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Organic Chemistry in Russian Universities. Achievements of Recent Years

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Abstract—An overview of the main scientific achievements of Russian universities in the field of organic chemistry over the period 2018–2023 is presented.

Keywords: organic chemistry, chemistry

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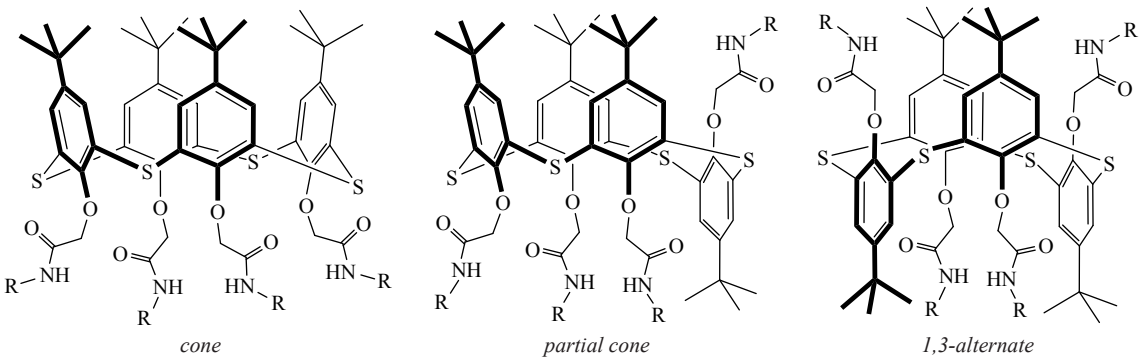
CONTENTS

INTRODUCTION

In 2018, the Russian Journal of Organic Chemistry published a shared review under the title "Modern Trends of Organic Chemistry at Russian Universities" [1]. The present review summarizes the results of research performed at Russian universities over the period since the publication of the first review. This review was written by a large group of authors from many cities of Russia, and we believe that it will be of interest to readers, demonstrating the trends and lines of development of organic chemistry not only at Russian universities, but also in Russian science.

1. DEPARTMENT OF ORGANIC CHEMISTRY OF KAZAN FEDERAL UNIVERSITY

The Department of Organic and Medicinal Chemistry of Kazan Federal University, formerly the Department of Organic Chemistry transformed in 2021, over the past years has mainly focused on the synthesis of organic compounds with unique properties. However, in addition to synthetic work, the Department has been intensively developing interdisciplinary approaches to supramolecular chemistry, which make it possible to create, on the basis of newly synthesized organic compounds, complex systems that mimic living systems. Quite recently, effective methods have been



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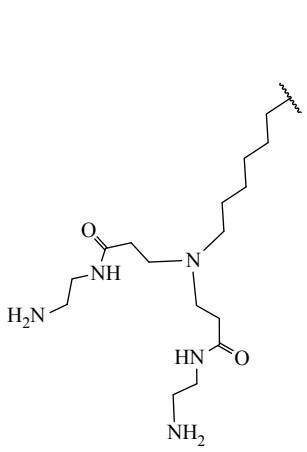
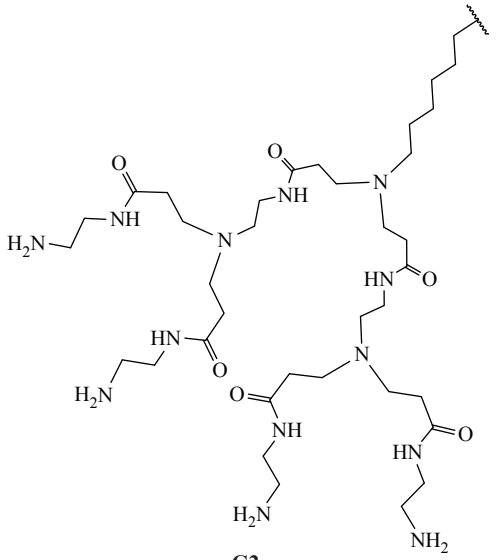


G1



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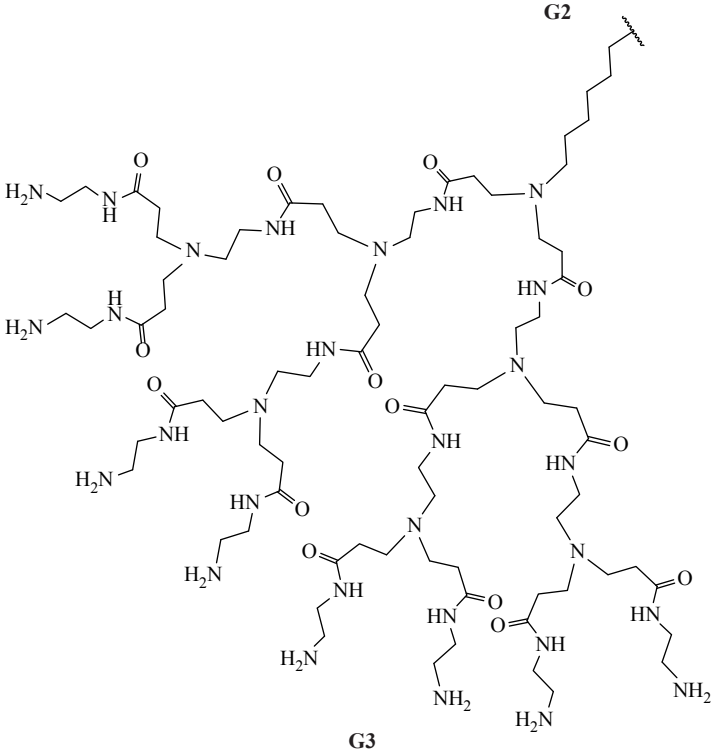


G3

Fig. 1.1. G1−G3 poly(amidoamine) dendrimers on the thiacalix[4]arene platform.

developed for the synthesis of water-soluble derivatives of thiacalix[4]arene in different conformations (*cone*, *partial cone*, and *1,3-alternate*), as well as mono- and decasubstituted pillar[5]arenes, allowed the synthesis of a wide range of compounds containing amide, carboxyl, ammonium, lactide, amine, thioether, and phosphorus-containing moieties, as well as residues of natural amino acids. The synthesized compounds were proposed as components of stable self-assembling supramolecular systems capable of inhibiting cholinesterase, binding a number of metal cations, amines, herbicides, dyes, and biopolymers, as well as drugs for targeted transport, prolonged action, and improved bioavailability.

During 5 years, one of the research directions of the group of **Prof. I.I. Stoikov** was the synthesis and study of the properties of water-soluble derivatives of thiacalix[4]arene containing quaternary ammonium [2–5] and poly(amidoamine) [6–12] groups (Fig. 1.1). The obtained compounds can not only act as antitumor and antimicrobial agents in themselves, but also exhibit the ability to bind biologically significant low- and high-molecular-weight substrates (catecholamines, DNA, proteins, and enzymes). A separate direction of research of the scientific group is the synthesis and electrochemical properties of phenothiazine derivatives [13–17]. These studies are performed in collaboration with the group of **Prof. G.A. Evtugin** (Department of Analytical Chemistry) and resulted in the development of highly sensitive electrochemical sensors based on a wide range of aromatic derivatives of phenothiazine.

Another direction of the group is the development of methods for the constructing 3D self-assembling nanoparticles of different charges and stabilities on the basis of polyionicpillar[5]arene, *p-tert*-butylthiacalix[4]-arene, and SiO_2 derivatives in aqueous media by combining them via electrostatic interactions [18–25]. Analysis of the self-association characteristics of each platform showed that the key role in the formation of an interpolyelectrolyte associate is played by the ability of the macrocycle to form the core of the associate, underlain by the intrinsic micelle-forming properties of the macrocycle. It was found that it is the sign of the surface charge (negative or positive), and not its value, that is a decisive factor for the recognition of biopolymers via electrostatic interactions [20, 22–25]. Evidence was obtained showing that to change the selectivity of the interpolyelectrolyte associate with respect to differently charged biopolymers (DNA or proteins), changing the conformation of the macrocycle (in the case of thiacalix[4]arene derivatives) without changing the nature of the functional groups would suffice [24].

The group of **Prof. I.I. Stoikov** has been continuing research into the properties of paracyclophanes, specifically pillar[*n*]arenes [26–35]. Thus, in recent years, regioselective synthetic approaches to polyfunctional pillar[5]arene derivatives have been developed [28]. Macrocycles containing one to three 1,4-quinone fragments (Fig. 1.2) were synthesized by the selective oxidation of 2-bromoethoxy derivatives of decasubstituted pillar[5]arenes with cerium(IV) ammonium nitrate under mild conditions [36]. A water-soluble pillar[5]arene containing a fluorescent label and nine sulfoethyl fragments was obtained for the first time. UV-Vis and fluorescence spectroscopy was used to show that the synthesized macrocycle forms 1 : 2 associates with natural proteins (binase, bleomycin, and lysozyme) with antibacterial activity [28].

In the field of creating materials for biomedical purposes, an approach to constructing supramolecular films containing pillar[5]arene fragments by the thiol/disulfide redox crosslinking of the corresponding sulfur-containing macrocycles was realized (Fig. 1.2). The resulting film structures exhibited the ability to self-heal under the action of atmospheric oxygen [36]. The ability of pillar[5]arene comprised in the films to form 2 : 1 complexes with the antimicrobial drug moxifloxacin ($\log K_{11} = 2.14$ and $\log K_{12} = 6.20$) was demonstrated by a set of physicochemical methods.

Films containing moxifloxacin effectively inhibited the growth of *Staphylococcus aureus* and *Klebsiella pneumoniae* biofilms on adhesive surfaces. The ability of pillar[5]arene containing *p*-amidopyridine fragments (Fig. 1.2) to form supramolecular 2 : 1 associates with Cu^{2+} and Pd^{2+} cations in methanol was also demonstrated [29]. It was shown that at a concentration of 10 mM the associates transformed into metallosupramolecular coordination polymers in the form of supramolecular gels. The resulting metallosupramolecular coordination polymers selectively sorbed 2,6-dinitro- and 2,4,6-trinitrophenols from water.

An important area of research at the Department is the design of monosubstituted pillar[5]arenes functionalized with phosphorus-containing, carboxyl, amide, and ammonium fragments [37–51]. On their

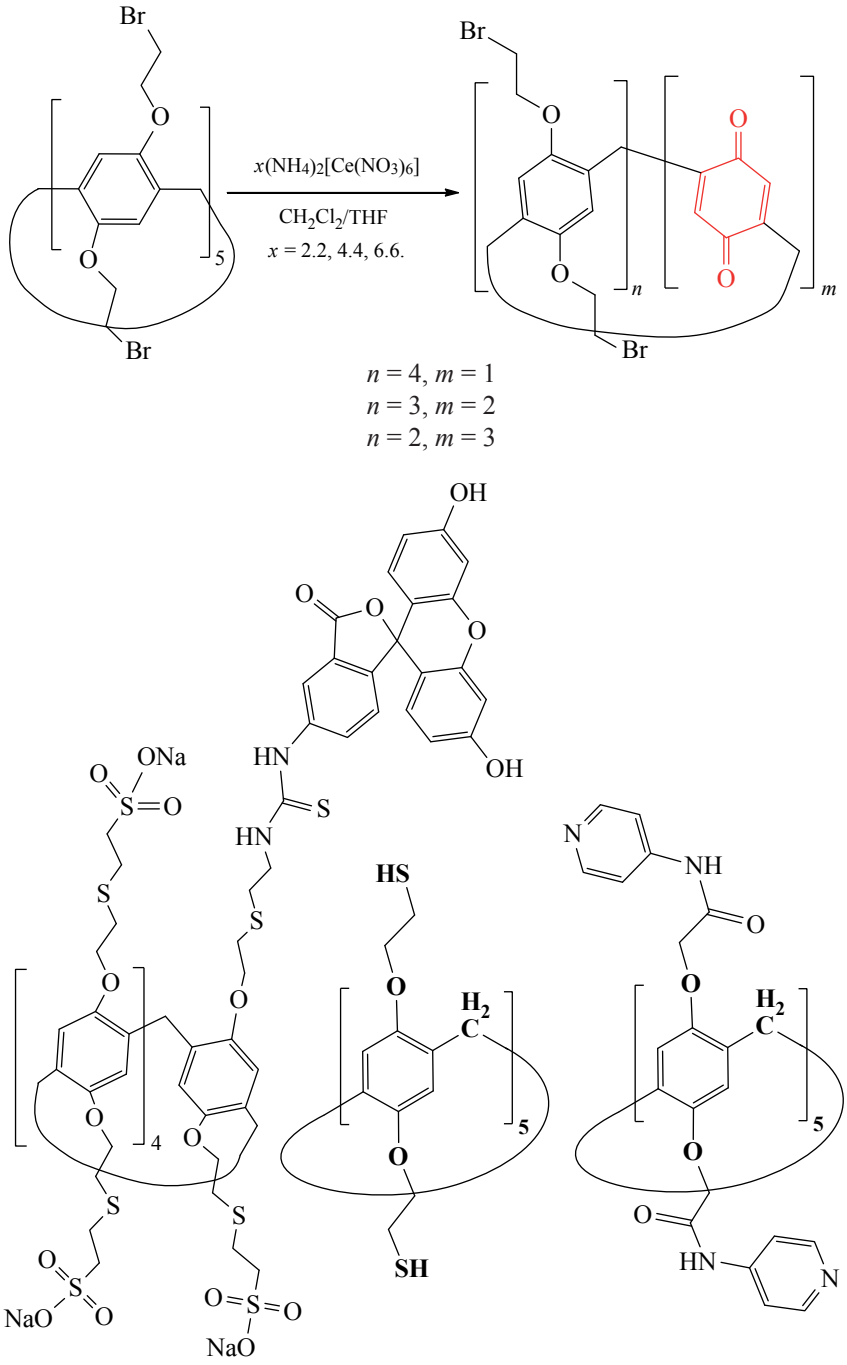


Fig. 1.2. Synthesis of quinone[*m*]pillar[5]arenes and structures of decasubstituted pillar[5]arenes.

basis, an approach to the formation of rotaxane structures [37], supramolecular polymers [39, 40], self-inclusion complexes, and solid lipid nanoparticles [41, 42] capable of encapsulating biologically significant substrates, was developed. Furthermore, an approach to the synthesis of water-soluble decasubstituted pillar[5]-arenes [43–45] containing amino acid fragments was proposed and realized, and the ability of these structures to bind herbicides [46] and dyes [47, 48] and inhibit cholinesterase [49] was demonstrated.

The scientific direction focused on the synthesis of amphiphilic (thia)calix[4]arene derivatives is actively developing at the Department of Organic and Medicinal Chemistry under the leadership of Corresponding Member of the Russian Academy of Sciences **I.S. Antipin** and Associate Professor, Doctor of Chemical Sciences

V.A. Burilov. Amphiphilic molecules have attracted special attention due to their ability to form highly ordered molecular assemblies in aqueous solutions, and these assemblies are capable of molecular recognition and multicenter interactions. Such systems are constructed using a popular modular approach of click chemistry (copper-catalyzed cycloaddition of azides and alkynes, CuAAC). The amphiphilic derivatives are synthesized by functionalization of the *1,3-alternate* conformer of thiacalix[4]arene; the fuctional groups include gallic acid dendrons [52], which are capable of stabilizing palladium particles in the form of nanodendrites, and fluorescein fragments (Scheme 1.1) [53, 54].

Amphiphilic triazoles derived from arylazidocalix-[4]arene with potential applications in micellar catalysis or DNA compaction were also obtained [55, 56]. Some polyamino derivatives of calix[4]arene exhibited anticancer activity [57].

The synthesized amphiphilic diacetylenic (thia) calix[4]arene derivatives (Fig. 1.3) were used to modify polydiacetylenic vesicles which show response to lanthanides [58], DNA [59], or nucleoside phosphates [60]. It was found [61] that diacetylenic (thia)calix[4]-arene derivatives can undergo reductive dealkylation in the presence of hydrazine.

Another line of research of the group of **Assoc. Prof. V.A. Burilov** is the synthesis of polymeric particles by aggregation and CuAAC fixation of macrocyclic imidazolium azides and alkynes (Fig. 1.4). The synthesized polymeric particles can be used as carriers for catalytically active palladium nanoclusters [62–64], and imidazolium azides can also be used for the synthesis of polyfunctional zwitterionic amphiphiles [65].

The introduction of imidazolium fragments into the macrocyclic platform opens the way to the synthesis of NHC ligands for metal complexes. Based on thiacalixarene, precursors and bis-NHC complexes of Pd(II) [66–68] and heteroligand NHC−pyridine complexes of Pd(II) [69], which showed high activity in coupling and reduction reactions, were prepared.

Kazan Federal University and RIKEN (KFU−RIKEN) research joint laboratory «Biofunctional chemistry laboratory», under the supervision of Assoc. **Prof. A.R. Kurbangalieva** and **Dr. Katsunori Tanaka**, developed an effective strategy for the synthesis of heterogeneous structurally well-defined glycoalbumins capable of selectively recognizing cancer cells [70–73]. Two sequential click reactions were used to synthesize clusters, which were further used to introduce two different asparagine-linked glycans into the structure of an azide-containing linker, and then the resulting *N*-glycanazides were immobilized on fluorescently labeled albumin.

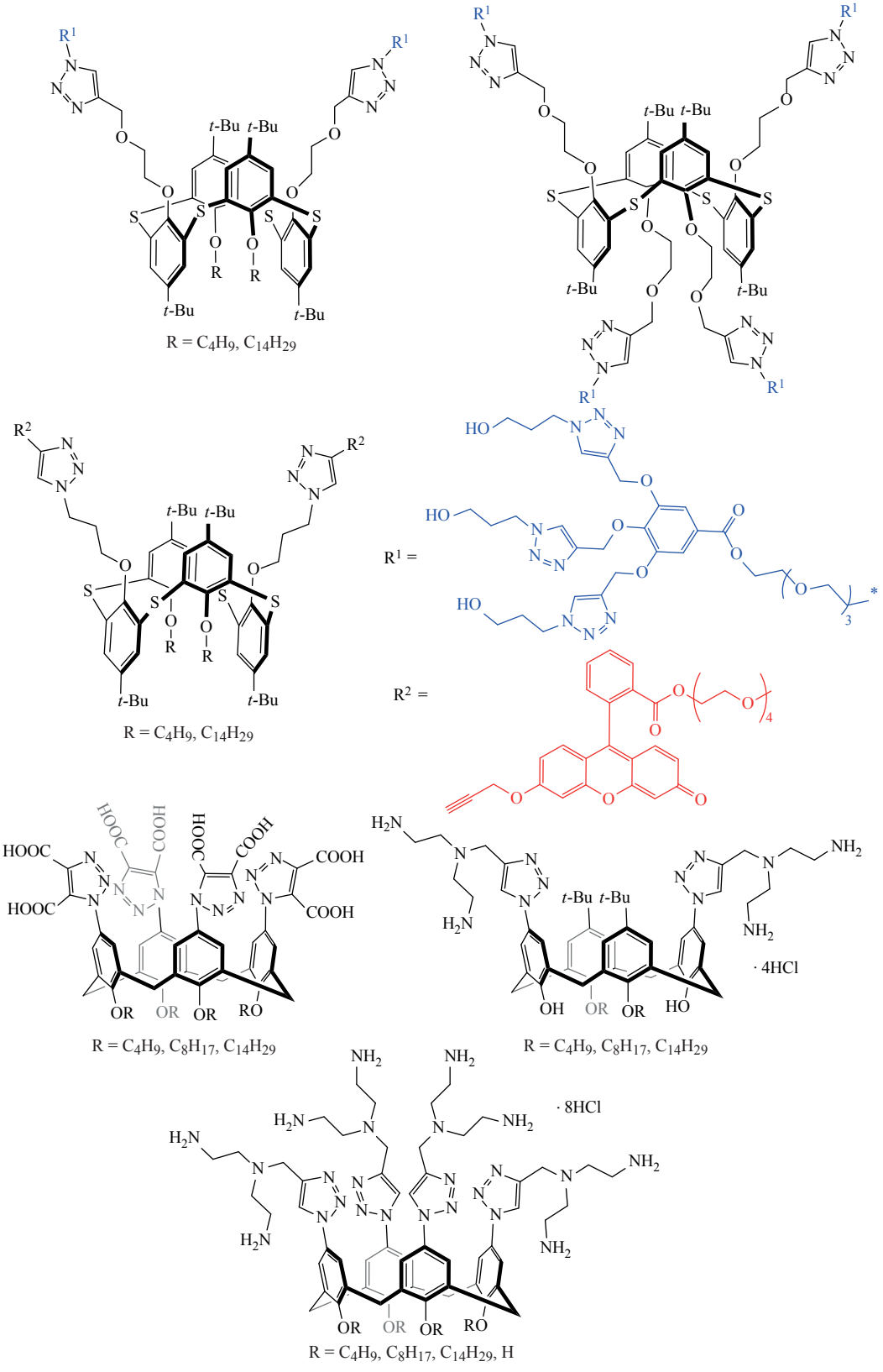
A study of the selectivity of interaction of the synthesized glycoconjugates with various cancer cell lines revealed two leader glycoclusters, based on which *N*-glycoalbumins with a higher degree of heterogeneity, viz. containing fragments of four different *N*-glycans (Scheme 1.2a), were synthesized for the first time [71, 73]. It was shown that increasing degree of heterogeneity favors more effective recognition of cancer cells in a living organism. Fluorescence microscopy was used to reveal the key relationships between the structure of a heterogeneous glycoconjugate and its ability to interact with cancer cells in vitro, ex vivo, and in vivo experiments [70–73], which is an important step toward understanding the mechanism of glycan pattern recognition associated with cancer targeting.

The members of the KFU−RIKEN laboratory developed a promising technique for drug delivery to cancer cells by means of a glycosylated albumin−based artificial metalloenzyme [74, 75]. A ruthenium catalyst was introduced into the hydrophobic binding pocket of human serum albumin, and *N*-glycans to ensure recognition of specific cells and delivery to them of the whole delivery system were immobilized on the protein surface. The ruthenium complex catalyzed the conversion of the prodrug to the anticancer agent umbelliprenin [74]. Thus, the targeted delivery of the catalyst to cancer cells was accomplished, and evidence was obtained for the in vivo applicability of the artificial metalloenzyme.

A preparative scale and convenient synthesis of stereoisomerically pure 2,6-disubstituted derivatives of the 1,5-diazacyclooctane series has been proposed, which involves the alkylation of the reaction products of acrolein with optically active amino alcohols [(*R*)-(–)-2-phenylglycinol and (1*S*,2*R*)-(–)-*cis*-1-amino-2-indanol] followed by the catalytic hydrogenolysis of the resulting heterocyclic products (Scheme 1.2b) [76, 77]. Different behaviors of 2,6-disubstituted derivatives of 1,5-diazacyclooctane in the hydrogenolysis reactions in the presence of Pearlman's catalyst has been revealed.

In the framework of research on biologically active five-membered *O*-heterocycles, methods for the synthe-

Scheme 1.1.


R = C4H9, C14H29
R = C4H9, C14H29
R1 =
R2 =
R = C4H9, C8H17, C14H29
· 4HCl
R = C4H9, C8H17, C14H29
· 8HCl
R = C4H9, C8H17, C14H29, H

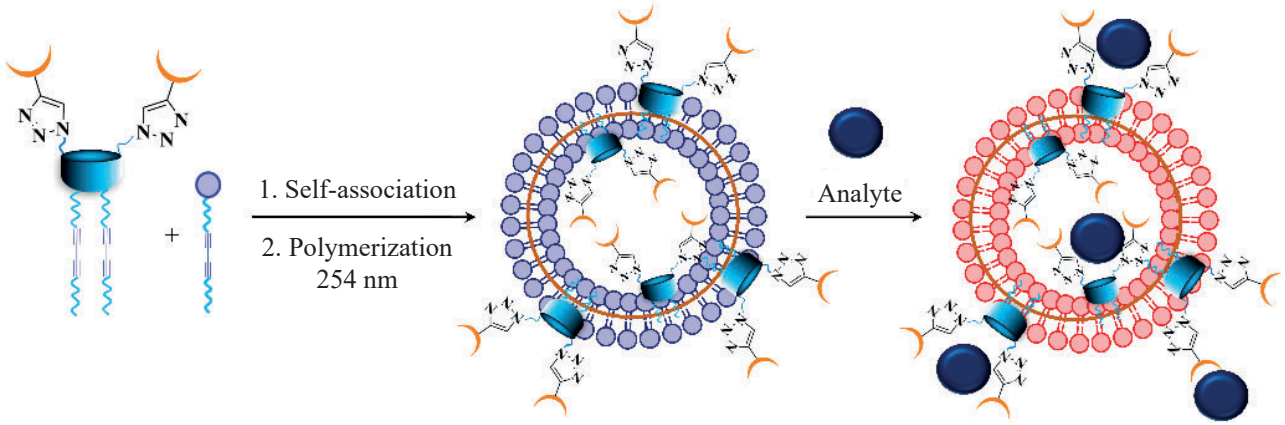


Fig. 1.3. Sensors based on modified polydiacetylenic vesicles.

sis of optically active sulfones and disulfones derived from 3,4-dihalo-2(5*H*)-furanones, monoterpene alcohols, thiols, and dithiols were developed [78–80]. A fluorescent furanone derivative containing a sulfonyl group and a 2-(benzothiazol-2-yl)-4-bromophenol residue was synthesized, and its ability to penetrate into *Staphylococcus aureus* cells was assessed [81]. Among the synthesized compounds, those with pronounced antimicrobial and antifungal activities against *Staphylococcus aureus*, *Bacillus cereus*, *Streptococcus spp*., and *Candida albicans* were found (Scheme 1.2c). It was found that combining 5(*S*)-(*l*-menthyloxy)- and 5(*S*)-(*l*-bornyloxy)-2(5*H*)-furanone sulfones with aminoglycoside antibiotics, antifungal agents, and benzalkonium chloride enhance the antimicrobial or antifungal effect of these drugs in both mono- and mixed cultures [78, 82–84].

2. DEPARTMENT OF ORGANIC CHEMISTRY OF St. PETERSBURG STATE UNIVERSITY

To the 300th anniversary of the founding of St. Petersburg State University

The Department of Organic Chemistry of St. Petersburg State University has a rich history and traditions. To date, tthe Department employs 7 professors, 10 associate professors, as well as a senior lecturer and assistant of the Institute of Chemistry of St. Petersburg University. From 2018 to 2023, the Department performed research projects funded by the Russian Science Foundation (18 projects, ten of which are for young scientists), the Russian Foundation for Basic Research (6 projects), and the Grant Council under the President of the Russian Federation (1 project). This review outlines the current research areas of the Department and the most important results obtained over the past 5 years. The research is mainly focused on organic synthesis, primarily of heterocyclic compounds, including the study of reaction mechanisms by quantum-chemical calculations. Two research groups are developing new approaches and methods of organic analysis.

The research group headed by **Dr. Chem. Sci., Prof. I.A. Balova**, **Director of the Institute of Chemistry**, works in the field of acetylene chemistry, developing the traditions laid down at St. Petersburg University by Acad. A.E. Favorskii, who created a unique scientific school [85]. The research is related to the development of convenient methods for the synthesis of various heterocyclic systems by cyclizations of mono- and diacetylenes. A general synthetic approach to macrocyclic enediyne systems annulated with heterocycles was developed to obtain synthetic analogs of natural enediyne antibiotics using the Nicholas reaction as a key reaction for enediyne ring closure. The benzothiophene–azaenediyne core has been identified as a lead structure for further modification and search for structures with anticancer activity (Scheme 2.1) [86, 87]; furthermore, a few other heterocyclic enediyne analogs were prepared [88–90].

