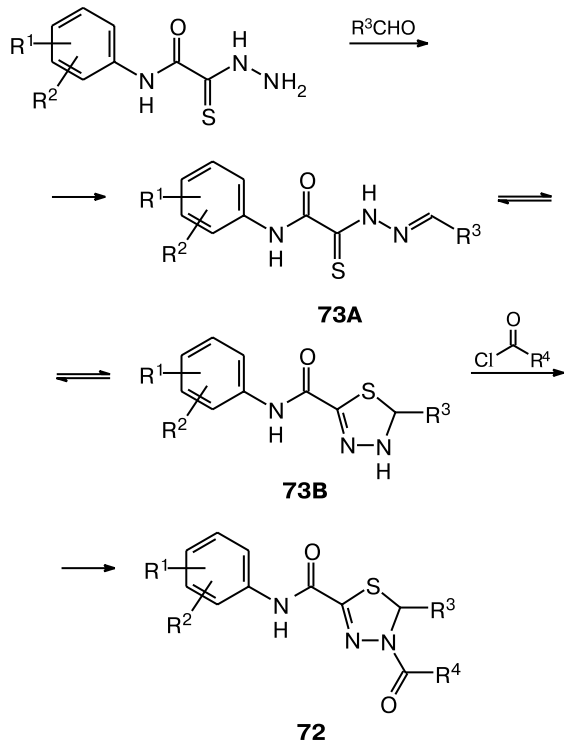
71

70

For the synthesis of 5-aryl-substituted thiadiazoles **70**, the following approach has proved more successful. Instead of benzoyl chlorides, the method includes the use of thiohydrazones **71**, prepared preliminarily from aromatic aldehydes, and subsequent oxidation with $FeCl_3$ [31] (Scheme 35).

Hydrazones smoothly react with aliphatic, aromatic, and heteroaromatic acyl chlorides giving rise to the corresponding 2-carbamoyl-4,5-dihydro-1,3,4-thiadiazoles **72**. An interesting feature of the hydrazones of oxamic acid thiohydrazides **73** deserves attention: they exist in solutions as two tautomers, a linear **A** and ring **B** ones. The reaction with chlorides involves apparently the ring tautomer **B** of the hydrazone[53] (Scheme 36).

Scheme 36



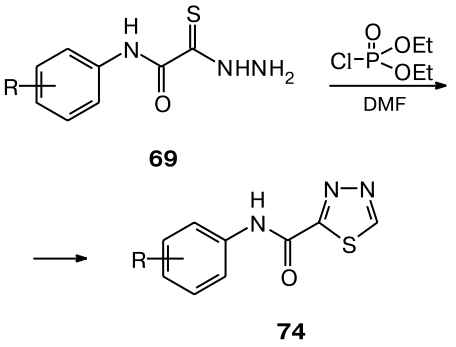
73A

73B

72

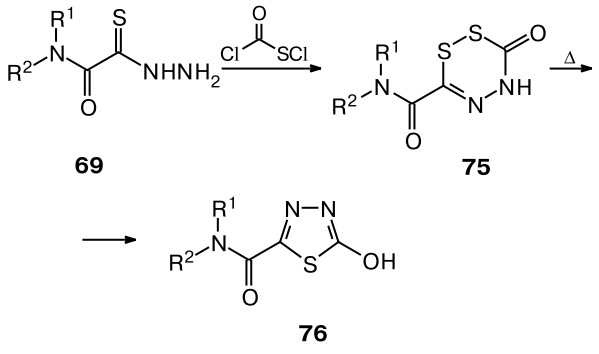
The synthesis of unsubstituted carbamoyl-1,3,4-thiadiazoles **74** in high yields by treatment of oxamic acid thiohydrazides **69** with a new cyclizing reagent, a solution of diethyl phosphorochloridate in DMF, under mild conditions has been reported. The attempts to prepare 5-unsubstituted carbamoyl-1,3,4-thiadiazoles using ethyl orthoformate or ethyl formate, which are usually employed in this type of reaction, proved unsuccessful[54] (Scheme 37).

Scheme 37



Apart from 1,3,4-thiadiazoles, it is possible to prepare six-membered sulfur-containing heterocyclic compounds. For instance, oxamic acid thiohydrazide **69** reacts with chlorocarbonylsulfenyl chloride at room temperature to give 6-carbamoyl-5,6-dihydro[1,2,4,5]dithiadiazin-3-one **75**, which readily speits off sulfur on heating being converted into 5-carbamoyl-2-hydroxy-1,3,4-thiadiazole **76**[34] (Scheme 38).

Scheme 38



Thus, the data surveyed in the review demonstrate the extensive synthetic potential of monothiooxamides and thiohydrazides of oxamic acids. These compounds can underlie the syntheses of diverse sulfur- and nitrogen-containing heterocyclic compounds.

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