A new class of compounds, 4-ethynyl-5-iodo-1,2,3-triazoles, was obtained by the Cu-catalyzed cycloaddition of organic azides to 1-iodobuta-1,3-diynes [91, 92]. These products were transformed, via the Sonogashira–Hagihara and Suzuki–Miyaura cross-coupling reactions, to the corresponding 4,5-bis(arylethynyl)- and 5-aryl-4-ethynyltriazoles, which showed interesting fluorescent properties [93, 94]. An approach for the synthesis of previously unknown heterocyclic azides, 4-azidocinnolines and their derivatives (Scheme 2.2, A) [95], was developed, and the newly synthesized

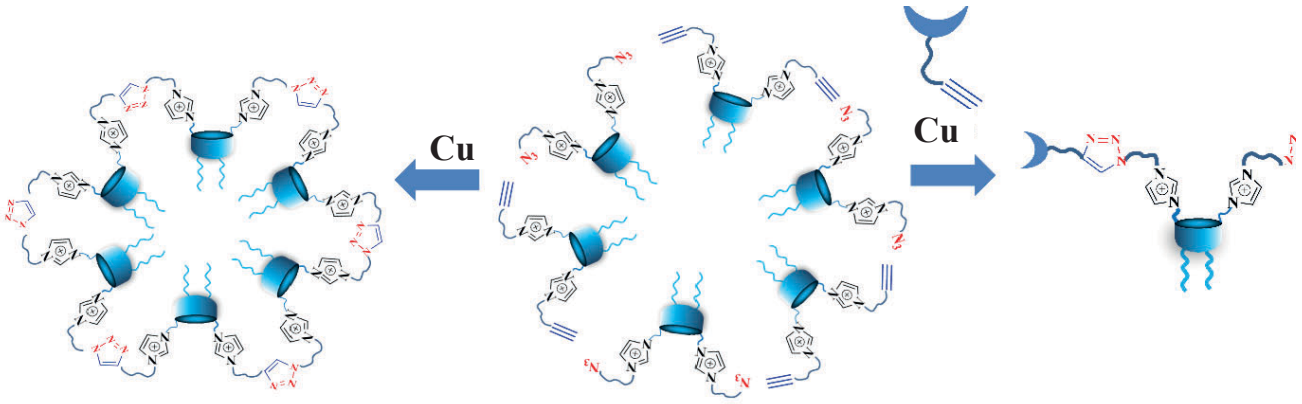
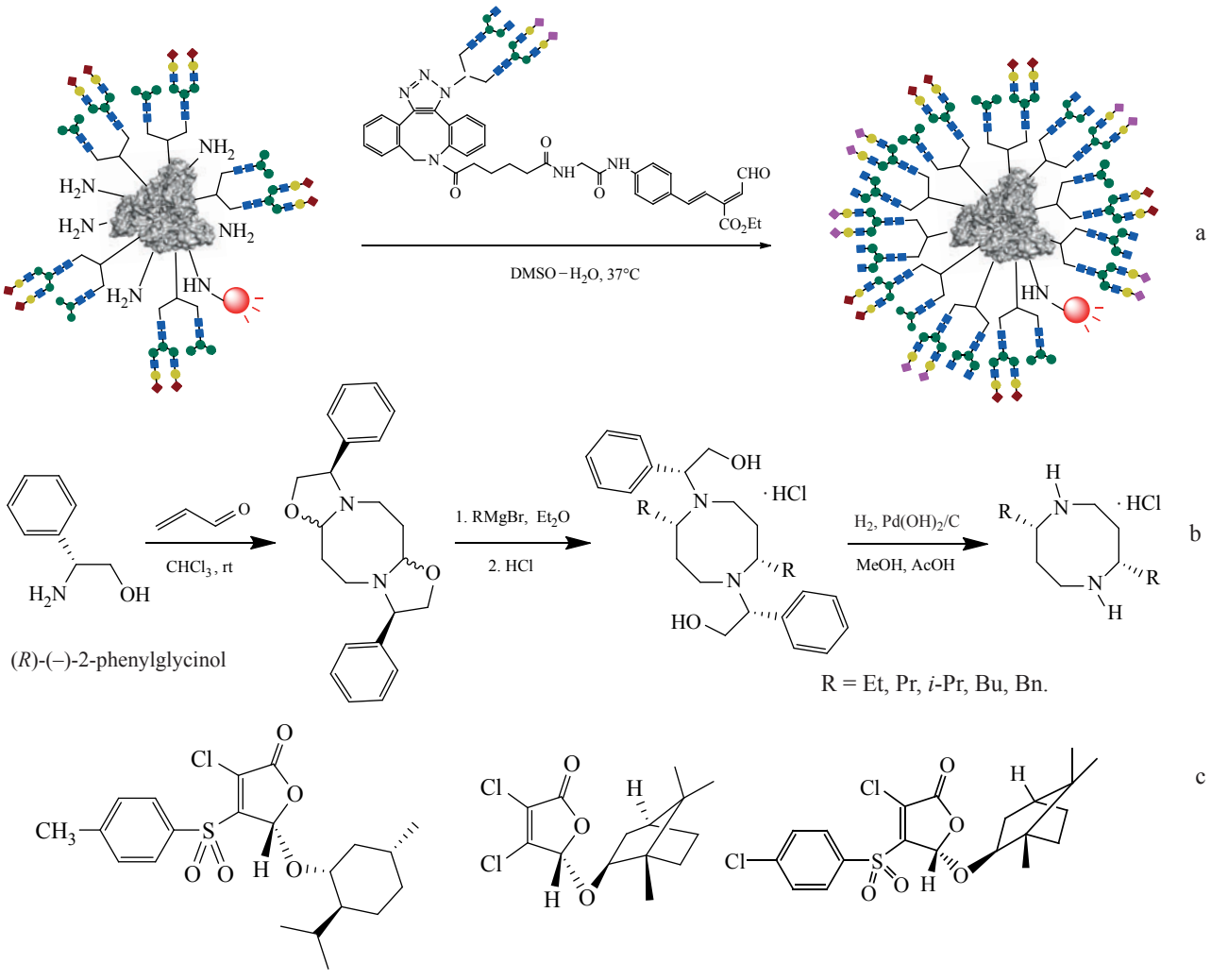


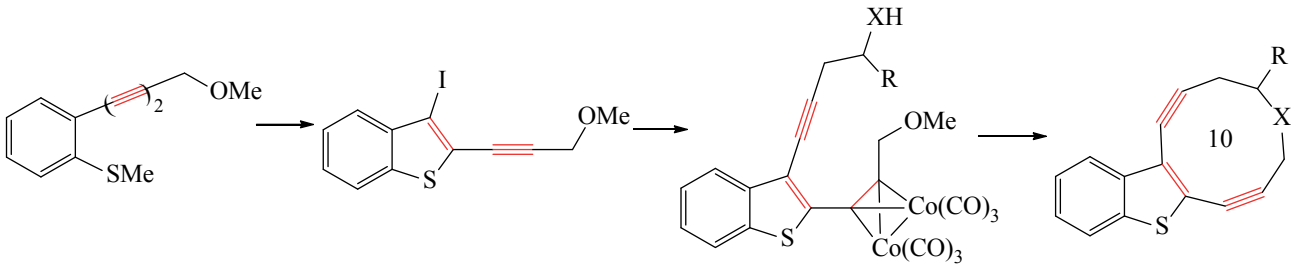
Fig. 1.4. Applications of macrocyclic imidazolium CuAAC precursors.

compounds are now studied as potential fluorophores [96]. Derivatives of the cinnoline series were found to be of interest as selective inhibitors of the enzyme protein phosphotyrosine phosphatase 1B (PTP1B) (Scheme 2.2, B) (in collaboration with Dr. Biol. Sci. A.O. Shpakov, Sechenov Institute of Evolutionary Physiology and Biochemistry, Russian Academy of Sciences) [97–100].

Scheme 1.2.



Scheme 2.1.



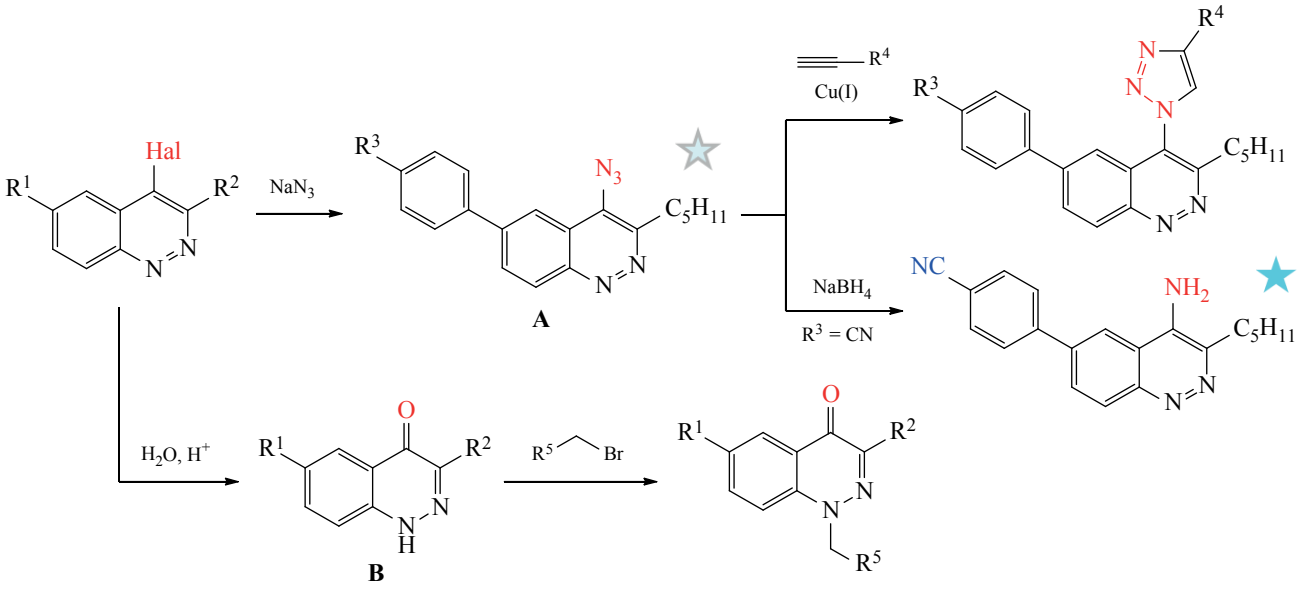
X = O, R = H, CH_2CO_2Me; X = NSO_2Ar, R = H.

The effect of the nature of substituents and solvents on the 5-*exo*/6-*endo* selectivity of the Richter cyclization of *o*-(arylethynyl)arenediazonium salts was studied to prepare Combretastatin A analogs [99]. A general synthetic approach to cycloalkynes fused with heterocycles was developed for further use of the synthesized compounds in bioconjugation reactions such as the strain-promoted addition of azides to the triple bond (SPAAC) in the cycloalkyne (Scheme 2.3). Imaging experiments on metabolically labeled Hek293 cells were carried out using a new heterocyclononyne reagent [101], and new fluorescent oxacyclononynes were obtained [102].

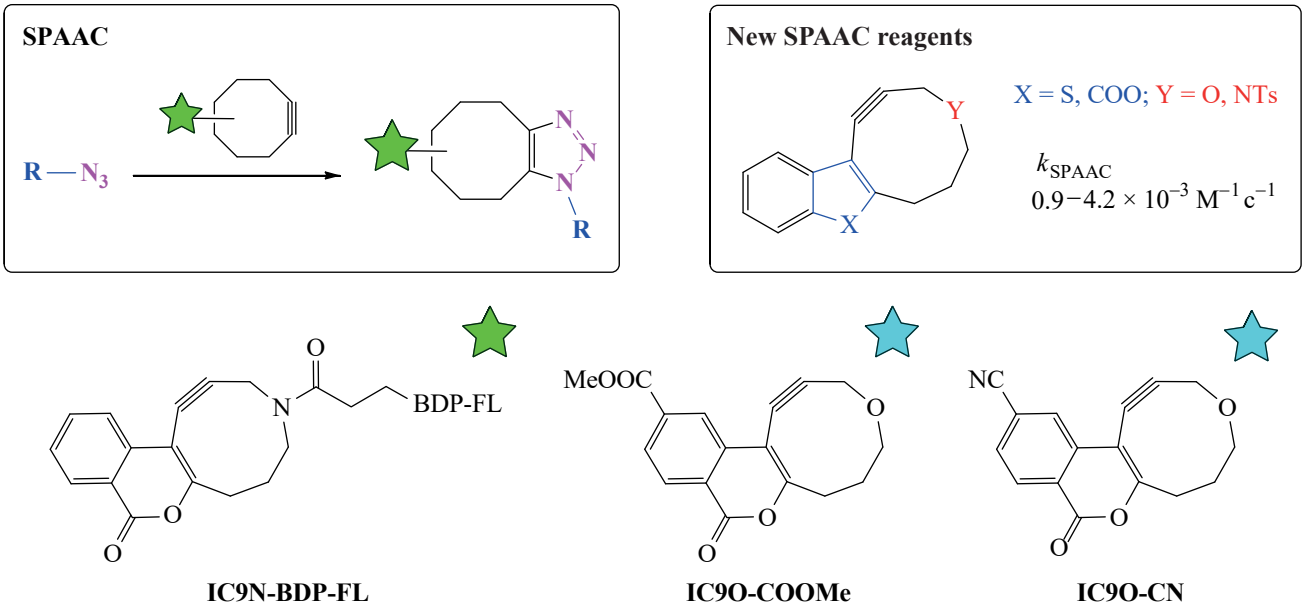
An important related area of research of the scientific group was the synthesis and study of new Pd- and Cu-containing acyclic and cyclic diaminocarbene complexes for the design of new catalytic systems [103–109].

The research group of **Dr. Chem. Sci., Prof. A.F. Khlebnikov** synthesized 2*H*-azirine-2-carboxylic acid chlorides [110–113], whose reactions with nucleophiles make it possible to obtain, along with various derivatives of these acids [114], azirine-containing building blocks with diazo, azido, and other active functional groups with high synthetic potential (Scheme 2.4). Methods for the synthesis of azirinyl-substituted diazo compounds were developed [112, 113], and cyclization and heteroannulation reactions of these compounds gave alkyl 5/4-hydroxy-3*H*-benzo[*e*]-indole-4/5-carboxylates [115], alkyl 2-hydroxy-3-oxo-2,3-dihydro-1*H*-pyrrole-2-carboxylates [113], cyclo-heptatrienyl-substituted azirines, pyrroles, and isoxazoles [116], and benzo/furo/thieno[*e*]-fused 1*H*-indol-7-ols [117]. Methods were developed for the synthesis of 2-(azidocarbonyl)-2*H*-azirines [118], which were then easily transformed into derivatives of 2-(1*H*-tetrazol-1-yl)acetic acid [119], benzo- and heterofused

Scheme 2.2.



Scheme 2.3.



IC9N-BDP-FL **IC9O-COOMe** **IC9O-CN**

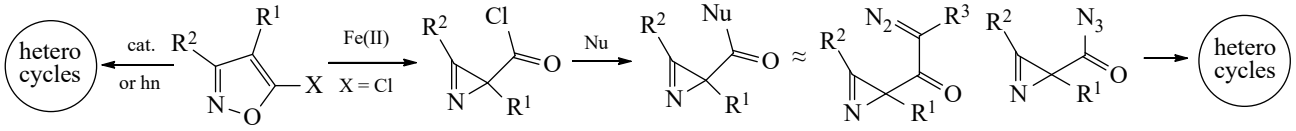
1*H*-pyrrolo[2,3-*b*]pyridin-6(7*H*)-ones, α-aminopyrroles [118], and alkyl 5-aminopicolinates [120].

Methods for the synthesis of bi-, ter-, and quaterpyridines, based on the Fe(II)/Au(I)-catalyzed relay isomerization of 4-propargylisoxazoles, were developed [121–123]. The discovered catalytic rearrangements of isoxazoles with unsaturated substituents in the 4-position allowed the synthesis of oxazole [124], *N*-aminopyrazole [125], indole-3-carboxylic acid [126], pyrrole [127], and 4-aminopyridine [128] derivatives. Methods for the preparation of pyrimidine derivatives [129], 1*H*-pyrrole-2,3-diones [130], 4-oxo-1,4-dihydropyridine-3-carboxylates [131], nicotinates [132], 4-hydroxypyridin-2(1*H*)-ones [133], and α-aminopyrroles [134] by $Mo(CO)_6$-catalyzed reactions of isoxazoles and oxadiazoles were devloped. Heterocyclic hybrids containing azirine [111, 135], pyrrole [127, 132, 136–140], imidazole [110], oxazole [112, 141], triazole [111, 142, 143], and tetrazole [119] moieties were obtained by reactions of functionalized azirines.

The research group of **Dr. Chem. Sci., Prof. M.S. Novikov** focused on the development of synthetic approaches based on new reactions of 2*H*-azirines, N–N, and N–O azoles, conjugated azapolyenes, and diazo compounds (Scheme 2.5). A general methodology for the substitution of halogen at the azirine ring was developed, and simple methods were proposed for the formation of C–Hal [144], C–O [145], C–N [146] and C–C [147] bonds at the C^2 atom of the azirine. For some of the functionally substituted azirines obtained by these methods, radically new azirine ring expansion reactions were discovered. These reactions formed the basis of convenient methods of synthesis of new derivatives of pyrrole [148, 149], 1,3-oxazine [150], butenolide-containing derivatives of α- and β-amino acids [151], as well as imidazo[1,2-*a*]-, imidazo[1,5-*a*]-, and pyrazolo[1,5-*a*]pyridines [152, 153]. A series of works were devoted to new applications of available 3-arylazirines as annulating agents in copper-catalyzed syntheses of various pyrrolo- and furo-fused heterocycles [154–156].

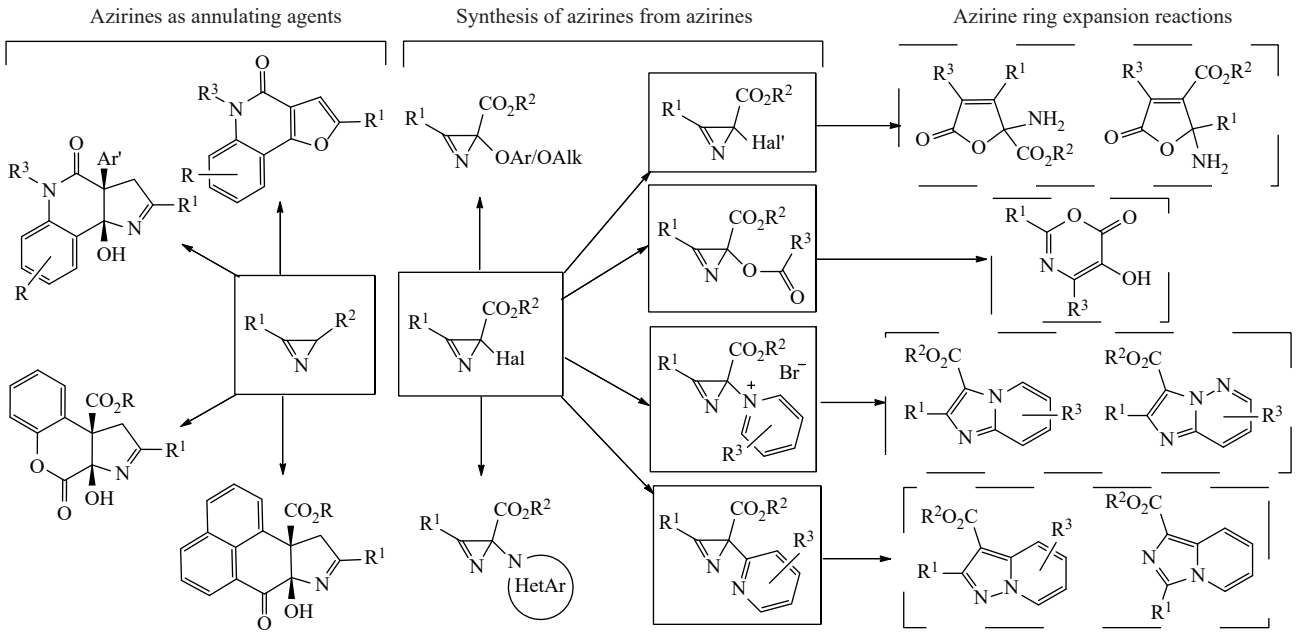
New applications of diazo compounds and diazonium salts as annulating agents for the transformation of some heterocyclic systems into others, specifically azirines

Scheme 2.4.



Nu = NRR', OR, SR, N-heterocycles, CH_2N_2, NaN_3 ...

Scheme 2.5.

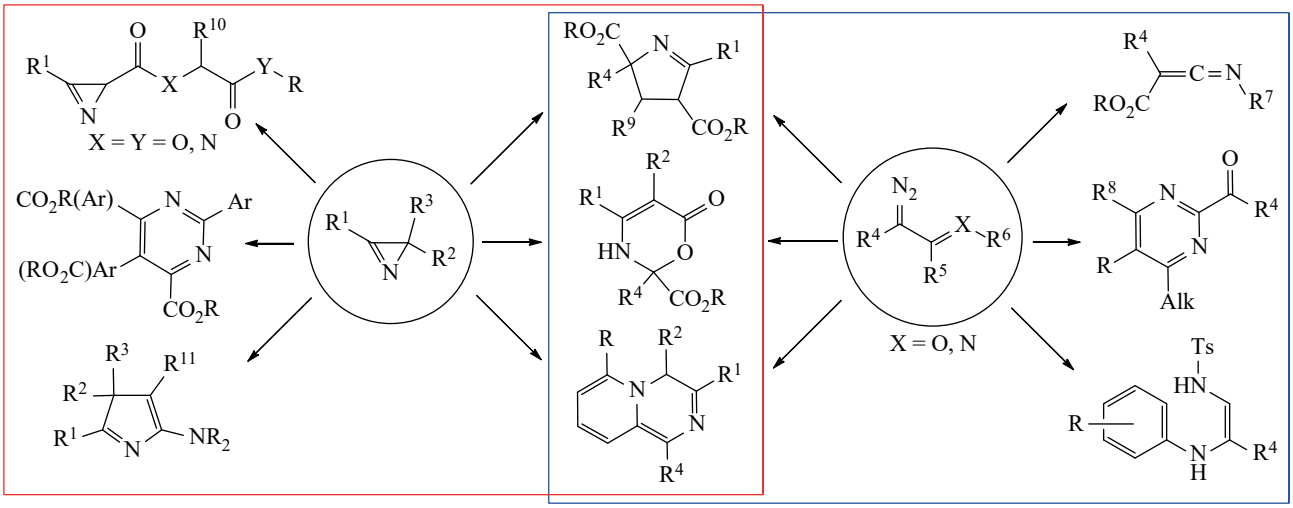


into pyrazino[2,3-*b*]indoles [157], azete derivatives [158, 159], 3,4-epoxypyrrolines [160]; pyrazoles into dihydropyrimidines [161]; 1,2,3-triazoles into 4-pyrrolin-2-ones [162]; 1,2,4-oxadiazoles into 1,3,5-oxadiazines [163]; pyrroles into pyrrolo[1,2-*c*][1,3]oxazinones [164]; and isoxazoles into 1,2,3-triazoles [165], were proposed. These syntheses involve the intermediate formation of conjugated azapolyenes, whose unique chemistry is one of the main priorities of the research group.

Rhodium-catalyzed reactions of 1-sulfonyl-1,2,3-triazoles with azirines and various N–O and N–N azoles were successfully employed to generate the first representatives of di- and triazapolyenes. As a result, it was possible to develop effective methods for the synthesis of new pyrrole [166] and imidazole [167, 168] derivatives and compounds with a new 1,4,8-triazaocta-1,3,5,7-tetraene core [168]. The discussion of the new concepts developed on the basis of the obtained results can be found in reviews [169, 170] written jointly with other research groups of the Department.

Since 2019, the research group of **Dr. Chem. Sci., Assoc. Prof. N.V. Rostovskii, Head of the Department,** has been making efforts in three areas: 2*H*-azirine chemistry, reactions of diazo compounds (1,2,3-triazoles), and search for compounds with high antibacterial and cytotoxic activity (Scheme 2.6). Complexes of 2*H*-azirines with dirhodium tetraacetate were synthesized and characterized for the first time [171]. A convenient and efficient method for the synthesis of 2*H*-azirine-2-carboxylic acids was developed [172] and found to be also suitable to prepare azirinomycin, a natural representative of azirines. Several ways of modification of the carboxyl group in 2*H*-azirine-2-carboxylic acids were studied [173, 174]. In the testing of the antibacterial activity of azirinecarboxylic acids and their derivatives against ESKAPE bacteria (in collaboration with Dr. Med. Sci L.A. Kraeva, St. Petersburg Pasteur Institute of Epidemiology and Microbiology), 3-phenyl-2*H*-azirine-2-carboxylic acid showed the highest antibacterial potency. The medicinal chemistry of 2*H*-azirines was described in detail in the review [175]. It was shown that azirine-2-carboxylates undergo oxidative dimerization under the action of triethylamine to form pyrimidine-4,6-dicarboxylates [176], and azirine-3-carboxylates generated from vinyl azides undergo similar dimerization to pyrimidine-4,5-dicarboxylates under photochemical conditions [177]. Gold(I)-catalyzed reactions of trisubstituted azirines with ynamides can be used to prepare different types of aminopyrrole derivatives [178]. Research in the chemistry of carbenes resulted in the photochemical synthesis of ketenimines from diazo compounds and isocyanides [179]. New methods for the synthesis of 1-pyrrolines [180], 1,3-oxazin-6-ones [174], and pyridopyrazines [181], based on metal-catalyzed reactions of azirines with diazo compounds and their

Scheme 2.6.



analogs (pyridotriazoles), were developed. The reactions of pyridotriazoles, accompanied by a release of nitrogen, were summarized in the review [182]. 1-Sulfonyl-1,2,3-triazoles were also used as a source of carbenes; their reactions with 3,4,5-trisubstituted isoxazoles and primary anilines lead to 2-aroylpyrimidines [183] and ethene-1,2-diamines [184], respectively.

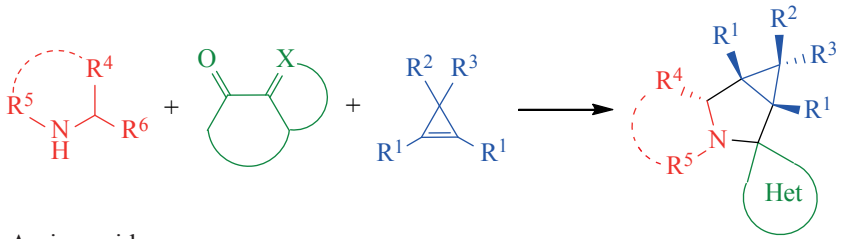
The research group of **Dr. Chem. Sci., Assoc. Prof. A.V. Stepakov** conducted studies on the (3+2)-cycloaddition reactions of azomethine ylides generated in situ from cyclic ketones and amines to cyclopropene dipolarophiles. These reactions were used as the basis for the development of a general method for the regio- and diastereoselective synthesis of functionalized cyclopropa[*a*]pyrrolizidines and 3-azabicyclo[3.1.0]hexanes spiro-fused with heterocyclic moieties (Scheme 2.7) [185–191]. The natural alkaloid tryptanthrine and 11*H*-benzo[4,5]imidazo[1,2-*a*]indol-11-one were introduced into synthetic practice for the first time as carbonyl components for the generation of azomethine ylides, and the suitability of peptides as an amine component for generating azomethine ylides in situ was demonstrated for the first time. Some of the synthesized spiroheterocycles showed a pronounced antiproliferative effect on the K562, HeLa, and CT26 cell lines [192–194].

Inter- and intramolecular reactions involving *N*-acyliminium cations (Scheme 2.8) were studied as a tool for constructing new carbon–carbon and carbon–heteroatom bonds [195, 196], the possibility to generate *N*-acyliminium cations from 3-aryl-4-hydroxy-1-methyl-3,4-dihydroquinazolin-2(1*H*)-ones in situ was demonstrated, and their reactions with alkenes, leading to quinolino[1,2-*c*]quinazolines, were realized [197].

The Pd(II)-catalyzed oxidative cycloaddition of NH-substrates to alkynes was used to develop a one-stage synthesis of novel heterocyclic systems with an azepino[3,2,1-*ij*]quinazoline core (Scheme 2.9) [198].

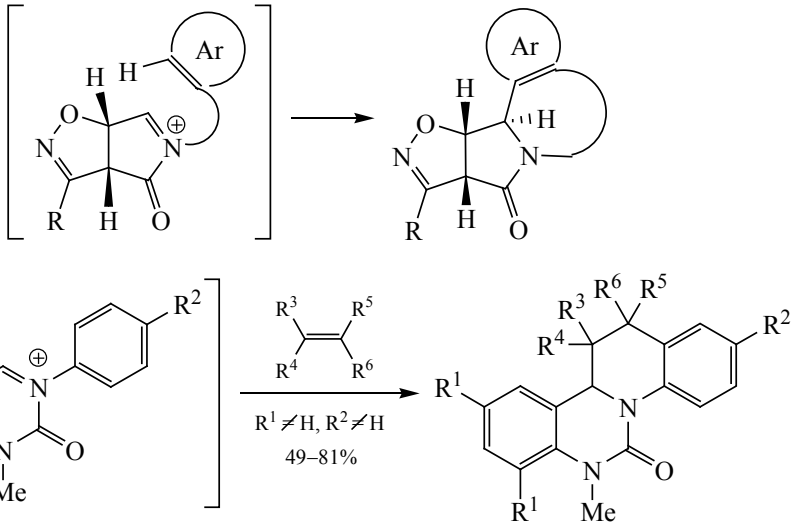
The research of **Cand. Chem. Sci., Assoc. Prof. M.M. Efremova** (formerly the research group of Dr. Chem. Sci., Prof. A.P. Molchanov) is devoted to (3+2)- and (3+3)-cycloaddition reactions of 1,3-dipoles to *N*-vinyl derivatives of pyrroles and indoles: the effect

Scheme 2.7.

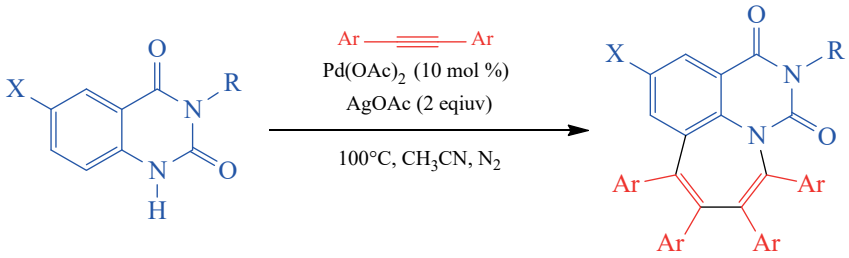


α-Amino acids,
Gly-Gly, Gly-Gly-Gly, benzylamines

Scheme 2.8.



Scheme 2.9.

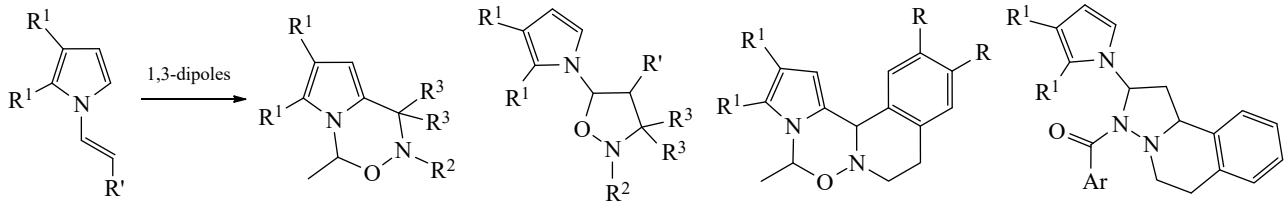


of substituents at the endocyclic double bond on the direction and selectivity of the reactions was studied (Scheme 2.10) [199–202].

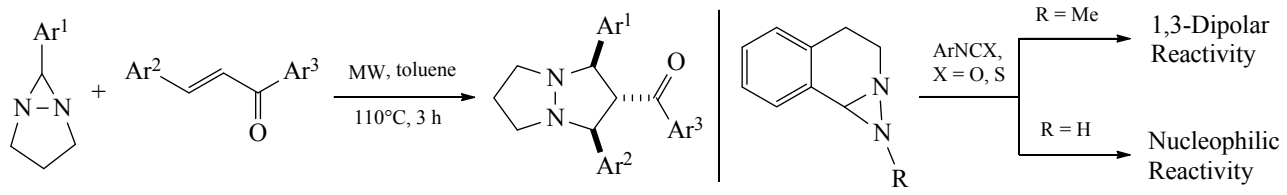
New aspects of the dual reactivity of diaziridines were explored (Scheme 2.11). Azomethine imines were for the first time generated from 6-aryl-1,5-diazabicyclo[3.1.0]hexanes under MW irradiation [203]. Evidence was obtained to show that the reactivity of diaziridines derived from 3,4-dihydroisoquinoline depends on the substituent at the nitrogen atom [204].

Previously unknown spiroisoxazolidines were prepared by reactions of nitrones with unsaturated compounds (Scheme 2.12) [205–207]. Some of the synthesized compounds were tested for antiviral activity against H1N1 influenza virus, as well as for anticancer activity.

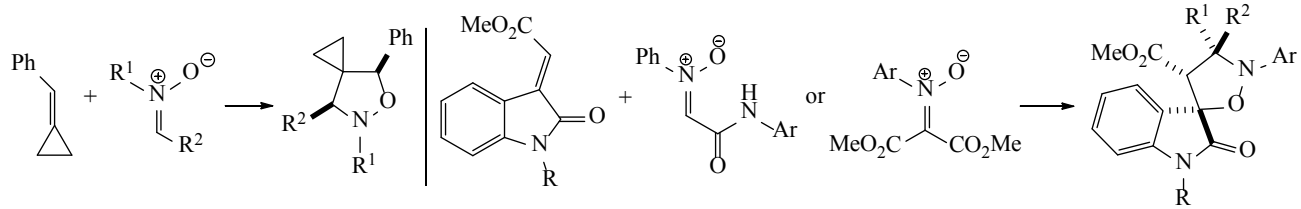
Scheme 2.10.



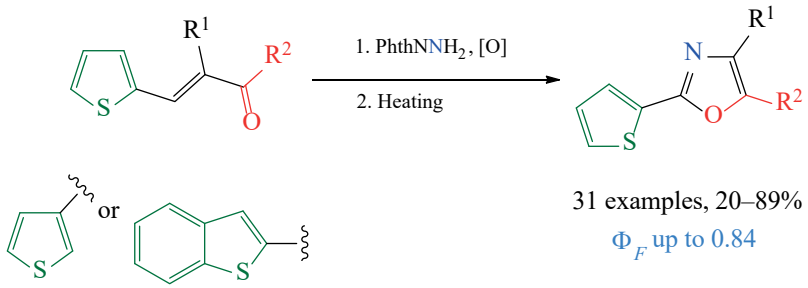
Scheme 2.11.



Scheme 2.12.



Scheme 2.13.



The chemo-, regio-, and stereoselectivity of the cycloaddition of nitrones to pyrrolyl- and indolylallenes [208] and cyclic allenes [209] were studied. In addition, methods for the synthesis of spirocyclic and bridged structures containing an isoxazolidine fragment were developed based on the cycloaddition reactions of nitrones, and the reaction products were tested for antiviral activity [210, 211].

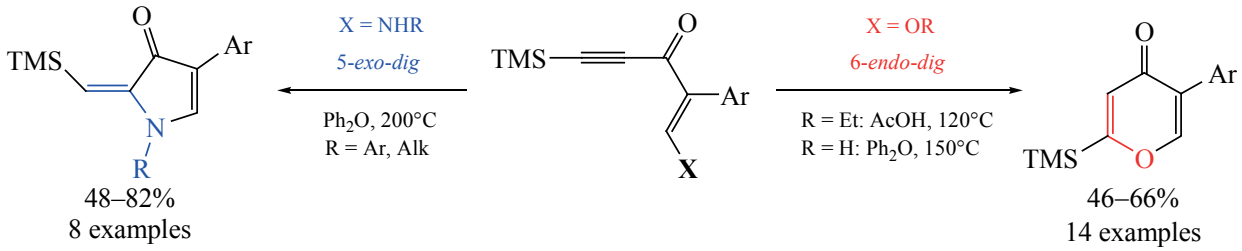
The general focus of the work of **Cand. Chem. Sci., Assoc. Prof. A.S. Pankova** (formerly the research group of Dr. Chem. Sci., Prof. M.A. Kuznetsov) was on the methods of synthesis of five- and six-membered nitrogen heterocycles, based on transformations of aziridines and (trimethylsilyl)pentenynones, and the reactivity of the synthesized compounds. A series of thiophenyl-substituted oxazoles with pronounced fluorescence (Scheme 2.13) were synthesized by thermal recyclization of 2-acylaziridines [212]. The latest achievements in the chemistry of 2-acylaziridines are presented in the review [213].

The construction of heterocycles via electrophile-initiated cross-coupling of enynones is covered in the review [214]. The dependence of the selectivity of cyclization of pent-1-en-4-yn-3-ones on the substituent at the double bond is demonstrated (Scheme 2.14): 6-*endo-dig* cyclization of 1-ethoxy(hydroxy)pentenynones into 4*H*-pyran-4-ones [215] and 5-*exo-dig* cyclization of 1-pentenynones into 2-methylidenepyrrol-3-ones [216].

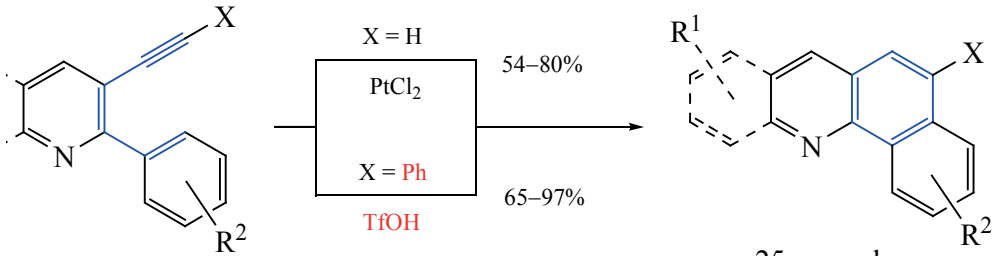
It was shown that cycloisomerization of 2-aryl-3-ethynylpyridines and quinolines can be used as a convenient method for the synthesis of benzo[*h*]-quinolines and benzo[*c*]acridines, and, therewith, the choice of catalyst, TfOH or $PtCl_2$, depends on the substituent at the triple bond (Scheme 2.15) [217]. The potential of synthetic schemes based on cyclization of *ortho*-ethynylbiaryls and recent advances in this field are demonstrated in the review [218].

Since 2021, the group of **Dr. Chem. Sci., Prof. D.S. Bolotin** has been performing research in one of the fields of electrophilic organocatalysis: creation of analogs of metal complex catalysts that have a nonmetal with a kinetically labile coordination vacancy as the

Scheme 2.14.



Scheme 2.15.



central atom, namely, a σ-hole (positive electrostatic potential) localized on the nonmetal (Scheme 2.16, σ-holes are highlighted in color).

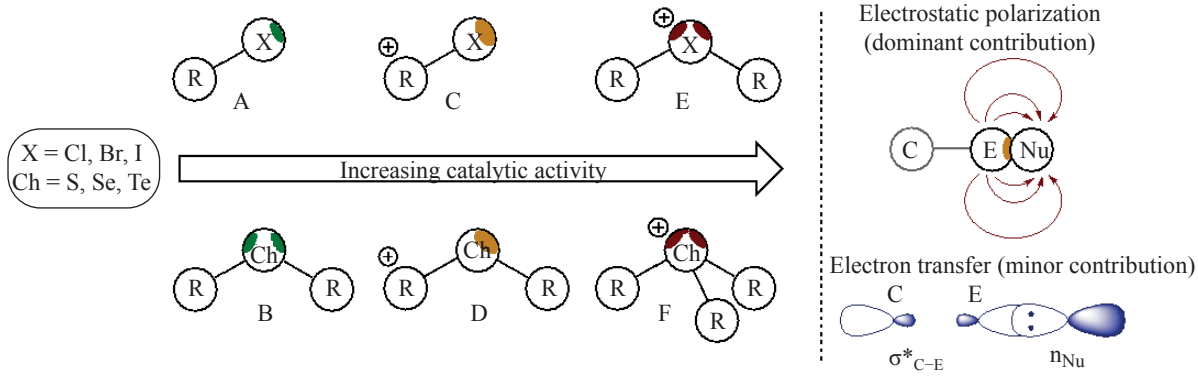
First-generation catalysts—uncharged organoelement compounds with strong electron-acceptor groups (**A** and **B**) — are of little promise for use in catalysis because of their low stability under most reaction conditions and low catalytic activity [219]. Cationic heterocyclic compounds with an exocyclic halogen or chalcogen atom (**C** and **D**, second-generation catalysts) are much more active and have moderate stability under reaction conditions [220]. The most promising are hypervalent halonium (**E**) [221–226] and chalconium salts (**F**) [221, 223, 227], which was confirmed in reactions leading to the synthesis of a series of heterocyclic compounds. Quantum-chemical calculations were carried out to find the most effective hetero element and showed that organotellurium and organoiodine compounds have the greatest potential as noncovalent electrophilic catalysts [223, 228].

The research group of **Dr. Chem. Sci., Prof. L.A. Kartsova** developed new approaches to selective chromatographic (HPLC, GC, HPTLC, HILIC) and electrophoretic (CZE, MEKC, MEEKC, CEC, LECE) analysis of biologically active compounds (steroid hormones, amines and amino acids, drugs, catecholamines, polyphenol antioxidants, etc.) with UV and MS detection in plasma, blood serum, urine, culture fluids, biological tissues (brain tissue, liver tissue cells) using complexation processes, including ligand exchange, with the participation of ionic liquids and organized media (crown ethers, macrocyclic antibiotics, cyclodextrins, micelles formed by anionic and cationic surfactants, microemulsions, and hyperbranched polyethyleneimine polymers [229–238].

The metabolic profiling of biological fluids and medicinal plant extracts was performed [239–241]. A variant of the simultaneous determination of antituberculosis drugs and their metabolites in human blood plasma by RP-HPLC–ESI(+)–MS/MS with multiple reaction monitoring (MRM) was proposed [242]. The developed variant of electrophoretic analysis of a mixture of native amino acids using a background electrolyte containing Cu^{2+} ions made it possible to detect all amino acids due to the formation of Cu^{2+}–amino acid complexes that absorb in the UV region and was adapted to analysis of culture fluids during the development of a cellular model of the nonalcoholic fatty liver disease (NAFLD) [243].

The analytical potential of polymer nanoparticles (copolymers of styrene and divinylbenzene with terminal sulfo- and quaternary ammonium groups) as

Scheme 2.16.



modifiers of electrophoretic systems in the determination and intracapillary concentration of organic acids, catecholamines, and amino acids for targeted metabolic profiling was revealed. The detection limits of analytes were reduced 2–10 times, which has allowed their determination in biological fluids [244–246]. A hybrid version of intracapillary electrophoretic concentration was proposed, with the use of a chiral selector synthesized from cyclodextrin and an imidazolium ionic liquid, which ensured the concentration of ketoprofen and ketorolac enantiomers 290–390 times and reduced their detection limits to 12–55 ng/mL [247, 248].

The main areas of research of the group of **Dr. Chem. Soc., Prof. I.G. Zenkevich** are directed to the improvement of chromatography–spectroscopy methods of identification of organic compounds and related problems. The proposed type of recurrence relations (1), which assume equally spaced arguments and apply to properties (A) depending on temperature, pressure, or component concentrations, was used to fit retention time curves as an effective method for the detection of formation of hydrates of organic compounds under HPLC conditions [249–254].

$$A(x) = aA(x + \Delta x) + b, \Delta x = \text{const}, \quad (1)$$

$$B(n) = aB(n + 1) + b. \quad (2)$$

Such recurrence relations are applicable for control, correction, and recovery of the physicochemical characteristics of organic compounds [255], fitting of the temperature dependence of water solubility [256], and solution of other problems. Moreover, the fact that relation (2) is fulfilled for chromatographic retention indices (RI) of related compounds turned out to be a key criterion that allows this group of compounds to be classified as a series of homologs or (in the general case) congeners. This criterion in combination with chemical properties was used for identification of previously unknown products of partial hydrolysis of tetraethoxysilane [257].

It was established by HPLC–MS that the main products of the oxidation of alkylphenols with iron(III) chloride are formed by the nucleophilic addition of the starting alkylphenols to intermediate quinone methides, and these products can be further oxidized to mixtures of oligomers [258]. This process is analogous to the oxidation of phenols in aqueous solutions with dissolved atmospheric oxygen, which made it possible to establish the mechanism of oxidation of natural flavonoids [259, 260]. Evidence was obtained to show that dialkyl phosphonates and trialkyl phosphites [261], as well as alkyl dichlorophosphates, dialkyl chlorophosphates, and their thioanalogs [262] can be reliably identified by their chromatography–mass spectrometry characteristics. The contribution of finer steric effects in the chromatographic parameters of amino acid derivatives was considered in [263]. Among the "usual" set of impurities of ethyl alcohol, vinyl ethyl ether and formaldehyde diethyl acetal were identified for the first time [264].

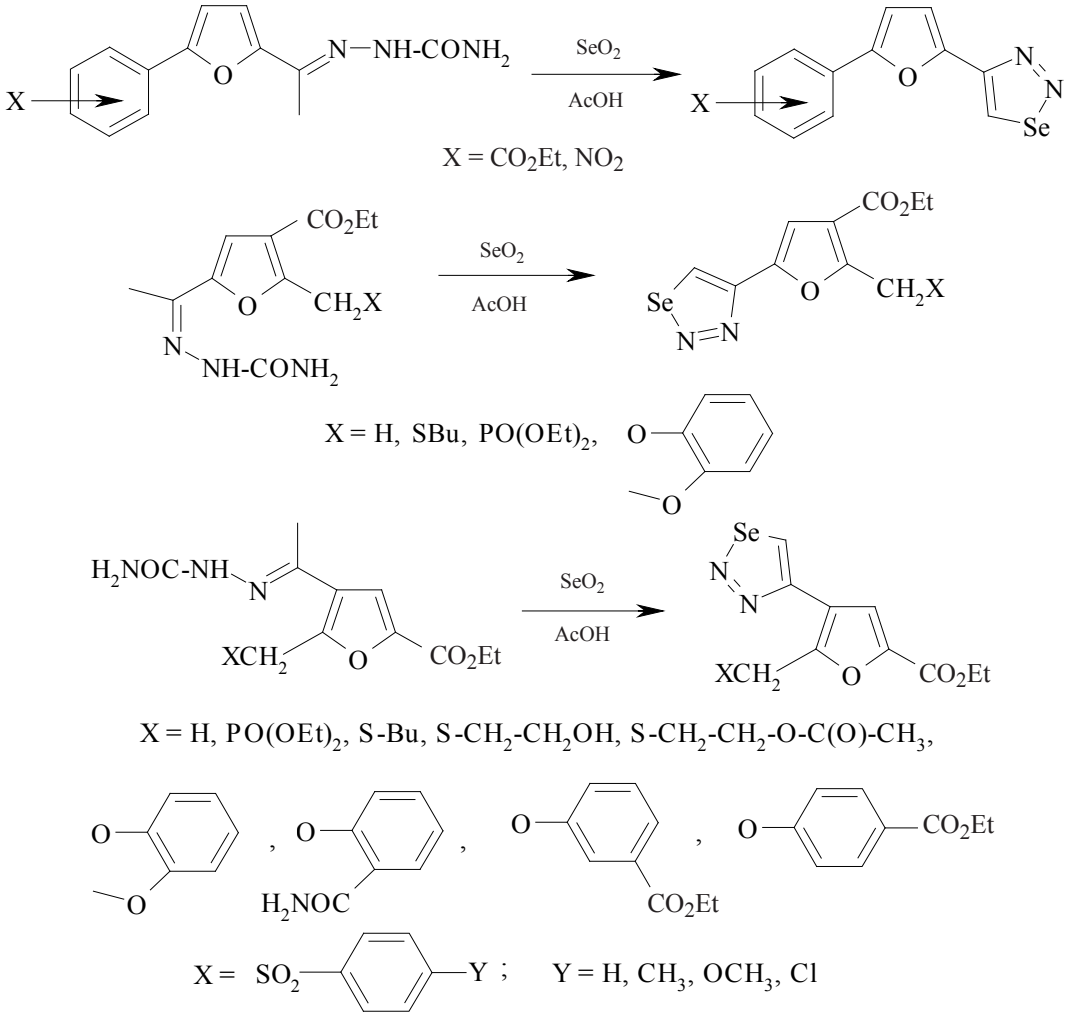
3. St. PETERSBURG STATE TECHNOLOGICAL INSTITUTE (TECHNICAL UNIVERSITY). HYBRID HETEROCYCLIC SYSTEMS DERIVED FROM FURAN, DIHYDROPYRAN, AND 1,2,3-THIA- AND -SELENADIAZOLE: SYNTHESIS AND FUNCTIONALIZATION

A combination of conjugated π-excessive and π-deficient heterocyclic fragments in one molecule leads to electron transfer within the system. As a result, a bipolar structure is formed, and the chemical properties of both heterocycles change noticeably. Such compounds are used in drug design, medical diagnostics, and as monomers for π-alternating polymers for organic semiconductors.

An example of such systems is represented by [1,2,3-thia(seleno)diazol-4-yl]furans. They are being successfully studied at the Department of Organic Chemistry, St. Petersburg State Technological Institute (Technical University) [SPbSTI(TU)]. We previously showed that to ensure the thermal stability of 2- and 3-(1,2,3-thiadiazol-4-yl)furans, it is necessary to introduce an acceptor substituent (an ester group or a phenyl ring with a nitro or an ester group) into the furan ring [265, 266]. It was found that the same is true of 2- and 3-(1,2,3-selenodiazol-4-yl)furans, which were synthesized by the oxidation of semicarbazones derived from the corresponding acetylfurans [267–270] with selenium dioxide in acetic acid (Scheme 3.1).

1,2,3-Selenodiazolylfurans are much less thermally stable than thiadiazoles. Therefore, to obtain functionally substituted compounds, functional groups should be introduced at stages preceding semicarbazone synthesis and selenodiazole ring formation. A valuable quality of selenodiazoles is their ability to slowly decompose with a release of hydrogen selenide under nearly physiological conditions. Therefore, they can be useful as drugs against the consequences of selenium deficiency.

Scheme 3.1.



Unlike the labile selenodiazolylfurans, their sulfur analogs can be involved in the targeted transformation of the 1,2,3-thiadiazole fragment into other functional groups. This transformation leads to furan derivatives, which are very difficult or impossible to obtain by other methods. Reactions that occur during cleavage of the 1,2,3-thiadiazole ring in the absence and in the presence of proton donors were studied on an example of ethyl 5-methyl-4-(1,2,3-thiadiazol-4-yl)furan-2-carboxylate (Scheme 3.2) [271].

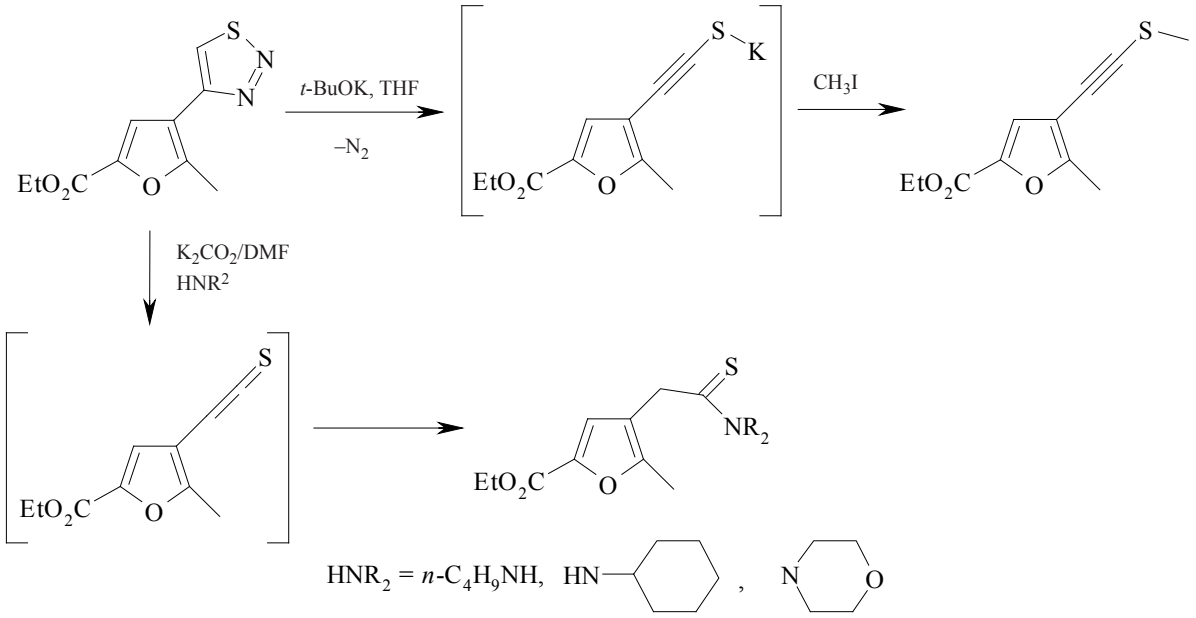
It turned out that in the presence of an amino group in the side chain, this reaction can also be carried out intramolecularly [272], as a 6-*endo*-dig-cyclization reaction (Scheme 3.3).

Alkylation of The resulting thionolactam is alkylated exclusively at sulfur, and in the reaction with phenacyl bromide, the thiazole ring that forms is oxidized by atmospheric oxygen to the tricyclic phenylfuro[3,2-*d*]-thiazolo[3,2-*a*]pyridin-4-ium salt.

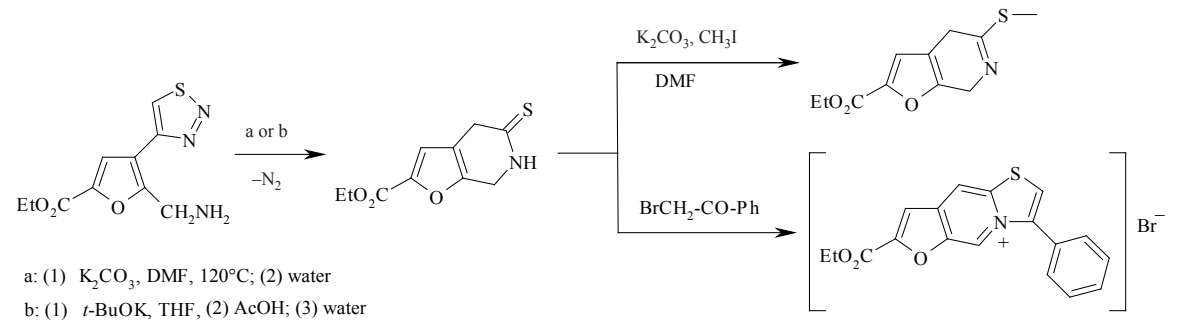
The formation of another hybrid heterocyclic π-donor–π-acceptor electron transfer system 3-furanylpyridazine was studied on an example of isomeric bromoacetyl derivatives of furylmethanecarboxylic acids [273]. These compounds were used to alkylate acetoacetic ester, the resulting 1,4-diketones were treated with hydrazine hydrate, and cyclic azines formed during the reaction underwent aromatization to pyridazines under the action of atmospheric oxygen. As a result, the reaction formed both 3-(furan-2-yl)- and 3-(furan-3-yl)-pyridazines containing a dialkoxyphosphorylmethyl substituent in the furan ring and an ester substituent in the pyridazine ring (Scheme 3.4).

Among annulated systems with charge transfer from the π-donor to π-acceptor part of the common conjugated

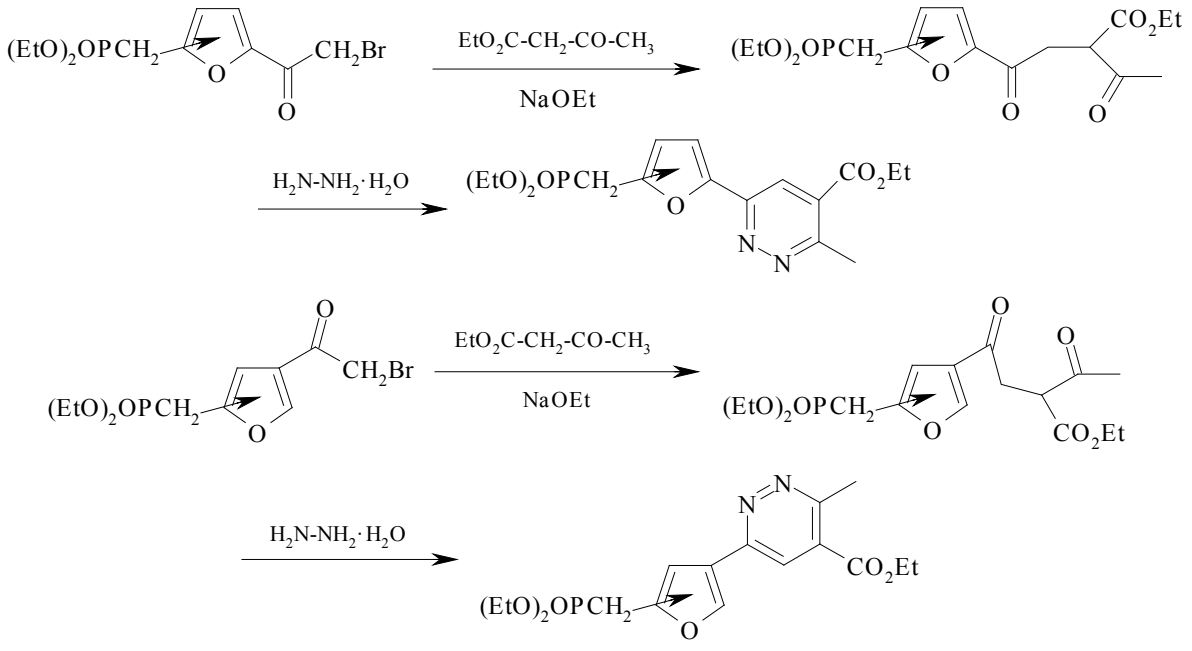
Scheme 3.2.



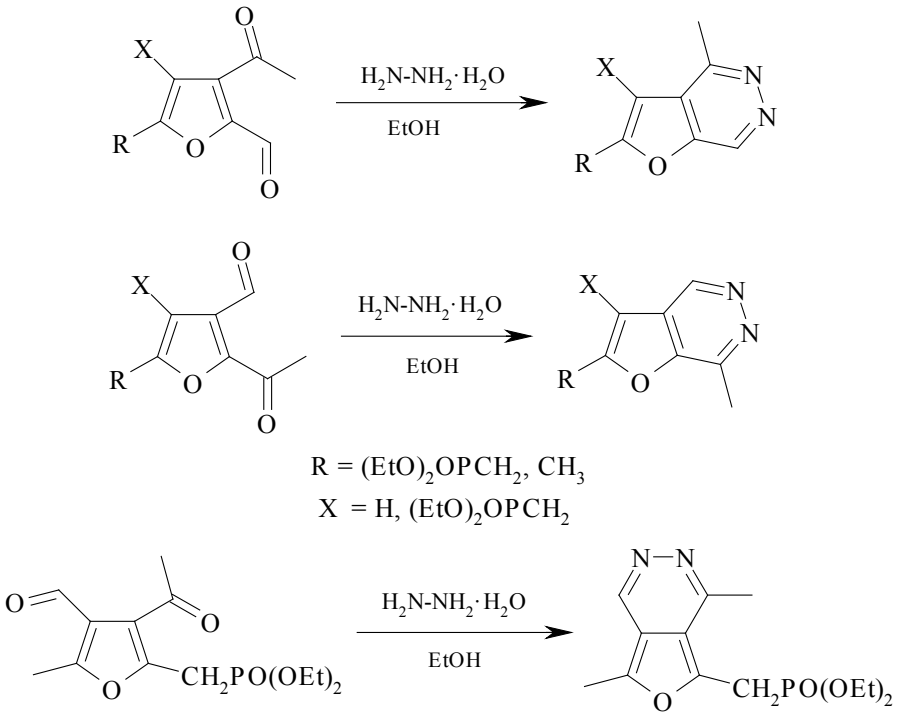
Scheme 3.3.



Scheme 3.4.



Scheme 3.5.



$R = (EtO)_2OPCH_2, CH_3$

$X = H, (EtO)_2OPCH_2$

system we studied for the first time phosphonomethylated furopyridazines [274]. The starting materials were phosphonomethylated formylacetylfurans, which were then treated with hydrazine hydrate to obtain the corresponding furo[2,3-*d*]- or furo[3,4-*d*]pyridazines, which have a phosphorus-containing substituent in the α- or β-position of the furan ring (Scheme 3.5).

Recently, increasing interest has been focusing on hybrid heterocyclic systems comprising annulated heteroaromatic and saturated heterocyclic medium-sized fragments. This interest is associated with a higher conformational mobility of such structures, which allows them to easier coordinate to biological substrates. A convenient method for the annulation of the dihydrothiapyran ring to the furan ring, due to which we were able to synthesize three hybrid systems with these heterocycles annulated in three different ways (Scheme 3.6) [275].

The example of ethyl 4-(diethoxyphosphoryl)-4,7-dihydro-5*H*-thiopyrano[3,4-*b*]furan-5-carboxylate to develop methods for the functionalization of positions 2 and 3 of this hybrid system. It was found that the dihydrothiapyran ring behaves as an acceptor substituent comparable in strength to the ester group in electrophilic substitution reactions in the furan fragment (Scheme 3.7) [276, 277].

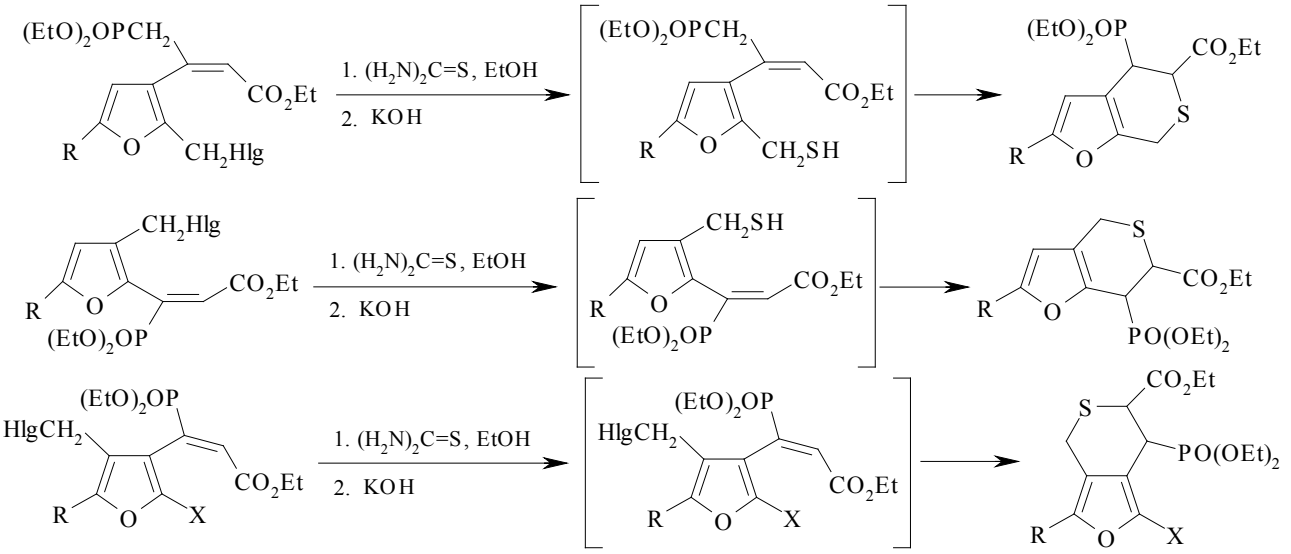
Thus, hybrid and annulated furan systems have become accessible compounds. This opens the way to previously unknown annulated systems comprising 1,2,3-thia- and selenadiazole, furan, and dihydropyran. The relative facility of their functionalization allows us to expect to find, among compounds of this series, suitable platforms for creating new generation drugs to replace bacteria- and virus-resistant ones.

4. ELECTROPHILIC ACTIVATION IN THE CHEMISTRY OF UNSATURATED AND HETEROCYCLIC COMPOUNDS. ORGANIC CHEMISTRY AT THE St. PETERSBURG STATE FOREST TECHNICAL UNIVERSITY

The electrophilic activation of organic compounds under the action of strong Brønsted acids (CF_3SO_3H, FSO_3H, HF, H_2SO_4, etc.), Lewis acids ($AlCl_3$, $AlBr_3$, SbF_5, etc.), and acidic zeolites results in the generation of highly reactive cationic species that can take part in various regio- and stereoselective transformations. The use of electrophilic activation in the chemistry of unsaturated compounds (alkenes, alkynes, allenes) and heterocycles allows the targeted synthesis of new organic compounds.

Nitroethylenes **1** containing electron-acceptor substituents X = CX_3 (X = F, Cl, Br) or X = CO_2Alk

Scheme 3.6.



R = H, Br, CH_3, *t*-Bu, X = H, Br

(nitroacrylates, Alk = Me, Et) in the vicinal position react with arenes in trifluoromethanesulfonic acid CF_3SO_3H (TfOH) to form diaryl-substituted oximes **2** [278, 279]. The reactions of nitroethylenes **1** first with arenes and then with nitriles gave 1,2,4-oxadiazoles **3** (Scheme 4.1) [280].

Trifluoromethylated allyl alcohols or their TMS ethers **4** in H_2SO_4 undergo quantitative cyclization to indenes **5**, which are isomerized to indenes **6** during chromatographic separation on silica gel (Scheme 4.2) [281]. Compounds **4** react stereoselectively with arenes in TfOH to form *trans*-indanes **7** [282]. Similar transformations of analogs of CF_3-substituted alcohols **4**, which contain one or two bromine atoms at the double bond, gave the corresponding alkenes or indenes [283].

Electrophilic transformations of various CCl_3-substituted conjugated enones **8** (R = Ar, Me) in TfOH can lead to intramolecular cyclization to give indanones **9** (for compounds **8** with R = Ar) [284] or, in reactions with arenes, indenes **10** (for compounds **8** with R = Me) (Scheme 4.3) [285]. Similarly, brominated CF_3-enones in TfOH form CF_3-indenes [286].

The reactions of dienones **11** with arenes involve multistage cationic tranflormations and lead to indene **12**, primarily *cis* isomers (Scheme 4.4) [287].

Conjugated diene acid **13** reacts with arenes in fluorosulfonic acid FSO_3H at a low temperature (–78°C) to form bicyclic ketones **14** (Scheme 4.5) [287].

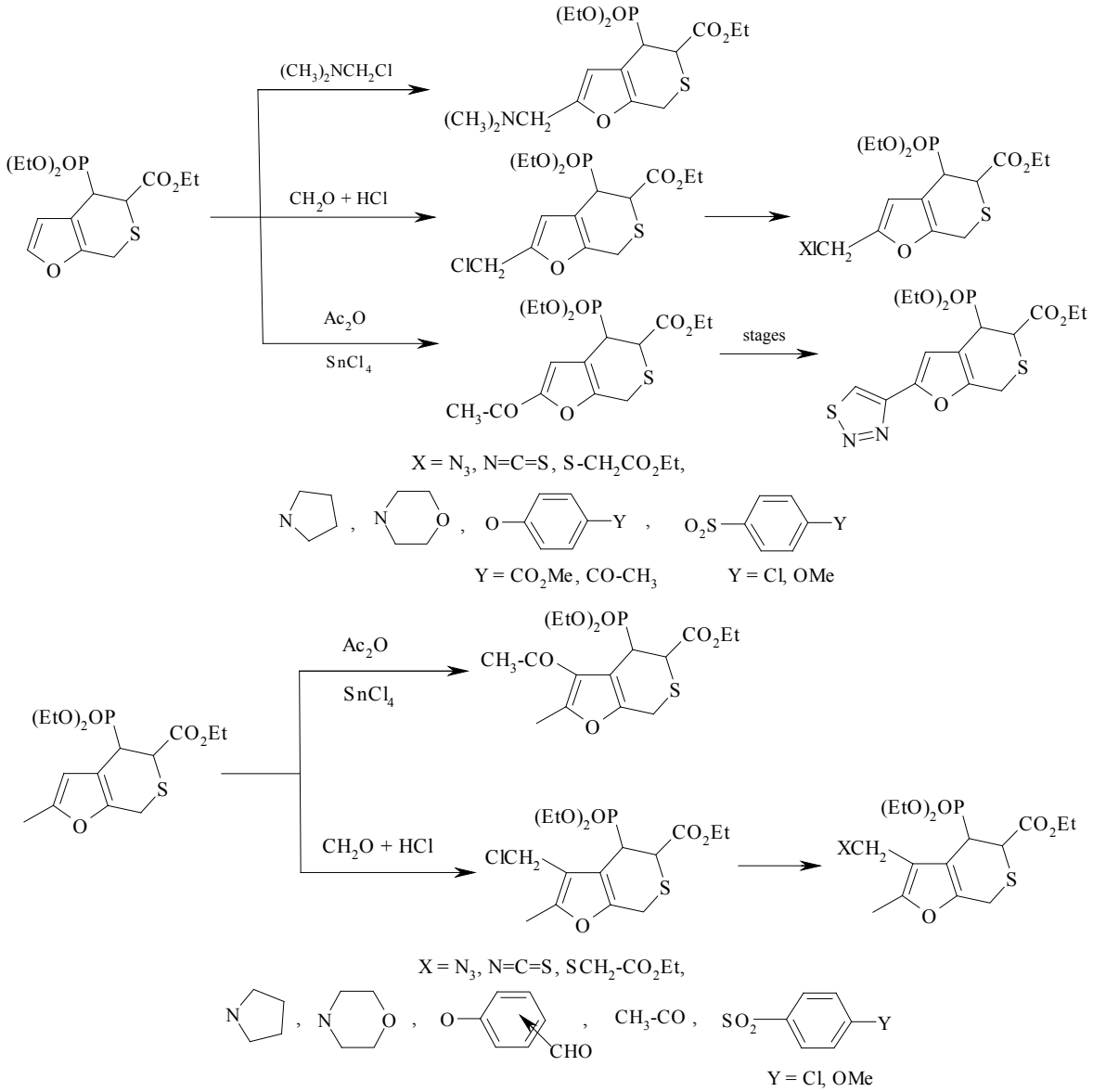
The reactions of cinnamic acid nitriles **15** with arenes under the action of TfOH or $AlBr_3$ involve the initial hydroarylation of the C=C bond to form compounds **16** and the subsequent cyclization of the latter to indanones **17** (only with TfOH) (Scheme 4.6) [288].

Electron-deficient allenes **18** with various acceptor substituents (P=O, S=O, or C=O) are protonated in strong Brønsted acids with the intermediate generation of species **19**, which then undergo cyclization to give stable cations **20**. The formation of the latter was detected by NMR [289–291]. Under the action of nucleophiles in acidic reaction solutions (hydrolysis and other nucleophilic reactions), cation **20** convert into derivatives of dihydro-1,2-oxaphosphole 2-oxide **21** [289, 290], thiochromene 1,1-dioxide **22** [291], and furanone **23** (Scheme 4.7) [292]; in addition, other products can also be obtained [289–291]. Structural analogs of furanones **23** are also formed by reactions of alkyl 4-hydroxybut-2-ynoates [>C(OH)–C≡C–CO_2Alk] with arenes under the action of TfOH or acidic zeolites [293].

A variety of alkynes can be successfully involved in electrophilic activation processes to synthesize new organic compounds. Thus, *O*- and *S*-aryl esters of 3-arylpropynoic acids **24** undergo cyclization to coumarins **25** (X = O, S) under the action of acidic zeolites (Scheme 4.8) [294, 295].

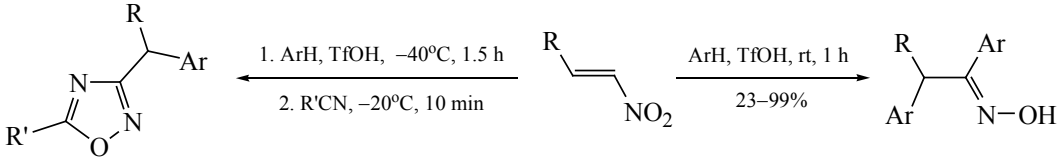
The one-pot tandem reactions of alkyl esters and amides of 3-arylpropynoic acids **26** with arenes (initial hydroarylation) and cyclohexane (a source of hydride

Scheme 3.7.



ions for the subsequent hydrogenation of the C≡C bond) in the presence of the Lewis acid $AlCl_3$ result in the formation of 3,3-diarylpropanoic acid derivatives [296]. However, these reactions are complicated by the exchange of the aryl groups Ar and Ar' in the resulting target compounds **27** under electrophilic conditions (Scheme 4.9). Conjugated acetylenic ketones enter the same reactions [296].

Scheme 4.1.



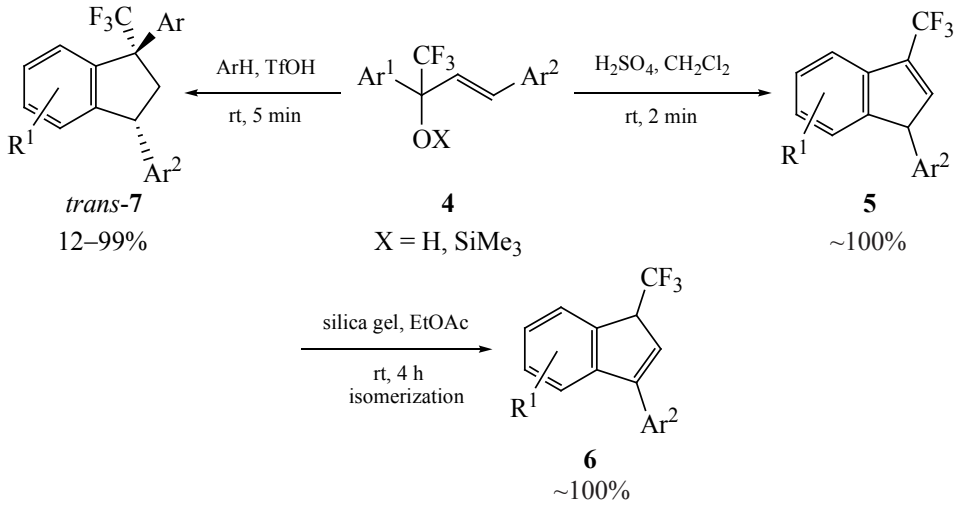
3
28–96%

1

2
23–99%

R = CX_3 (X = F, Cl, Br), CO_2Alk (Alk = Me, Et)

Scheme 4.2.



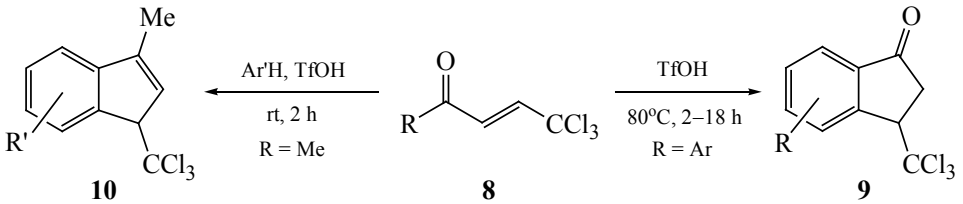
trans-**7**
12–99%

4
X = H, $SiMe_3$

5
~100%

6
~100%

Scheme 4.3.



10
20–47%

8

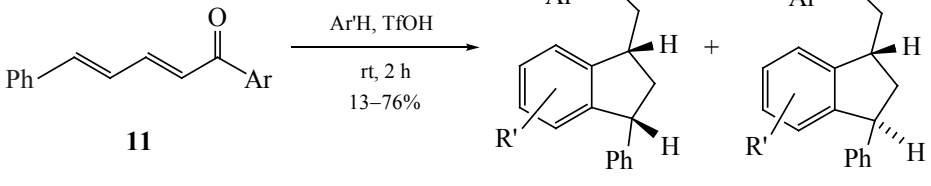
9
13–92%

Trifluoromethyl-substituted conjugated acetylenic ketones **28** react with arenes under the action of TfOH or acidic zeolites HUSY, leading to CF_3-indenes **29** (Scheme 4.10) [297].

Under electrophilic activation conditions, CF_3-propargyl alcohols **30** can be used to generate resonance propargyl–allenyl cations **31**, whose reactions with aromatic nucleophiles give isomeric CF_3-indenes **32**. Such reactions occur both in TfOH [298] and under the action of acidic zeolites [299]. The formation of one or another isomer of indene **32** depends on the electronic structure and reactivity of cation **31** (Scheme 4.11), as well as the nucleophilicity of the aromatic rings Ar, Ar' and Ar'' [298].

Cross-conjugated enynones **33** in H_2SO_4 at room temperature slowly (within 60 h) transform into dihydropyran-4-ones **34** [300]. In stronger and less nucleophilic acid systems (TfOH, TfOH–pyridine, zeolites), substrates **33** can be subjected to consecutive hydroarylation first of the C≡C bond to form compounds **35** and then of the C=C bond to obtain the final compounds **36** (Scheme 4.12). Linearly conjugated 1,5-diarylpent-4-en-2-yn-1-ones [Ar–CH=CH–C≡C–C(=O) Ar'] in the same transformations in TfOH give

Scheme 4.4.

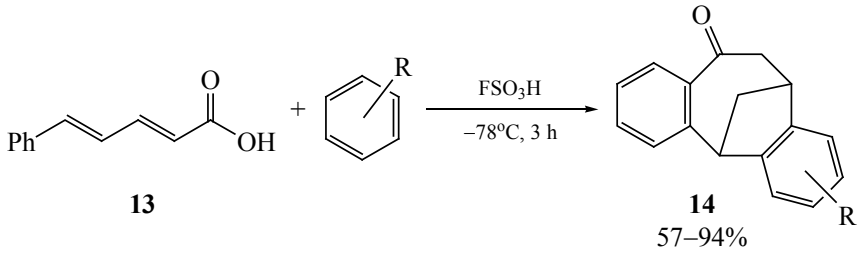


11

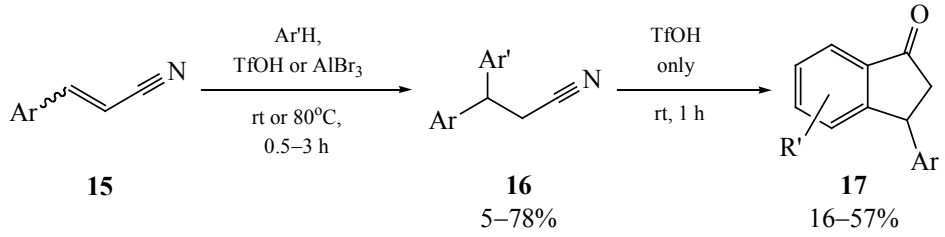
cis-**12** *trans*-**2**

cis- : *trans*-, 1–19 : 1

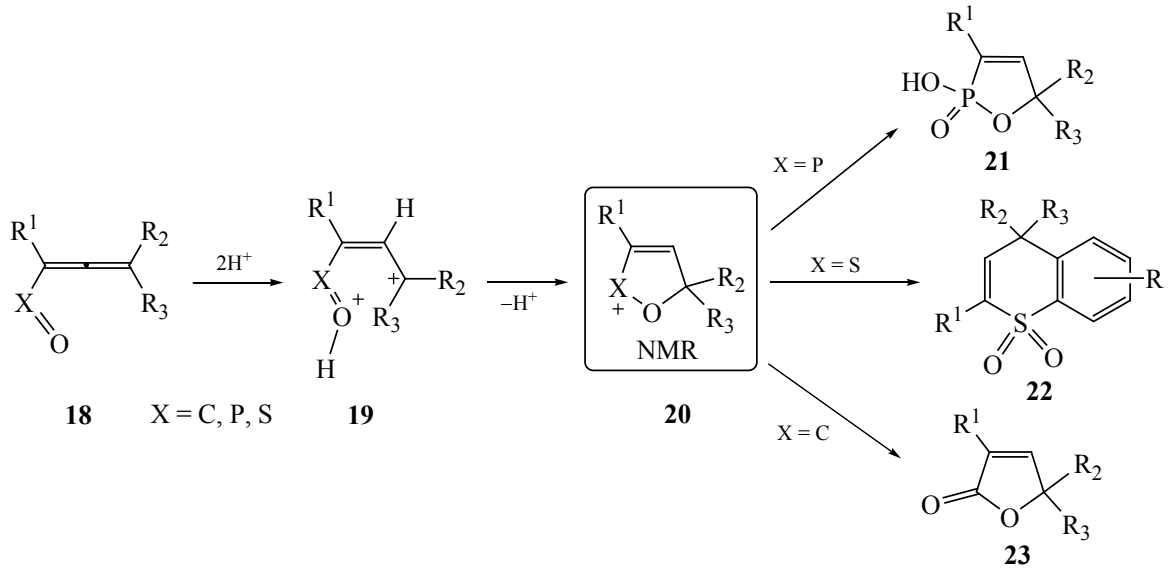
Scheme 4.5.



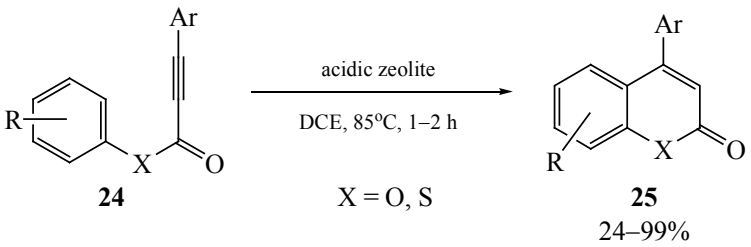
Scheme 4.6.



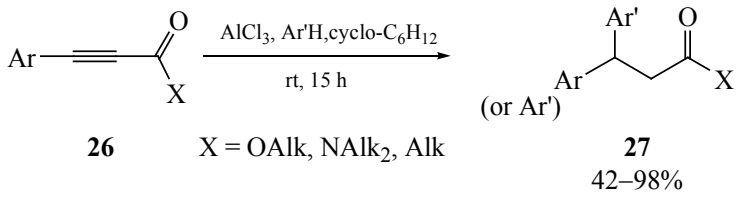
Scheme 4.7.



Scheme 4.8.



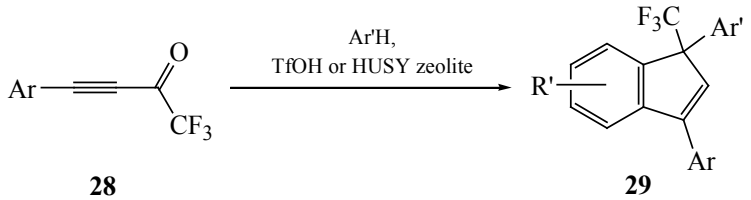
Scheme 4.9.



26 X = OAlk, $NAlk_2$, Alk **27**

42–98%

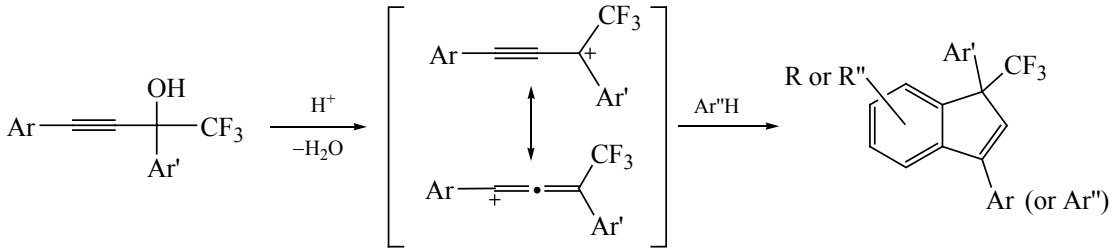
Scheme 4.10.



28 **29**

44–81%

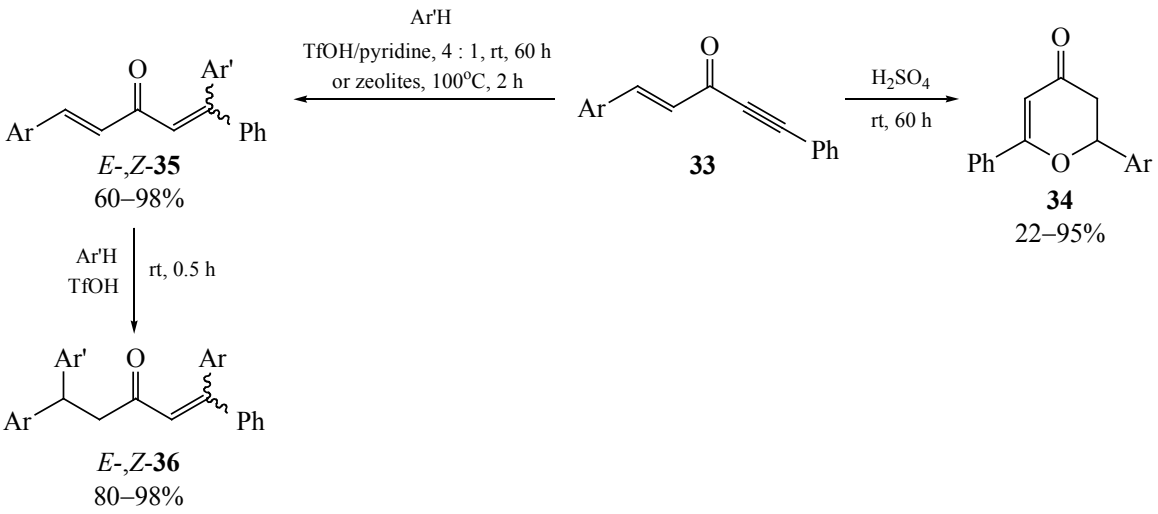
Scheme 4.11.



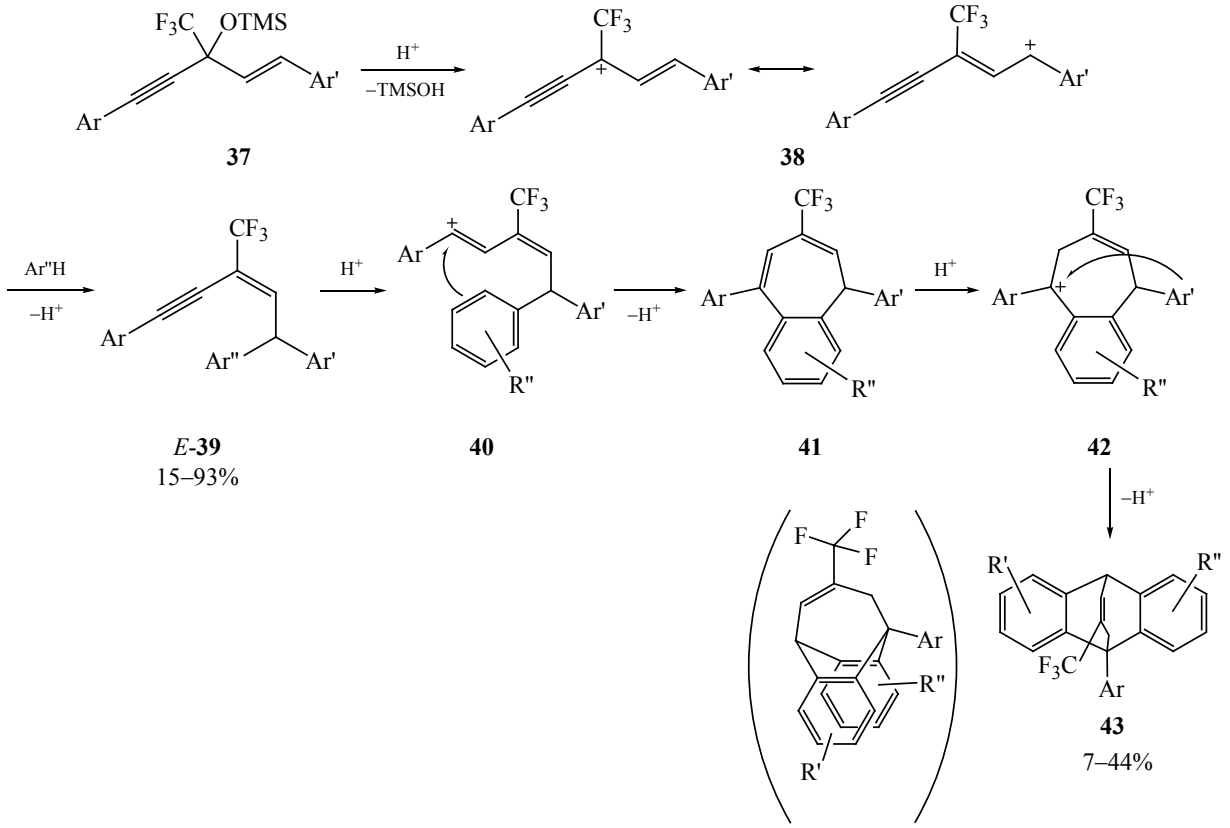
30 **31** **32**

36–98%

Scheme 4.12.



Scheme 4.13.



dihydropyranones **34**, and reactions with arenes give 1-alkylideneindanes [302].

Trimethylsilyl ethers of propargyl allyl alcohols **37** in TfOH convert into cross-conjugated resonance propargyl allenyl cations **38**, whose reactions with arenes occur regio- and stereoselectively, leading to enynes *E*-**39** [303]. The reaction can be stopped at the stage of formation of the latter products, when it is performed with a small quantity of TfOH (1.5 equiv) and for a short time (5–30 min). At the same time, when an excess of TfOH is used, the starting TMS esters **37** or enynes **39** further convert into bicyclic structures **43** via intermediates **40–42** (Scheme 4.13) [303].

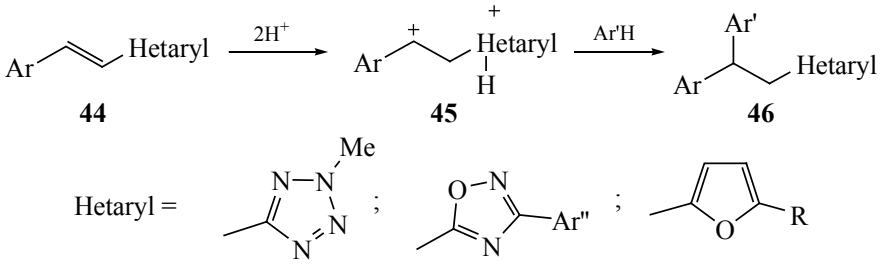
Electrophilic activation is of great importance in the chemistry of heterocyclic compounds. Using this synthetic approach, one can construct a variety of heterocycles (see the above-mentioned syntheses of compounds **3**, **21–23**, **25**, and **34**). In addition, effective structural modification of both the heterocyclic system itself and its side substituents is possible. Thus, a variety of alkenyl-substituted heterocycles of the tetrazole [304], 1,2,4-oxadiazole [305], and furan [306, 307] series of general structure **44** undergo regioselective hydroarylation by the exocyclic C=C bond in reactions with arenes under the action of TfOH, AlX_3 (X = Cl, Br), or acidic zeolites to form products **46** in high yields. The reactions occur via the intermediate generation of reactive dications **45** as the result of the protonation of the heteroatom and the carbon atom of the C=C bond (Scheme 4.14). Furan derivatives **46** showed good antimicrobial properties [306, 307].

Similarly, alkynyl-substituted tetrazoles and oxadiazoles **47** [308, 309] react with arenes in TfOH involve hydroarylation of the C≡C bond to form products **48** as an *E*/*Z* isomer mixture (Scheme 4.15).

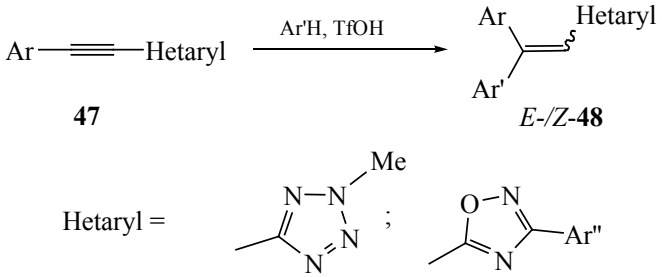
The aldehyde groups in benzimidazole and quinoline carbaldehydes **49** [310, 311] can be electrophilically activated by the protonation of both the C=O group and the heterocycles. As a result, in the reactions of hetaryl carbaldehydes **49** with arenes the intermediate cationic species give diarylmethyl derivatives **50** of the heterocycles (Scheme 4.16).

The reactions of 5-hydroxy-1-pyrrolines **51** with arenes in TfOH form 5-arylpyrrolines **52** as the result

Scheme 4.14.



Scheme 4.15.



of the structural modification of the heterocyclic system itself [312]. In Brønsted acids TfOH or H_2SO_4, pyrrolines **51** undergo dehydration and quantitatively convert into 3*H*-pyrroles **53** (Scheme 4.17) [311].

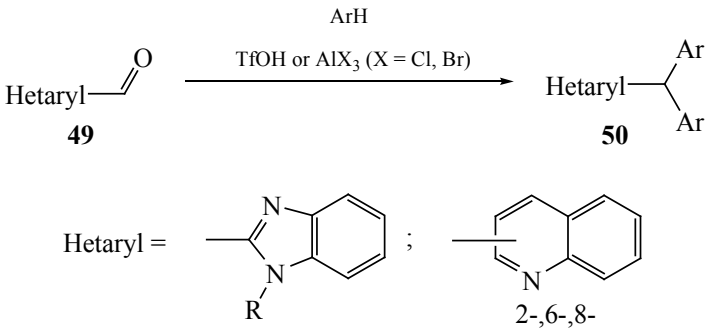
The protonation of diazohomophthalimides **54** leads to the generation of reactive diazonium cations **55**, which can react with nucleophiles [313]. In electrophilic substitutions with arenes, cations **55** give arylated derivatives **56**. Reactions in the HF–pyridine system lead to monofluorinated homophthalimides **57**, whereas the use of the HF–pyridine–*N*-bromo(or chloro)succinimide system allows one to obtain fluoro–bromo(or chloro)-substituted substrates **58** (Scheme 4.18) [313].

The reactions of trimethylsilyl ethers of CF_3-substituted α-hetarylalkanols **59** with arenes in TfOH provide, depending on the structure of the substrates and the reaction conditions (amount of acid, time, and temperature), diverse thiophene and benzofuran derivatives (Scheme 4.19) [314, 315]. The products are formed by the following reactions: arylation of the side chain and heterocyclic system, ionic hydrogenation, hydrodehalogenation and aryldehalogenation in the heterocycle, etc. The resulting CF_3-substituted thiophenes and benzofurans, as well as the previously reported CF_3-indanes [316], showed different types of antimicrobial activity [315, 316].

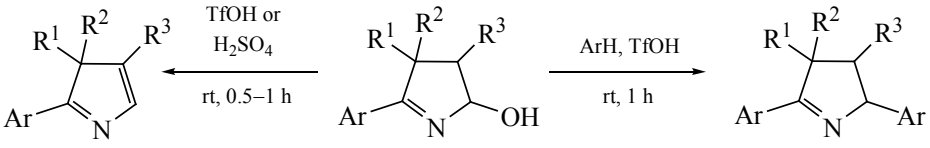
5. DEPARTMENT OF ORGANIC CHEMISTRY OF LOMONOSOV MOSCOW STATE UNIVERSITY

Seven laboratories of the Department of Organic Chemistry of the Faculty of Chemistry of Lomonosov Moscow State University conduct research in a wide variety of areas of organic chemistry and related disciplines.

Scheme 4.16.



Scheme 4.17.

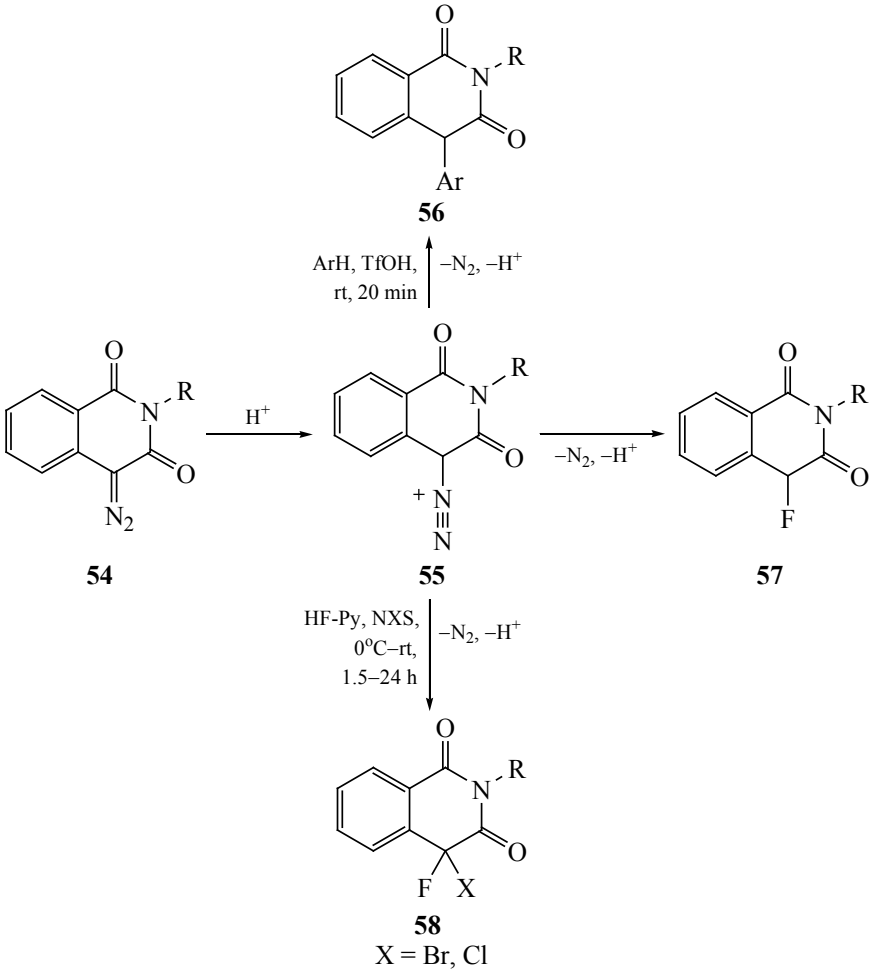


53 ~100% **51** **52** 55–98%

Laboratory of Organoelement Compounds (**head Academician of the Russian Academy of Sciences, Professor I.P. Beletskaya**). Over the past years, the laboratory headed by Acad. Beletskaya has conducted its main research in two areas: metal-catalyzed addition reactions at multiple bonds and catalytic substitution reactions. In addition, a number of catalytic processes have begun to be carried out under visible light irradiation under photoredox catalysis conditions. First of all, a series of studies on divergent addition reactions to activated alkynes is worth noting [317–319]. For example, the reactions with the same reagents, *o*-(trifluoroacetyl)anilines and propiolates in the presence of silver triflate selectively produce quinoline-3-carboxylates. The replacement of silver with copper and addition of a base leads the formation of indoline derivatives. Finally, the use of phosphine catalysis gives rise to isomeric quinoline-2-carboxylates (Scheme 5.1).

To solve the problem of irretrievable scattering of the expensive platinum catalyst used in the synthesis of organosilanes, two-phase systems were proposed, which allow the hydrophobic layer of the resulting organosi-

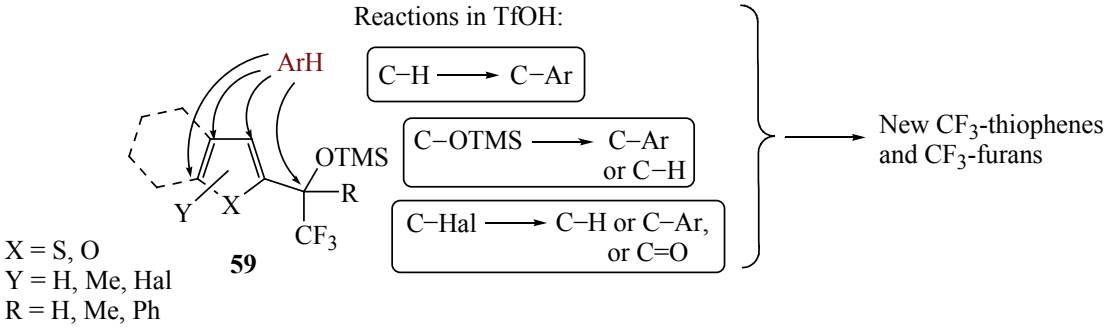
Scheme 4.18.



56 **54** **55** **57** **58**

X = Br, Cl

Scheme 4.19.

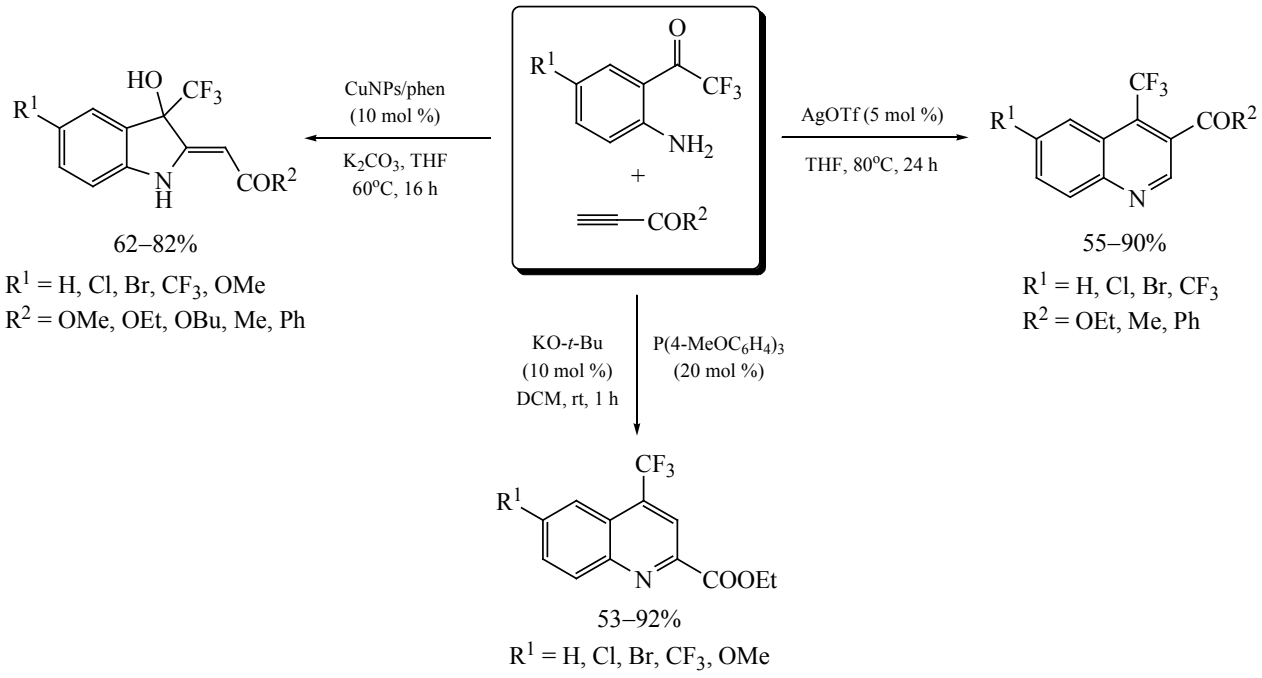


lane to be separated from the polar layer containing platinum after the reaction has been completed. These catalytic systems make it possible to implement hydrosilylation of alkenes and alkynes with very high yields and almost quantitative anti-Markovnikov selectivity [320]. The K_2PtCl_4 system (without additional ligand) – ethylene glycol (Scheme 5.2) has proven itself best in this process.

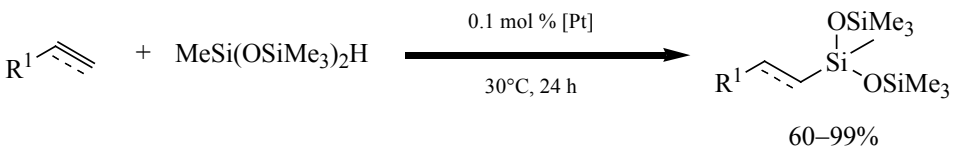
An important place in the research is occupied by photocatalytic and photoinitiated reactions, for example, the reaction of direct photoinitiated iodosulfonation of disubstituted acetylenes with tosyl iodide, which regioselectively occurs as trans-addition, has been studied [321]. Under optimized conditions, symmetrical diaryl- and alkylaryl-acetylenes form adducts with yields ranging from high to nearly quantitative, and the reaction occurs regioselectively (Scheme 5.3).

Photoredox processes involving Ru(II) complexes were studied on an example of the trifluoroethoxylation of styrenes with diazonium salts (photoredox-catalyzed Meerwein reaction) (Scheme 5.4) [322]. It was shown

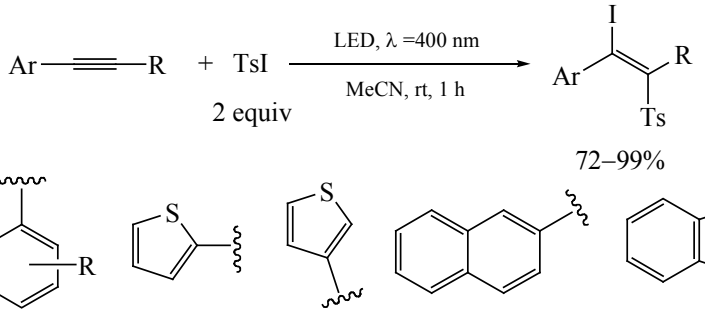
Scheme 5.1.



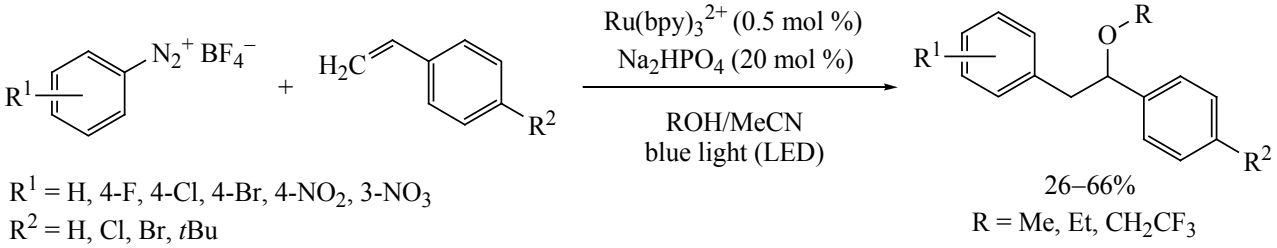
Scheme 5.2.



Scheme 5.3.



Scheme 5.4.

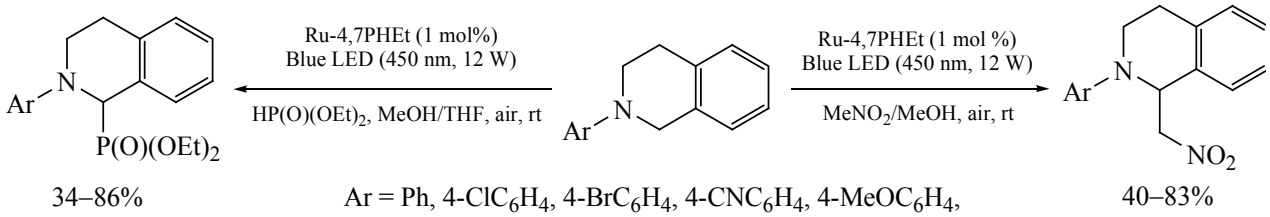


that the yields with this catalyst are generally not lower and in some cases even higher than the yields obtained in the presence of the classical $Ru(bpy)_3^{2+}$ catalyst. The Ru-PhenC catalyst with phenanthroline-3,8-dicarboxylic acid as a ligand can be easily separated from the reaction products during processing the reaction mixture and used to success in 5 cycles without significant loss of yield.

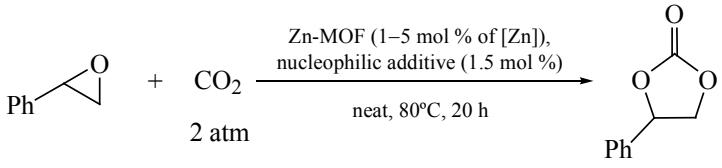
A method for the synthesis of new Ru(II) complexes with a phenanthroline ligand containing phosphonate substituents in positions 3 and 8 (**Ru-3,8PHEt**) and 4 and 7 (**Ru-4,7PHEt**) was developed [323]. The catalytic activity of the synthesized complexes was studied on an example of the α-functionalization reactions (nitromethylation, phosphonation, and cyanation) of the model *N*-phenyl-1,2,3,4-tetrahydroisoquinoline with various nucleophiles under visible light irradiation (Scheme 5.5). The catalyst could be recycled up to 7 times.

The performance of Zn-containing metal-organic framework polymers (MOFs) based on 2,5-thiophenedicarboxylic acid, 1,4-diazabicyclo[2.2.2]octane, and polyols (Zn-NIIC-10) was assessed in epoxide carboxylation reactions [324]. The use of these catalysts in the synthesis of cyclic carbonates from mono- and disubstituted epoxides in high yields was

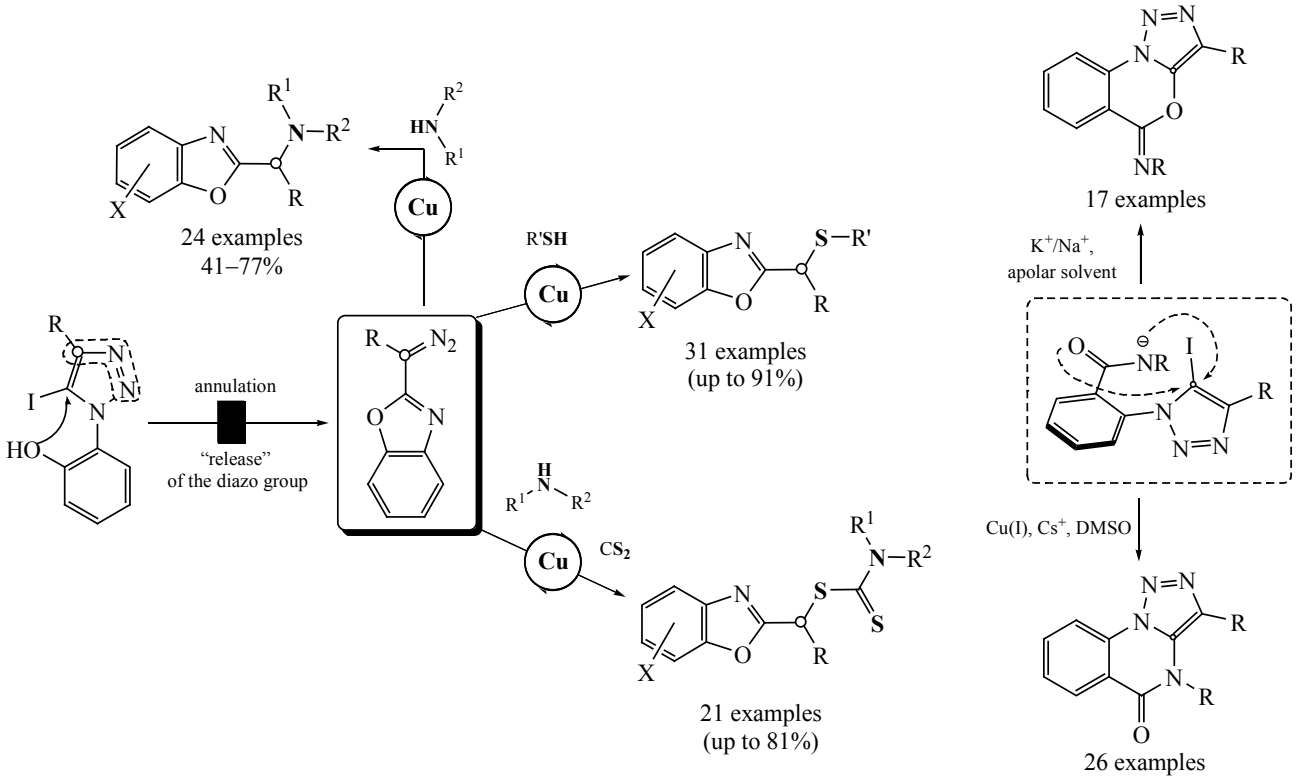
Scheme 5.5.



Scheme 5.6.



Scheme 5.7.



demonstrated (Scheme 5.6) and recyclability of up to five consecutive cycles without loss of catalytic activity was demonstrated. Another Zn-MOF catalyst based on bis-1,1'-1,2,3-benzotriazolylmethane (Bbtm), too, turned out to be effective in reactions of monosubstituted epoxides with CO_2 [325].

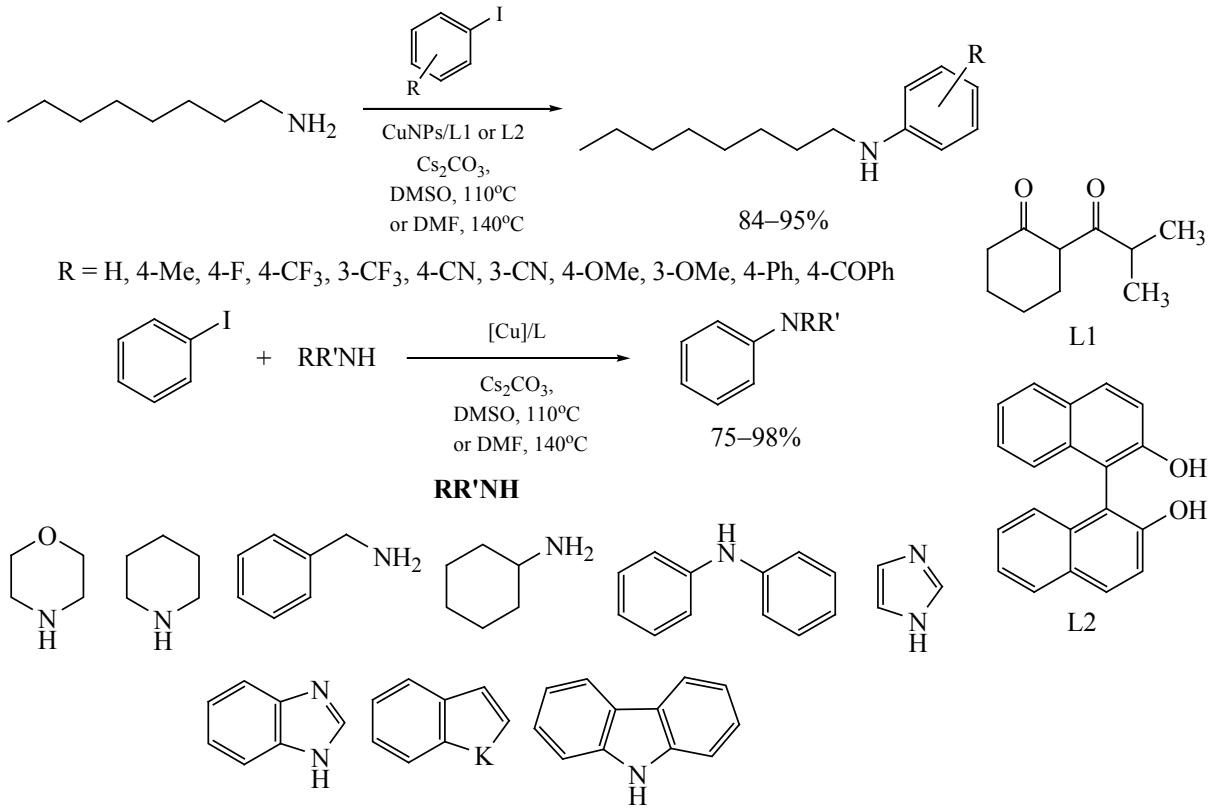
The azide–alkyne cycloaddition reaction involving iodoalkynes and various aryl azides was used to obtain a wide range of *N*-aryl-substituted 5-iodotriazoles, precursors for the synthesis of fused heterocyclic systems [326]. Due to the tautomeric equilibrium with the open diaza form, further copper-catalyzed reactions of carbene insertion into the N–H and S–H bonds can be carried out (Scheme 5.7) [327, 328]. As shown on an example of benzamides, cyclizations involving ambident nucleophiles could be carried out chemoselectively at both the O- and N-reaction centers (Scheme 5.7) [329].

An extensive study of C–C, C–S, and C–N bond formation was carried out using copper nanoparticles (CuNPs) immobilized on various substrates [327]. As part of the study of the possibility to use free nanoparticles of copper and its oxides (Cu_2O NPs and CuO NPs), a series of catalytic arylations of various amines were performed [330, 331]. On an example of the model reaction of *n*-octylamine with iodobenzene, it was established that a high yield of the arylation product (up to 95%) is provided by nanoparticles of various sizes and compositions in the presence of the ligands 2-isobutyrylcyclohexanone (L1) and 1,1'-bi-2-naphthol (L2); reactions with iodobenzene derivatives containing electron-donor and electron-acceptor substituents in the *para* and *meta* positions were successfully implemented (product yields 84–93%) (Scheme 5.8).

Both aliphatic amines (morpholine, piperidine, benzylamine, and cyclohexylamine) and NH-heterocycles (product yields up to 98%), too, can undergo this reaction. The catalytic system CuNPs (25 nm)/L1 can be reused in at least 9 consecutive arylations with only a slight drop in the yield of the reaction product.

For many years, the Laboratory of Organoelement Compounds has been successfully conducting research on the synthesis of macrocyclic and macropolycyclic compounds by the Pd(0)-catalyzed amination of halo (hetero)arenes with oxadiamines and polyamines (Scheme 5.9) [332, 333]. As a result, numerous compounds were obtained, including those with axial [based on (*S*)-BINAM] and planar (based on

Scheme 5.8.

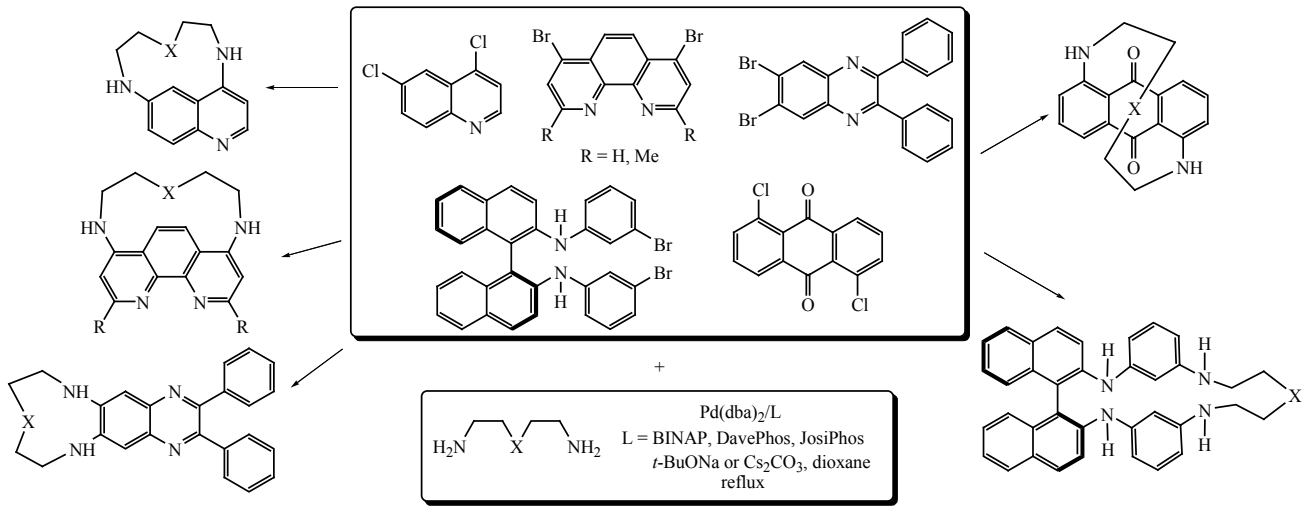


1,5-disubstituted anthracene and anthraquinone) chirality.

Methods for the synthesis of fluorescent detectors of metal cations and optically active organic compounds, which are open-chain derivatives of (*S*)-BINAM [334] and macrocyclic [335] and macrobicyclic [336] compounds containing fluorophoric substituents (Fig. 5.1).

The Laboratory of Organic Synthesis (**head Professor V.G. Nenajdenko**) deals with a wide range of synthetic problems. The research topics of the laboratory include the development of new methods for the

Scheme 5.9.



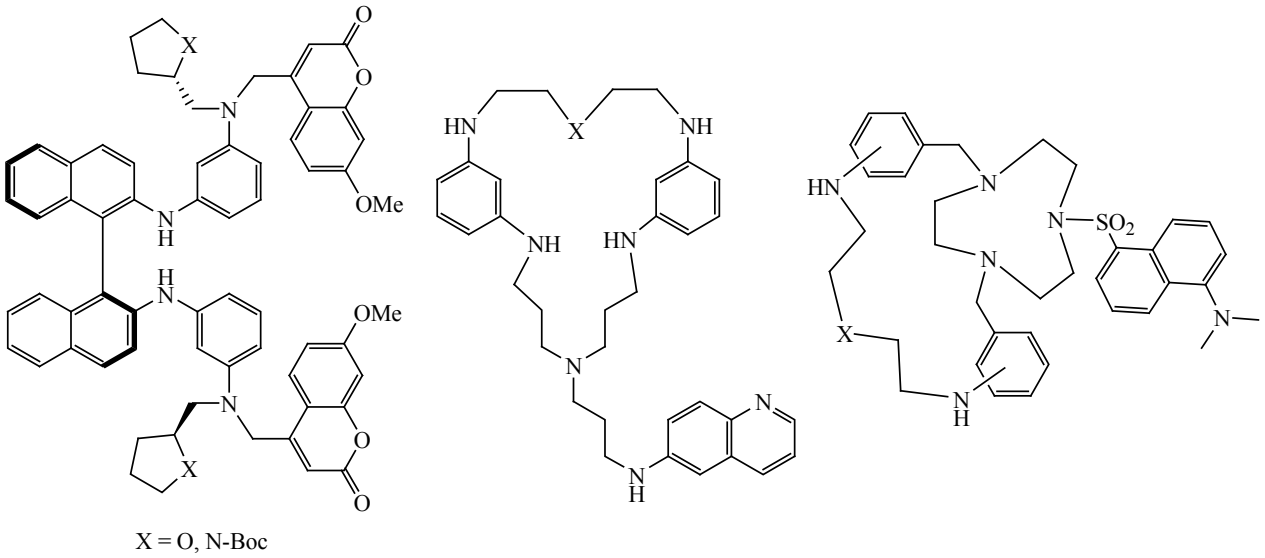


Fig. 5.1. Examples of fluorescent detectors based on (*S*)-BINAM and macrocyclic and macrobicyclic compounds.

synthesis of practically important compounds (ligands for processing of spent nuclear fuel), catalysis (metal complex, asymmetric, organocatalysis), the chemistry of fluorine-containing compounds, the chemistry of heterocyclic compounds, multicomponent reactions of isocyanides, and quantum-chemical calculations.

Organofluorine compounds play an extremely important role in modern organic and medicinal chemistry, agrochemistry, and materials chemistry. The use of fluorinated building blocks is a very convenient approach and in many cases an indispensable alternative to late-stage fluorination for creating structures with a strictly specified position of the fluorine atom. As a result of collaboration with colleagues from the Institute of Organic Chemistry of the Russian Academy of Sciences, a stereoselective approach to β-fluoro-β-nitrostyrenes was developed [337], based on the radical nitration and debromination of the corresponding β-bromo-β-fluorostyrenes (Scheme 5.10) [338].

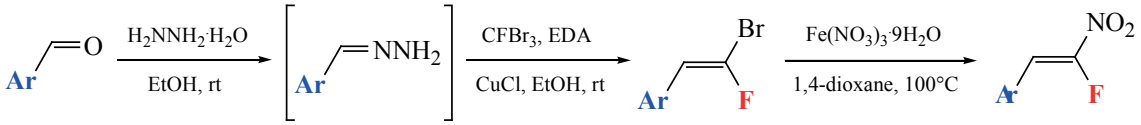
These compounds are effective dienophiles, electrophiles, and dipolarophiles, which opens up access to the creation of a wide range of new organofluorine compounds. For example, The Diels-Alder reaction of β-fluoro-β-nitrostyrenes with open-chain dienes formed the basis for the development of an approach to monofluorinated derivatives of cyclohexane and biphenyl (Scheme 5.11) [339, 340].

In turn, the Diels–Alder reaction of β-fluoro-β-nitrostyrenes with cyclopentadiene opened up access to new monofluorinated norbornenes [341], norbornadienes, and bicyclic fluoroanhydrides (Scheme 5.12) [342].

Another type of reactions characteristic of β-fluoro-β-nitrostyrenes is the conjugate Michael addition. For example, they react with pyrroles or indoles in catalyst- or solvent-free conditions at various temperatures depending on the reactivity and physical properties of the initial substrates [343]. Nitrous acid is easily eliminated from the resulting Michael adducts under the action of 1,8-diazabicyclo[5.4.0]-undec-7-ene, yielding new monofluorinated vinyl derivatives of pyrroles [344] and indoles [345] (Scheme 5.13).

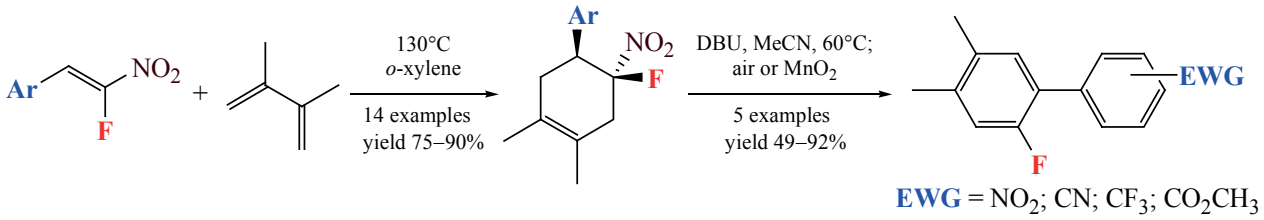
However, the possibility of creating new fluorinated heterocycles from β-fluoro-β-nitrostyrenes is the most important and sought-after area of their application.

Scheme 5.10.

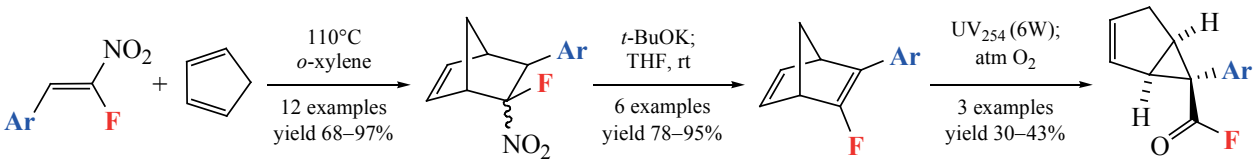


EDA = 1,2-ethylenediamine up to 95% up to 92%, only Z-isomer

Scheme 5.11.



Scheme 5.12.



For example, the reactions of β-fluoro-β-nitrostyrenes with sodium azide gave new monofluorinated triazole derivatives [346, 347]. Furthermore, we developed a synthetic approach to 4-fluoropyrazoles through the oxidative annulation of β-fluoro-β-nitrostyrenes with hydrazones generated in situ [348]. The enormous synthetic potential of β-fluoro-β-nitrostyrenes was demonstrated in the assembly of more complex bicyclic systems (Scheme 5.14).

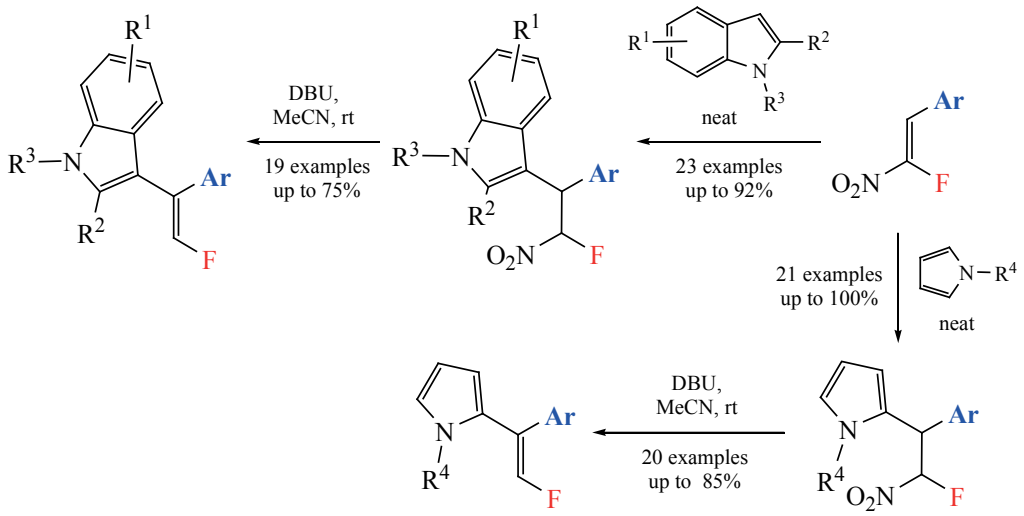
For example, a series of fluorinated indolizines were prepared by the copper(II) acetate–catalyzed oxidative [3+2]-annulation of β-fluoro-β-nitrostyrenes with in situ generated pyridinium ylides [349]. Similarly, monofluorinated pyrrolo[1,2-*b*]pyridazines [350] and pyrazolo[1,5-*a*]pyridines [351] were obtained from the corresponding dipoles. In addition, a wide range of nitrosoacetals were synthesized from various β-fluoro-β-nitrostyrenes, halogenated dicarbonyl compounds, and dipolarophiles of different electronic natures [352].

In turn, the use of azomethine ylides as dipoles in [3+2]-cycloaddition reactions to β-fluoro-β-nitrostyrenes opened up access to new monofluorinated pyrrolidines, pyrrolines, and pyrroles (Scheme 5.15) [353].

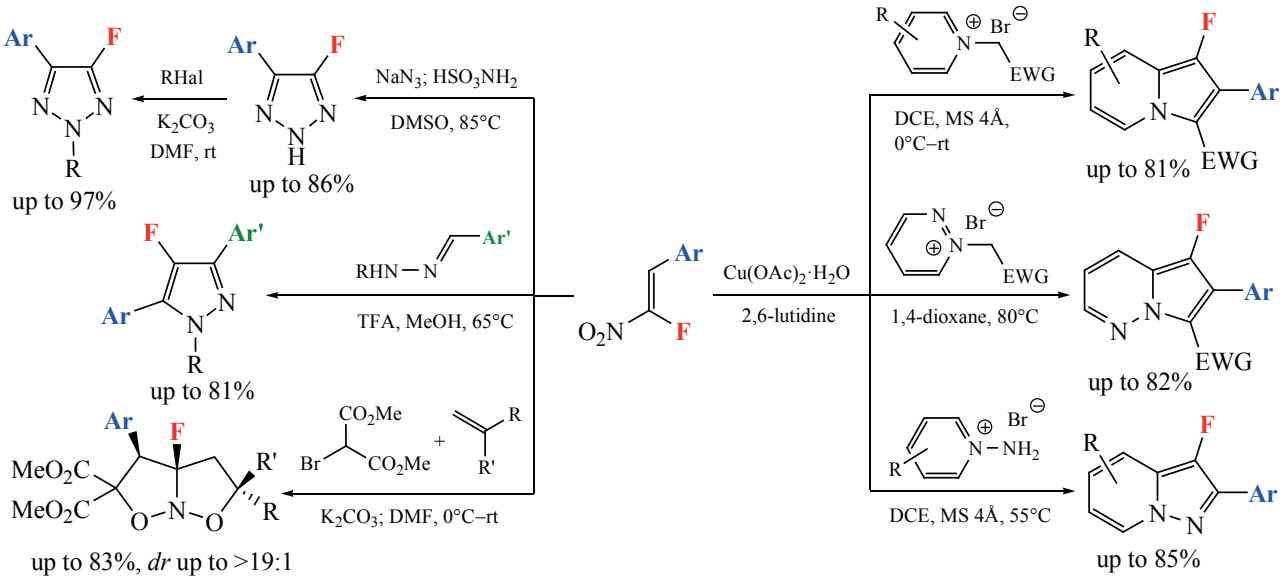
Another convenient method to obtain 4-fluoropyrroles from β-fluoro-β-nitrostyrenes in one synthetic step is the Barton–Zard reaction with 2-ethyl isocyanoacetate [354]. This transformation opened up access to fluorinated dipyrromethanes and their derived new fluorinated boron dipyrromethene dyes (Scheme 5.16) [355].

A few years ago, a new reaction for the catalytic olefination of carbonyl compounds was discovered [356,

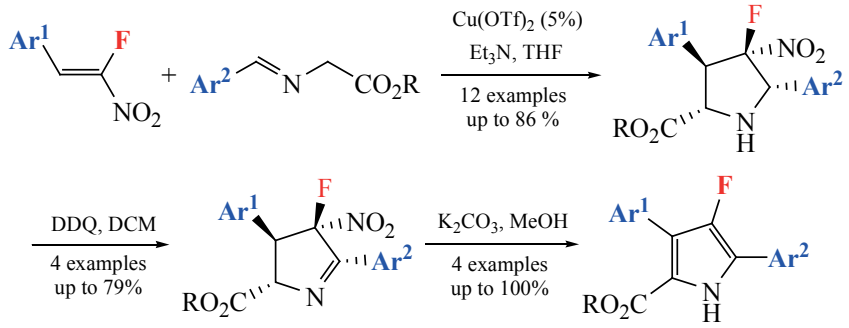
Scheme 5.13.



Scheme 5.14.



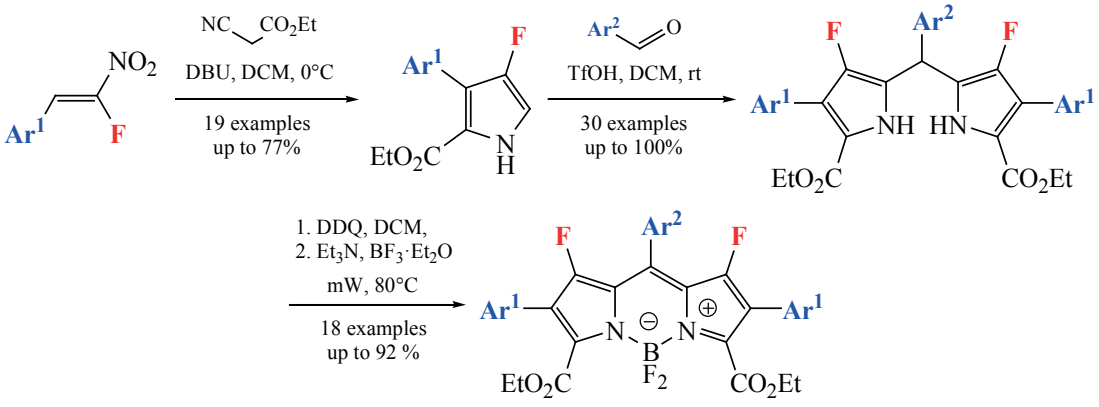
Scheme 5.15.



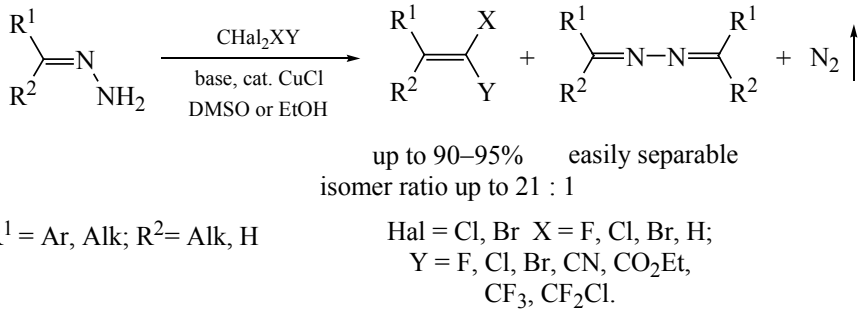
357]. The reaction has a high synthetic potential and allows stereoselective synthesis of alkenes containing different halogens and functional groups [358]. The olefination of freons was used to prepare fluorinated alkenes, convenient building blocks for the synthesis of more complex fluorinated compounds by reactions with nucleophiles (Scheme 5.17) [359–365].

Thus, α-fluoro- and α-trifluoromethylacrylonitriles [366], as well as α-fluoro-β-arylvinyl sulfones [367], vinyl sulfides [368], alkoxystyrenes [369], CF_3-imines

Scheme 5.16.



Scheme 5.17.

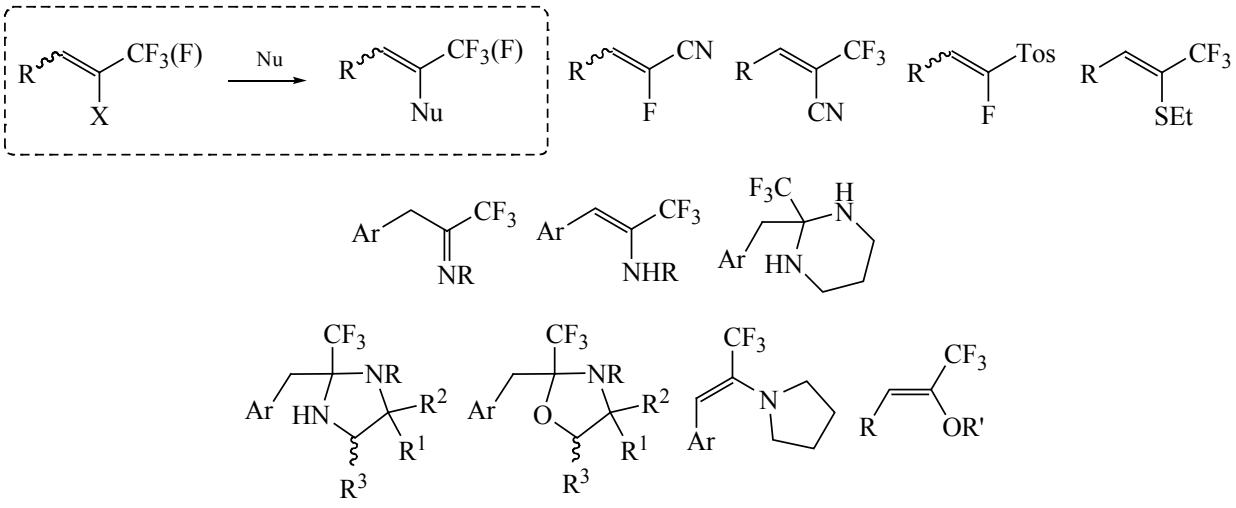


[370], CF_3-enamines [371–374], CF_3-imidazolidines, CF_3-oxazolidines, and CF_3-hexahydropyrimidines [375] were synthesized with high regio- and stereoselectivivities and high yields (Scheme 5.18).

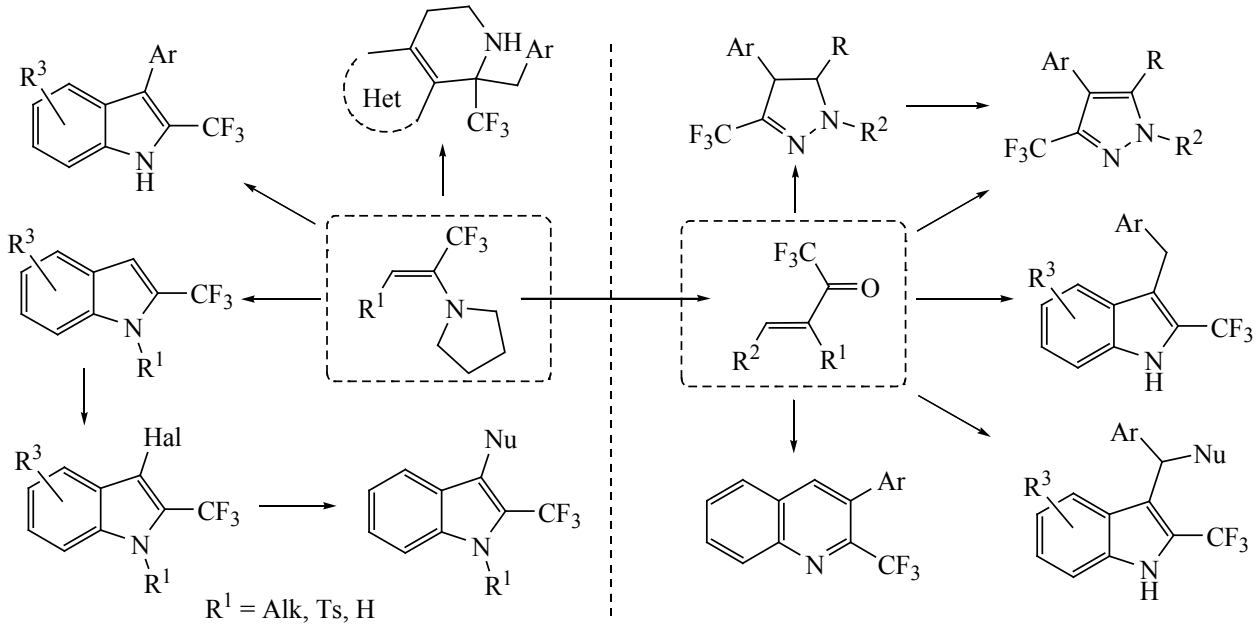
Of particular interest proved to be α-CF_3-enamines obtained by the reaction of fluorinated alkenes with amines [353–355]. These products are synthetic equivalents of benzyl trifluoromethyl ketones and were successfully used in the syntheses of indoles by the Leimgruber–Bach [376, 377], Fischer [378], and intramolecular cyclization of enamines generated in situ by the reaction of β-halo-β-trifluoromethyl(*ortho*-bromostyrenes) with primary aliphatic amines [379]. Trifluoromethyl enamines were also used in the synthesis of carbolines by the Pictet–Spengler reaction [559] and in the synthesis of α,β-disubstituted CF_3-enones by condensation with carbonyl compounds [380]. The reaction of CF_3-enones with hydrazines formed the basis for the development of synthetic approaches to fully substituted CF_3-pyrazolines and CF_3-pyrazoles hardly which are difficult to access by other methods [361]. The reductive cyclizations of CF_3-enones containing nitro groups in the aryl rings gave 2-CF_3-indoles [381, 382] and 2-CF_3-quinolines (Scheme 5.19) [383].

It was shown that dibromoalkenes can be successfully replaced by dichloroalkenes in the synthesis of terminal acetylenes by the Corey–Fuchs method [384]. Furthermore, the reaction of in situ generated lithium acetylides with ethyl trifluoroacetate was used to prepare in high yields CF_3-ynones [365], which were then involved in heterocyclization reactions with nucleophiles and binucleophiles, as well as cycloaddition (Scheme 5.20). Thus, reactions with hydrazines were laid as a basis to develop effective methods for the regioselective synthesis of 5-CF_3-pyrazolinols, 3- and 5-CF_3-pyrazoles [385, 386], and their derivatives [387], including a number of drugs, for example, Celebrex. Reactions with sodium azide were used to synthesize 3-CF_3-isoxazoles and 4-trifluoroacetyltriazoles [388], whose alkylation and arylation resulted in the

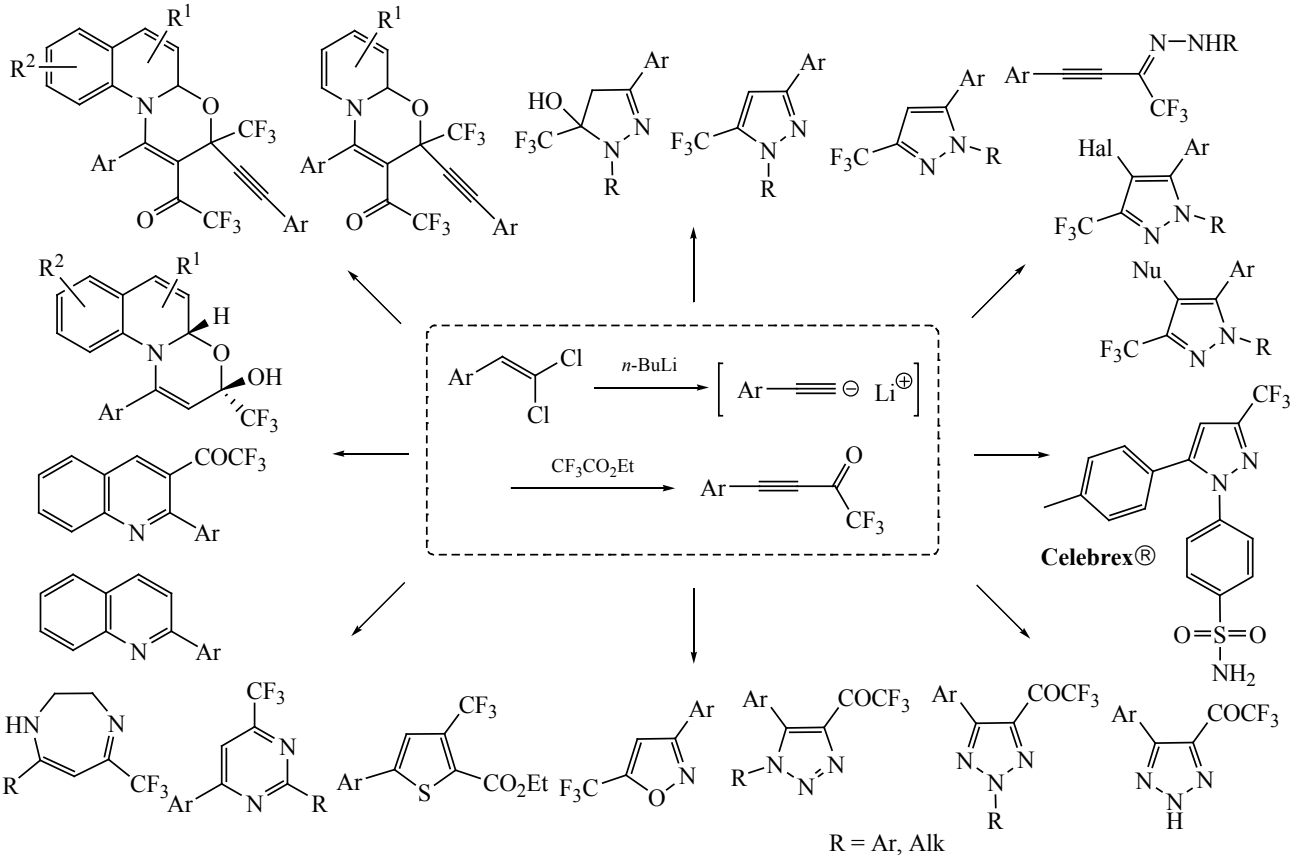
Scheme 5.18.



Scheme 5.19.



Scheme 5.20.



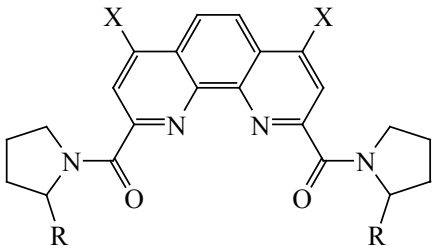
regioselective formation of the 2-*N* derivatives that showed interesting photochemical properties [389].

The [3+2] cycloaddition of substituted azides regioselectively produced the isomeric 1-*N*-substituted 4-trifluoroacetyltriazoles [390]. The reaction with methyl thioglycolate allowed efficient synthesis of methyl 3-(trifluoromethyl)thiophene-2-carboxylate derivatives [391]. The reactions of CF_3-ynones with amidines opened the way to the synthesis of 1,3-pyrimidines [392]. The reaction with ethylenediamine provided trifluoromethyl-containing [1,4]-diazepines in good yields [393]. A number of 1,3-oxazinopyridines [394] and 1,3-oxazinoquinolines [395–397] were synthesized by the reactions of CF_3-ynones with azines. The rearrangement of the latter products under the action of bases gave 2-arylquinolines and 2-aryl-3-trifluoroacetylquinolines [398].

As shown in [399], 1,10-phenanthroline-2,9-diamides with cyclic substituents exhibit high selectivity in the extractive separation of the Eu(III)/Am(III) pair. A series of pyrrolidine derivatives of phenanthroline diamides with different α-substituents were prepared synthesized (Scheme 5.21) [400]. Quantum-chemical calculations and extraction experiments revealed significant differences in the extraction properties of different diastereomeric forms [401].

Phenanthroline diamides were found to form 1 : 1 complexes with lanthanides [402]. Therewith, the coordination number varied from 9 to 10 depending on the ionic radius of the metal [403]. Sometimes, complexes of the ion-pair type formed, and this made it possible to explain the so-called "gadolinium kink" often observed in studies of the extraction properties of phenanthroline diamides [404]. To adjust the extraction and other properties of the ligands by varying the

Scheme 5.21.



structure of the substituents, a series of *N*,*N*'-dialkyl-*N*,*N*'-diaryl-1,10-phenanthroline-2,9-diamides was obtained [405]. It turned out that such compounds can form tight ion-pair complexes with uranyl nitrate [406] (Fig. 5.2) and also able to effect "superextraction" of uranium, specifically 1 mol of the ligand binds 2 mol of metal [407].

Macrocyclic derivatives of phenanthroline diamides were synthesized for the first time (Scheme 5.22) [408, 409], which demonstrated high separation factors (up to 40) for the Am(III)/Eu(III) pair.

A complex approach to the functionalization of phenanthroline diamides at the 4 and 7 positions of the heterocyclic core was developed. The nucleophilic aromatic substitution (S_NAr) of chlorine atoms in diamides allowed us to obtain the first examples of 4,7-difluoro-substituted diamides in yields of up to 88% (Scheme 5.23) [410]. The coordination chemistry of the fluorine-containing ligands was studied in detail in comparison with the starting chlorine-containing ligands, as well as the corresponding diamides without substituents in the 4 and 7 positions [411].

A preparative method for the synthesis of unsymmetrical fluorine-containing diamides starting from 4,7-dichloro-1,10-phenanthroline-2,9-diamides was developed (Scheme 5.24) [412]. It was found

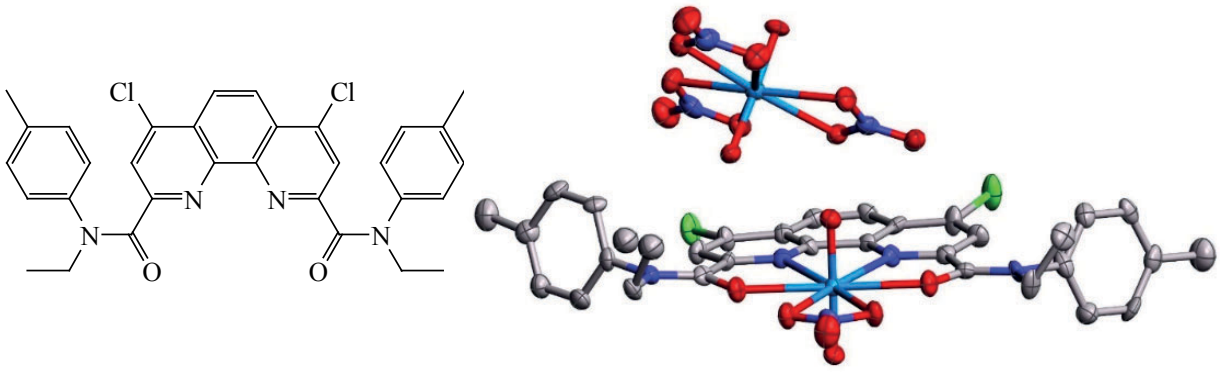
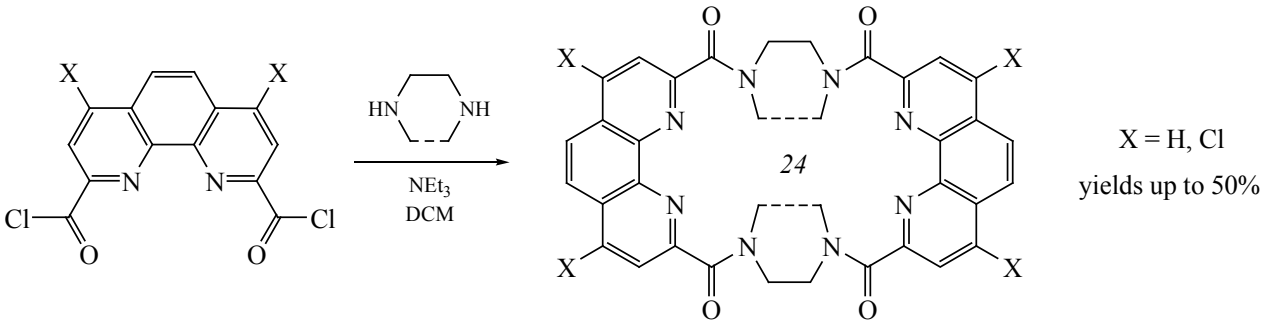
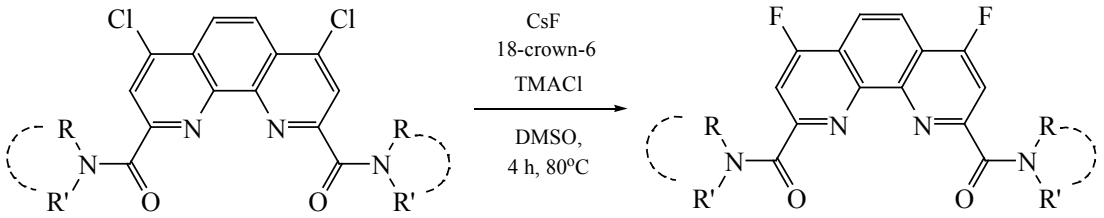


Fig. 5.2. Series of *N*,*N*'-diakyl-*N*,*N*'-diaryl-1,10-phenanthroline-2,9-diamides.

Scheme 5.22.



Scheme 5.23.



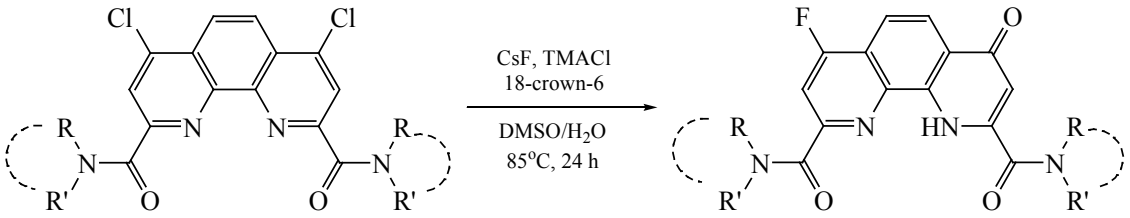
72–88%

that fluorine-containing ligands are easily hydrolyzed at room temperature to the corresponding hydroxy oxo derivatives, while the hydrolysis of 4,7-dichloro-substituted diamides occurs under much more severe conditions.

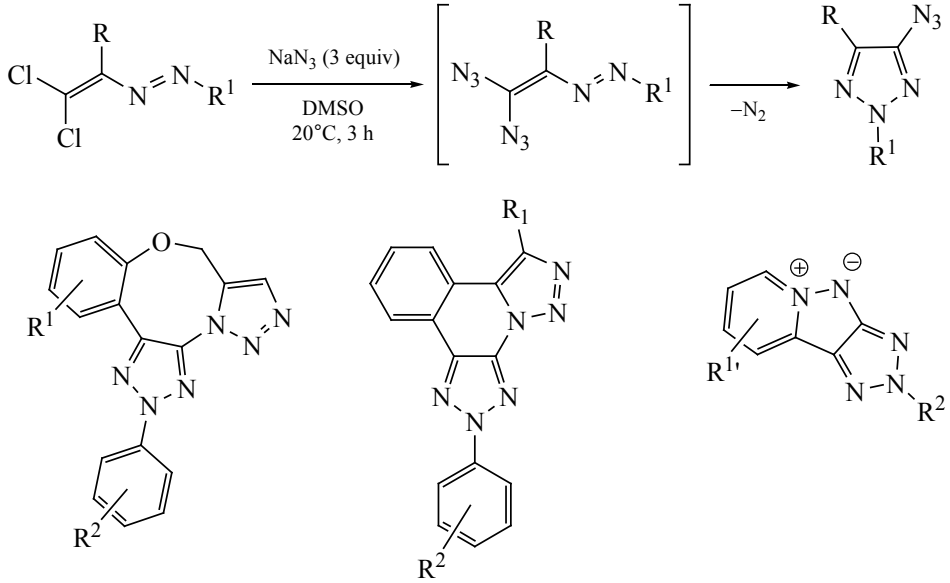
It was expected that the reaction of dichloro-1,2-diaza-1,3-dienes [413] with excess sodium azide would yield new bis-azides [414] via the addition of azide anions to the C=C bond. However, it turned out that the intermediate bis-azide quickly transformed into the corresponding azidotriazole (Scheme 5.25) [415]. This reaction is of a very general nature, as evidenced by numerous examples [416]. 2-Substituted triazoles are attractive from the point of view of their photophysical properties, but the resulting azidotriazoles demonstrated low luminescence quantum yields. After testing different variants of ring closure involving the azide group, we developed effective synthetic approaches to previously unknown heterocyclic systems [417–420], the luminescence quantum yields of some of which were close to quantitative. It was shown that activated dichloroalkenes can serve as useful starting materials for the synthesis of other unstable bis-azides [421, 422].

The Laboratory of Biologically Active Organic Compounds (**head Professor E.K. Beloglazkina**) has developed a general methodology for the synthesis of spiro-fused heterocycles by cycloaddition reactions of 1,3-dipoles of different types (azomethine ylides [423–425], nitrilimines [426–428], nitrile oxides [428]) and conjugated dienes [429]) at exocyclic C=C, C=S, and C=N bonds of 2-chalcogen-5-methylene-substituted imidazolones. The proposed synthetic methodology offers the following advantages: (1) universality: it allows one to obtain different types of polyspiroheterocycles with high stereoselectivity by varying the reagents used in the synthesis; (2) atom economy: in cycloaddition reactions, all atoms of the starting compounds are included in the product; and (3) high potential for the

Scheme 5.24.



Scheme 5.25.



use in the synthesis of biologically active compounds: the rigidity of the spiro-fused framework allows fixing the required spatial arrangement of substituents that are important in terms of interaction with biological targets. Some of the synthesized compounds showed high cytotoxicity with significant selectivity with respect to cancer cells, and one of them successfully passed preclinical trials as a drug for colorectal cancer therapy [423, 424].

A general synthetic approach to 5-methylidene-substituted 2-chalcogenimidazolones, starting from α-amino acids containing an additional group capable of elimination (morpholine or hydroxyl) in the β position and isocyanates, isothiocyanates, or isoselenocyanates [430, 431]. It should be noted that 5-methylidene selenohydantoins containing an exocyclic CH_2=C bond had never been described in the literature before our work. Then, the obtained dipolarophiles were used as a basis to develop a regio- and diastereoselective synthesis of mono-, di-, and trispiro derivatives of 2-chalcogen-5-methyleneimidazolones by the 1,3-dipolar cycloaddition of azomethine ylides, nitrilimines, azomethine imines, and nitrile oxides to these dipolarophiles [423–434]. The structural types of the synthesized compounds are shown in Fig. 5.3. By introducing additional chiral substituents into the initial dipolarophiles [435, 436], it proved possible to separate the mixture of diastereomers, formed in the 1,3-dipolar cycloaddition reaction, and then remove the auxiliary asymmetric group to obtain enantiomerically pure products.

In studying 1,3-dipolar cycloaddition reactions with 2-(chalcogen)imidazolone derivatives, we found that the spontaneous diffusion of volatile reactant vapors into a solution containing a stable precursor of an unstable reactive intermediate can provide the simplest method to carry out certain organic reactions, including 1,3-dipolar cycloaddition. The diffusion of tertiary amine vapors into a reaction mixture containing a dipolarophile and hydrazonyl chloride or hydroxyimidoyl halide allows one to implement 1,3-dipolar cycloaddition reactions even with unstable and easily dimerizing nitrile oxides and nitrilimines with quantitative yields, and these reactions are extremely easy in experimentation (Fig. 5.4) [428].

Along with 1,3-dipolar cycloaddition reactions, we also demonstrated the use of 5-methylidene-2-chalcogen-imidazol-2-ones as dienophiles in Diels–Alder reactions with cyclic and acyclic dienes, specifically cyclopentadiene, cyclohexadiene, 2,3-dimethylbutadiene, and isoprene (Scheme 5.26) [437, 438].

Three methodologies for the synthesis of bisaryl-spiro[azetidine-2,3'-indoline]-2',4-diones by the Staudinger ketene–imine cycloaddition reaction: the reaction of substituted 3-aryliminoindolin-2-ones with phenylacetic acid chlorides, which results in the predominant formation of the *trans* diastereomers of spiro-β-lactams, as well as procedures for the one-pot synthesis from isatinimines and phenylacetic acids in the presence of oxalyl chloride or *p*-toluenesulfonyl chloride as activating agents with the predominant

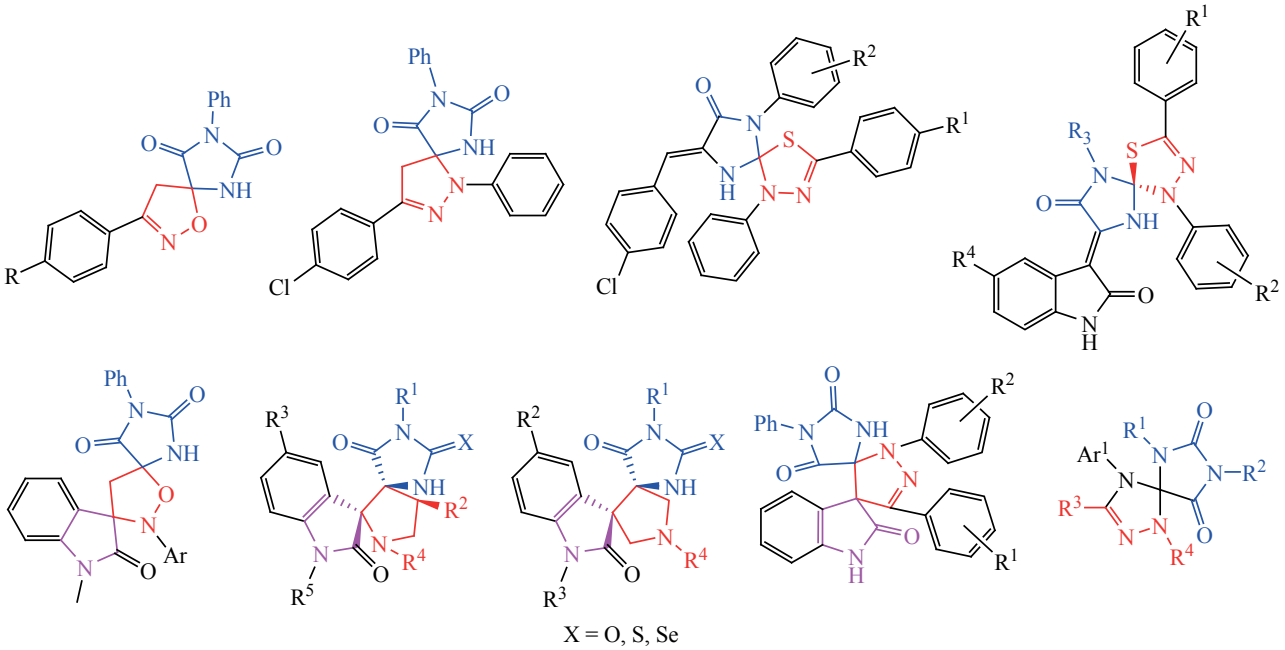


Fig. 5.3. Structural types of spiro derivatives of 2-chalcogenimidazolones obtained by 1,3-dipolar cycloaddition reactions.

formation of the *cis*-diastereomers of the products (Scheme 5.27) [439, 440].

Previously unknown spirooxindolo-γ-lactams with two spiro junctions in the azetidin-2-one ring were synthesized for the first time by the Staudinger ketene–imine cycloaddition reaction of activated 2-oxopyrrolidine-3-carboxylic acid derivatives with isatinimines. The reaction resulted in the diastereoselective formation of *trans*-diastereomeric products (Scheme 5.28) [441].

The laboratory is also developing methods for the synthesis of targeted therapeutic and diagnostic drug conjugates based on ligands for prostate-specific membrane antigen (PSMA) — promising targeted anticancer drugs [442–452]. New highly selective ligands for PSTM were synthesized and found to be superior in their affinity for PSMA than their known analogs. The synthesized ligands were used to obtain low-molecular conjugates with various anticancer drugs [docetaxel (Fig. 5.5), monomethyl auristatin E]

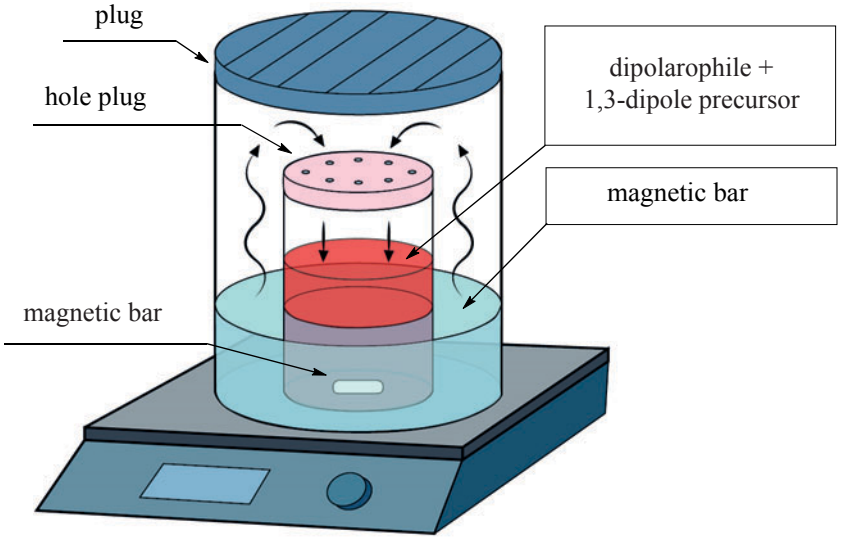
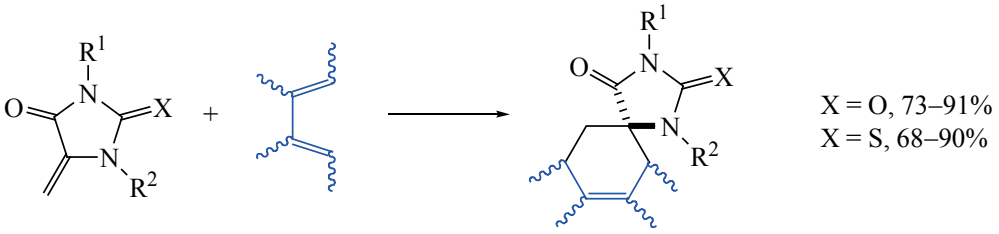


Fig. 5.4. Scheme of the device for carrying out diffusion mixing reactions.

Scheme 5.26.



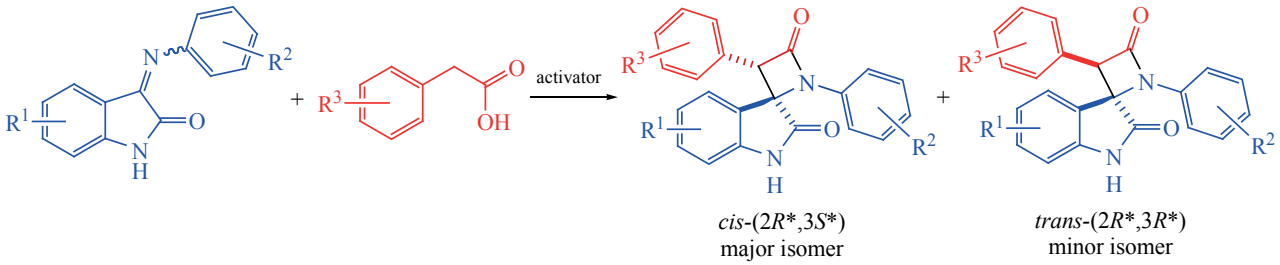
R^1 = Ar; R^2 = H, Ac, COOEt, Ts, Boc, CH_2Ph, C(O)NHPh

for treatment of prostate cancer, and also fluorescent conjugates with FAM-5, SulfoCy5, and SulfoCy7 dyes for intraoperative navigation.

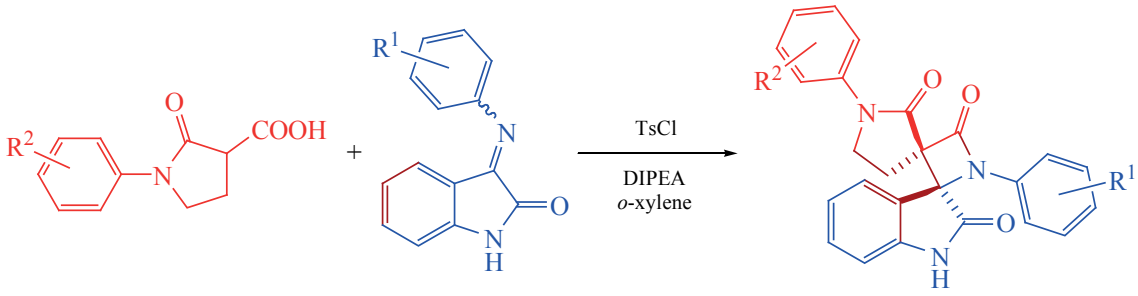
Potential dosage forms for these conjugates were developed for the subsequent preclinical and clinical trials. A full range of preclinical trials have already been conducted for these conjugates and further clinical trials are planned. Methods for the synthesis of dual conjugates with abiraterone/docetaxel, abiraterone/monomethyl auristatin E, enzalutamide/monomethyl auristatin E, and ispinesib/monomethyl auristatin E drug combinations were also developed.

New platinum-containing drugs for the therapy, diagnostics, and theranostics of cancer are being developed and studied [453–458]. Platinum(IV) prodrugs with axial ligands of different natures, synthesized from oxoplatinum via carbamate bond formation and copper-catalyzed azide–alkyne cycloaddition reaction (Scheme 5.29), were developed and found to be effective anticancer drugs capable of overcoming drug resistance and effectively accumulating in malignant neoplasms. Varying the nature of the axial ligand and its biological action and lipophilicity in Pt(IV) prodrugs allows one to adjust the pharmacological activity, selectivity, and penetrating ability of the drug. It was shown that the cytotoxic activity of Pt(IV) prodrugs depends on their lipophilicity and reduction rate and on the type of the linker between the axial ligand and Pt(IV), but is largely determined by the nature of the axial ligand. Photoactivated Pt(IV) prodrugs with riboflavin and boron dipyrromethene ligands, capable of controlled release of cisplatin upon light excitation were developed. Novel dual action photoactivated Pt(IV) prodrugs were obtained, which can act as agents for both photocontrolled chemotherapy and photodynamic therapy.

Scheme 5.27.



Scheme 5.28.



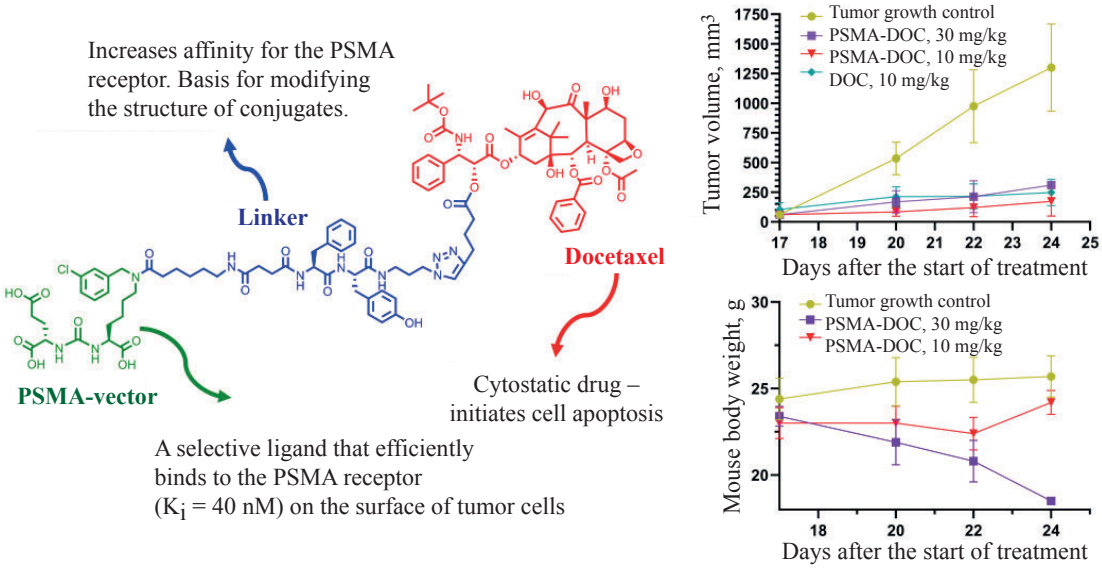
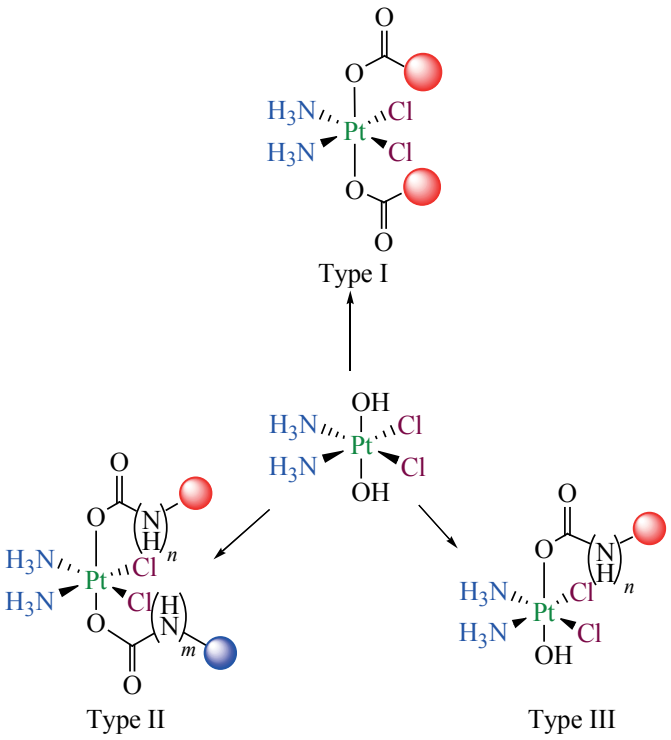


Fig. 5.5. Targeted docetaxel conjugate with ligand for prostate-specific membrane antigen [446].

The developed compounds demonstrated the ability to overcome cisplatin resistance and accumulate in cancer cells, high cytotoxic activity (150 times higher than that of cisplatin), and much higher maximum tolerated doses in vivo than drugs used in clinical practice.

The Laboratory of Supramolecular Chemistry and Nanotechnology of Organic Materials (**head Corresponding Member of the Russian Academy of Sciences, Professor S.P. Gromov**) developed a new scientific direction: the design, creation, and investigation of photoactive supramolecular devices and

Scheme 5.29.



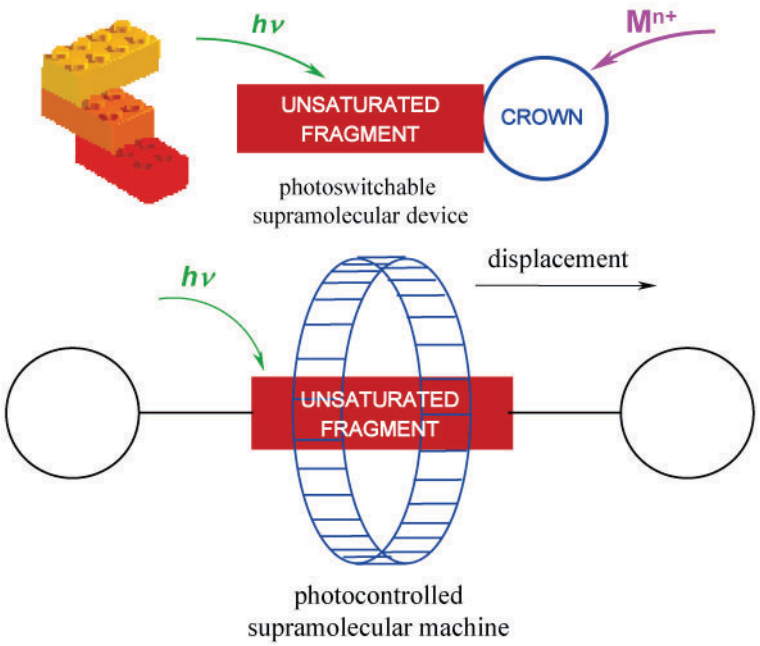


Fig. 5.6. Supramolecular mechanisms and machines for the generation, conversion, and transmission of energy and motion at nano levels.

machines [459]. This work was awarded the State Prize of the Russian Federation in the field of science and technology. The area of application of the developed photoactive supramolecular devices and machines is the creation of mechanisms and machines for the generation, conversion, and transmission of energy and motion at nano levels, as well as the creation of a nano instrument for monitoring and diagnosis of nano quantities of materials and substances (Fig. 5.6).

A crystallographic approach to the topochemical [2+2] photocycloaddition ([2+2] PCA) reactions of unsaturated compounds in a single crystal with the preservation of the crystal lattice has been developed [460]; the series of works was supported by the Zelinskii Prize of the Russian Academy of Sciences. The scope of application of the crystallographic approach to [2+2] PCA reactions is the construction of single crystal devices for reversible optical recording of information in the bulk of a sample (Fig. 5.7).

The group of **RAS Professor S.Z. Vatsadze** (together with colleagues from the USA) established that regardless of the polarity of the radical, reactions with isonitriles proceed at a high rate and explained this phenomenon in terms of the dualism ("stereoelectronic chameleons") of the electronic properties of the isonitrile group [461].

It was shown for the first time that new conjugates of bispidines, including those with monoterpenoids (Fig. 5.8), are able to catalyze such reactions as the Henri reaction [462, 463], the addition of diethylzinc to chalcones [464], the addition of terminal acetylenes to aldehydes [465], and the Chan–Lam–Evans reaction [466]. This part of the work was carried out jointly with colleagues from the Institute of Organic Chemistry, Siberian Branch, Russian Academy of Sciences (SB RAS).

The research focus of the group of molecular electrochemistry (**leader Professor T.V. Magdesieva**) is the design and synthesis of stable organic radicals. The current area is the synthesis of ambipolar molecules capable of forming three stable redox states (cation–radical–anion), which are in demand in organic electronics, as redox-active materials for organic batteries, etc. A series of works was performed on the design and synthesis of stable self-adjusting diarylnitroxyl radicals [467] that exhibit ambipolarity due to the dynamic stabilization of oppositely charged redox states: by introducing chameleon substituents capable of changing their donor-acceptor properties by rotation relative to the phenyl ring [468] or entering into additional intramolecular through-space interactions, which are switched on/off as needed [469]. A new synthetic approach to symmetrical diarylamines

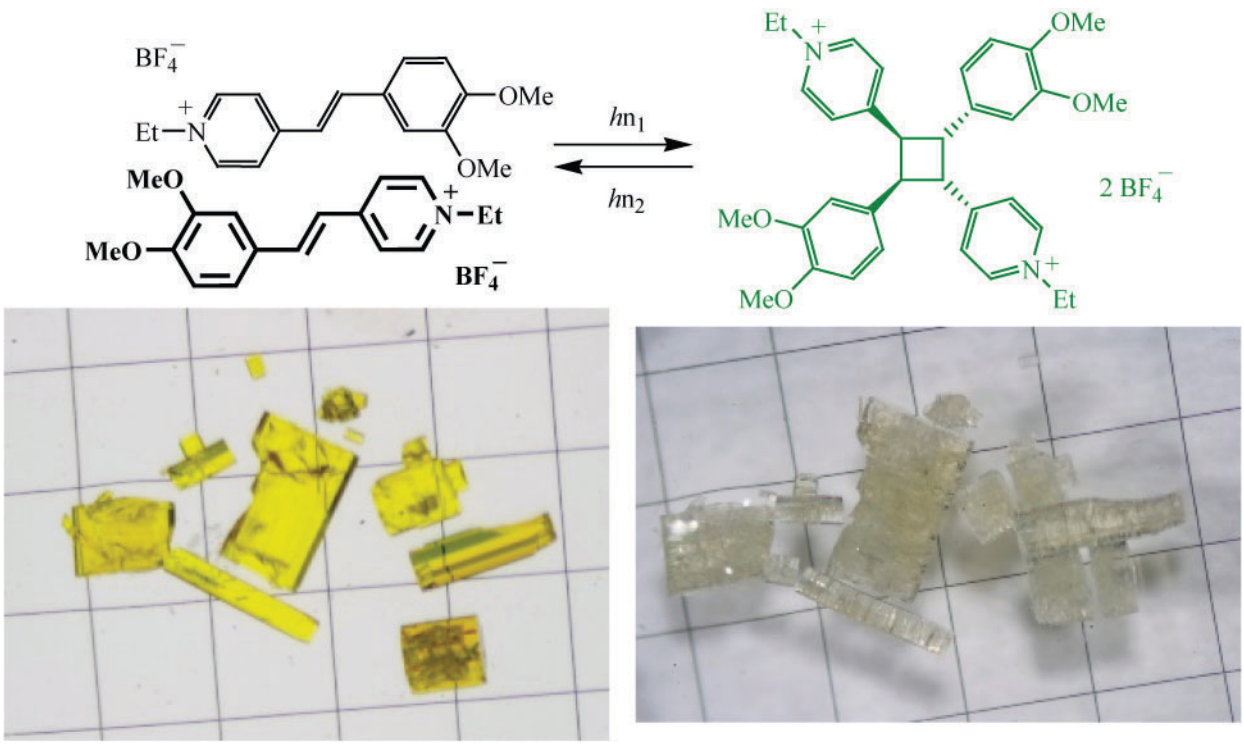


Fig. 5.7. Construction **of** single crystal devices for reversible optical recording of information in the bulk of a sample.

(precursors of target diarylnitroxides) by the Cu(I)-catalyzed amination of arylboronic acids with alkyl nitrites was proposed [470].

A new type of extremely rare neutral stable mixed-valence radicals [471] was synthesized, which exhibit pronounced ambipolarity and intense absorption in the entire visible and near-IR range, which is of interest for the creation of heat-insulating coatings. The population inversion (SOMO–HOMO inversion) inherent in the new radicals makes them interesting objects for spintronics.

The stability and properties of a radical depend largely on the gap between its oxidation and reduction potentials. A theoretical model [472] was proposed that clarifies the physical nature of the electrochemical gap ($\Delta E = E^{Ox} - E^{Red}$) in radicals and its relationship with spin density distribution. It was shown that the main factor that ensures the very existence of radicals in solution is the Coulomb repulsion that arises during reduction, when the second electron occupies the SOMO (Fig. 5.9).

Currently, the main research interest of the ***Laboratory of Physical Organic Chemistry*** (**head RAS Professor**

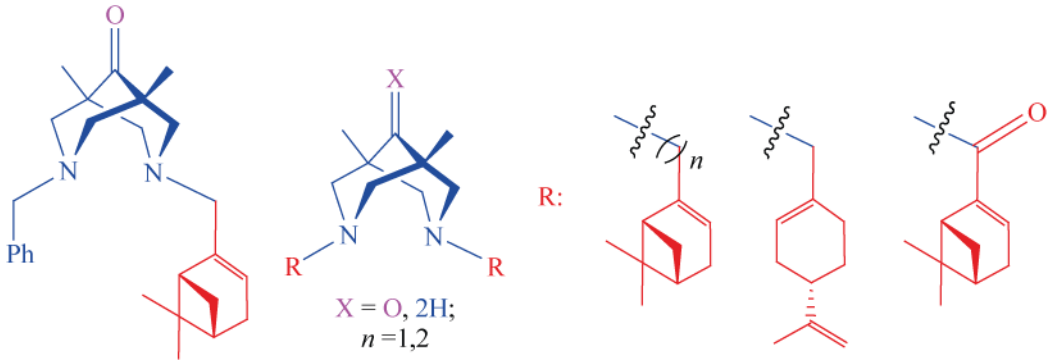


Fig. 5.8. New bispidine conjugates.

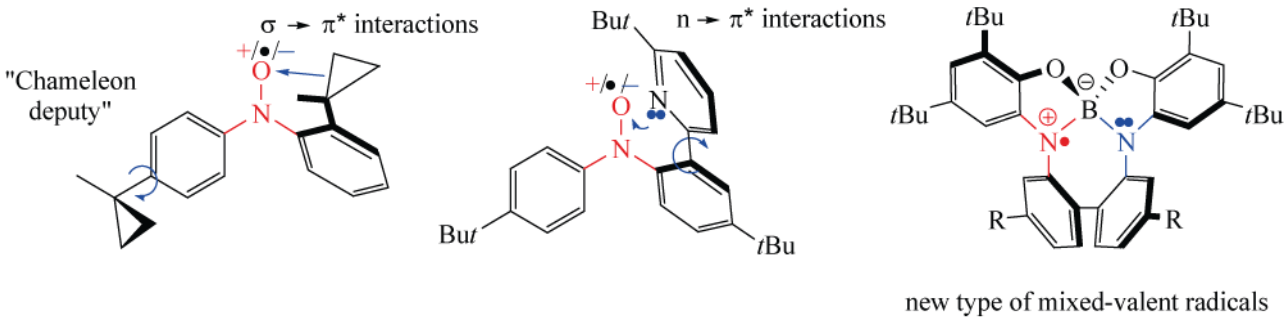


Fig. 5.9. Interaction between radicals.

S.S. Karlov) is focused on the synthesis of organic compounds of nontransition metals and metalloids and study of their structure, chemical properties, and potential applied applications.

One of the main lines of research of the group headed by Prof. Karlov is the synthesis of new initiators of polymerization of cyclic esters and study of the behavior of these initiators in polymerizations leading to the production of biodegradable polymers [473]. Over the past years, a lot of new metal (zinc, gallium, aluminum, and titanium) complexes have been synthesized, which have proven to be effective initiators of homopolymerization of L-lactide and ε-caprolactone. It should be noted that the synthesized derivatives are metal complexes that are traditionally considered nontoxic, which is important for the use of such polymers in medicine and pharmaceutics, and their activity in polymerization is often significantly higher compared to tin bis(2-ethylhexanoate) currently used in industry [473, 474]. An extremely important result was obtained in the study of the copolymerization of L-lactide and ε-caprolactone. It should be noted that the resulting copolymer poly(L-lactide-co-caprolactone) is one of the most important biodegradable copolymers. The two related homopolymers have opposite physical and mechanical properties: polycaprolactone has good elasticity and permeability but poor mechanical characteristics (viscosity), by contrast to polylactide. The preparation of a random copolymer poly(LA-stat-CL) could lead to biodegradable materials with improved properties, but in most cases the copolymerization of LA and CL gave block poly(LA-block-CL) or gradient poly(LA-grad-CL) copolymers because of the different rates of polymerization of these monomers with most of the studied initiators. Interestingly, the homopolymerization of CL is usually faster than that of LA, while the copolymerization of both monomers often results in the primary consumption of LA from the monomer mixture. The group of Prof. Karlov synthesized and tested two effective aluminum initiators: copolymerization in the presence of benzyl alcohol and initiated by an aluminum pyridinebisphenol complex led to poly(LA-stat-CL), and with an aluminum diethylenetriamine complex, poly[LA-stat-(4-benzyloxycarbonyl-4-methyltrimethylene carbonate)] formed (Fig. 5.10) [475, 476]. In addition, the mechanism of copolymerization of L-lactide and ε-caprolactone was studied in detail using DFT quantum-chemical calculations [477].

Two effective aluminum initiators: copolymerization in the presence of benzyl alcohol and initiated by the aluminum pyridinebisphenol complex gave poly(LA-stat-CL), and with the aluminum diethylenetriamine complex, poly[LA-stat-(4-benzyloxycarbonyl-4-methyltrimethylene carbonate)] formed. Along with the

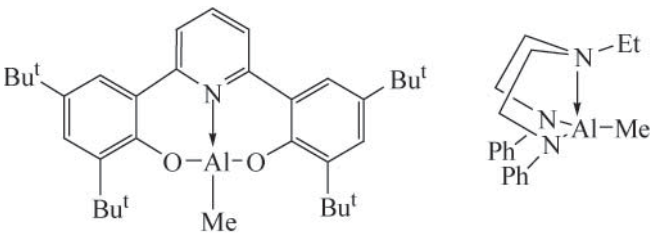


Fig. 5.10. Two effective aluminum initiators: copolymerization in the presence of benzyl alcohol and initiated by the aluminum pyridinebisphenol complex gave poly(LA-stat-CL), and with the aluminum diethylenetriamine complex, poly[LA-stat-(4-benzyloxycarbonyl-4-methyltrimethylene carbonate)] formed.

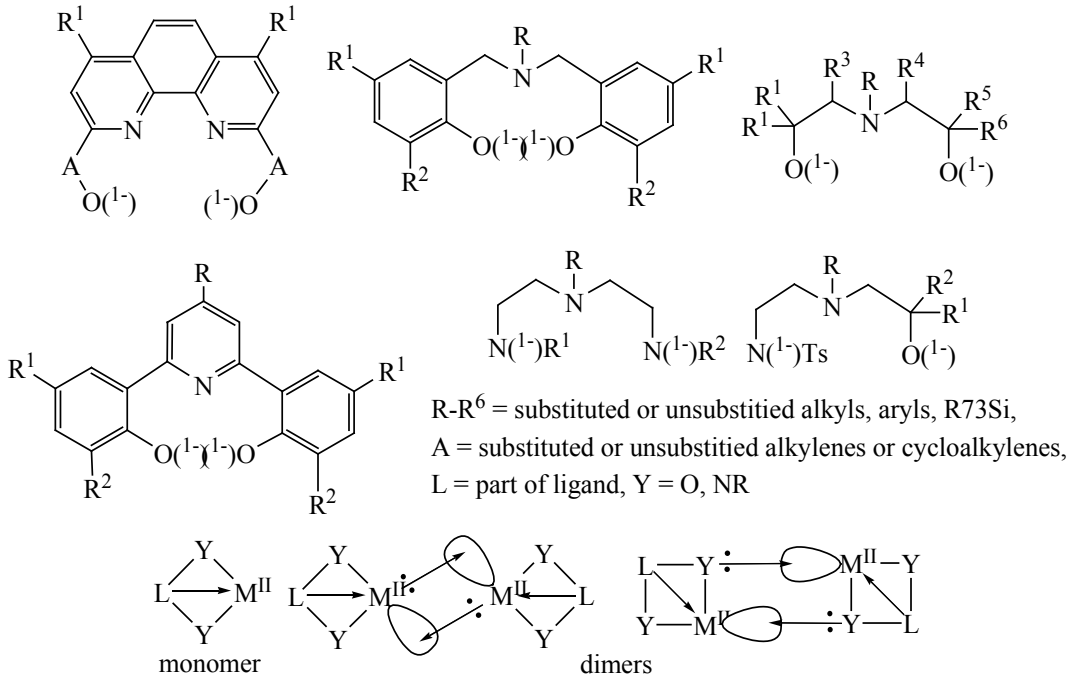


Fig. 5.11. Structural factors that determine whether tetrylenes exist as monomers or as one of the two diners.

above-described line of research, the group is dealing with the synthesis and study of the properties of tetrylenes: derivatives of Group 14 elements in the oxidation state (2+), which are heavy analogs of carbenes. The group carried out systematic studies on the synthesis of tetrylenes with different ligand environments (ligand types are shown in Fig. 5.11) [478]. The structural factors that determine whether tetrylenes exist as monomers or as one of the two diners (Fig. 5.11) were established. The behavior of the synthesized tetrylenes in insertion and cycloaddition reactions was studied. It was found that the monomeric structure of tetrylene promotes reaction that leads to the expected insertion and addition products, where the oxidation state of the Group 14 element atom becomes 4+. At the same time, dimeric tetrylenes often give mixtures of compounds that are difficult to identify.

Quite recently, an interesting feature of the insertion reaction of Lappert's germylene $Ge[N(SiMe_3)_2]_2$ into the S–S bond of diphenyl disulfide in the presence of various external bases (amines, phosphines) has been discovered: the lower the oxidation potential of the external base, the more actively it accelerates the oxidative addition reaction. However, this dependence has an extremum: ligands oxidized at potentials lower than that of the germylene itself have a weaker catalytic effect [479].

The group of Leading Researcher A.A. Prishchenko developed an organosilicon methodology for the synthesis of functionalized organophosphorus compounds and used it to prepare new derivatives of phosphonous and phosphinic acids containing organoelement (organosilicon, germanium, phosphorus and ferrocene) and heterocyclic (pyridine, furan, and pyrrolidone) fragments. The developed methods for the synthesis of water-soluble acids and their salts are based on the reaction of highly reactive bis(trimethylsiloxy)-phosphine with functionalized unsaturated synthons to form the corresponding alkyl phosphonites, whose subsequent aminomethylation and carboxyethylation leads to trimethylsilyl phosphinates of different structures. The resulting esters are subjected to methanolysis, which occurs under mild conditions and leads the target phosphinic acids, while treatment of functionalized alkyl phosphonites with sodium methylate in methanol results in the formation of the corresponding sodium salts in high yields (Fig. 5.12).

The synthesized compounds are of interest as promising biologically active agents and effective multipurpose polydentate ligands [480–484].

Section 6 is prepared in the framework of the implementation of the state assignment for the Department of Organic Chemistry of Lomonosov Moscow State University.

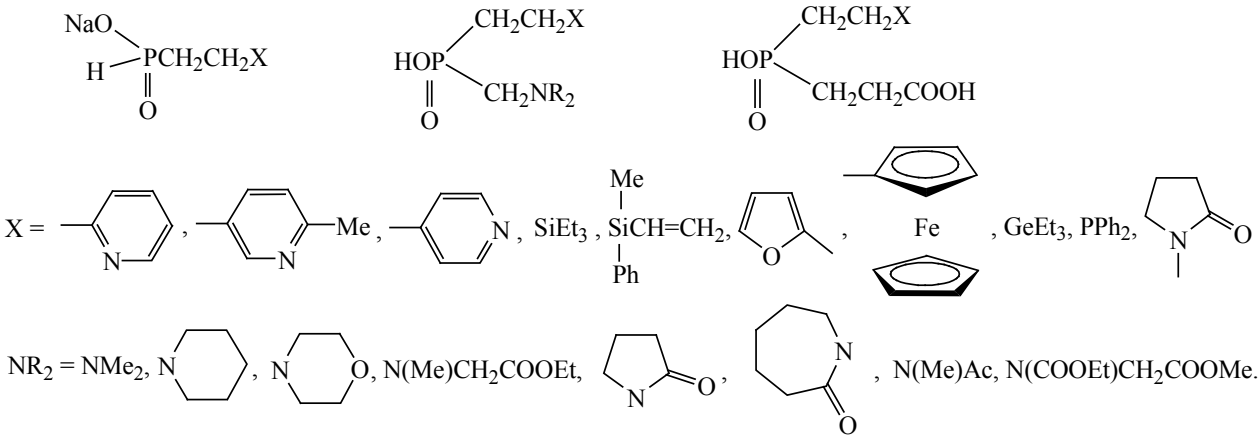


Fig. 5.12. High-yield formation of sodium salts.

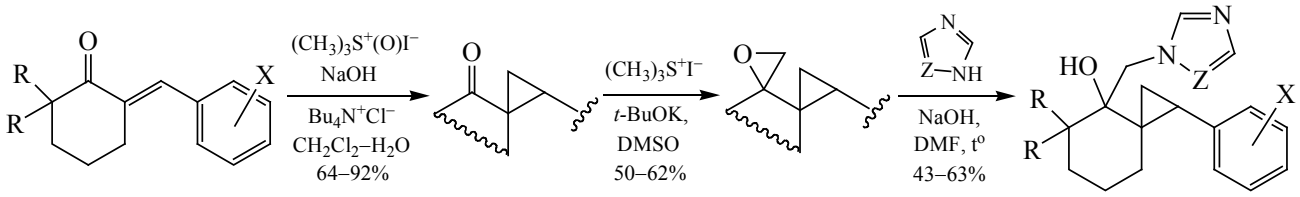
6. DEPARTMENT OF CHEMISTRY AND TECHNOLOGY OF ORGANIC SYNTHESIS, MENDELEEV UNIVERSITY OF CHEMICAL TECHNOLOGY OF RUSSIA

The Department of Chemistry and Technology of Organic Synthesis (DCTOS) of the Mendeleev University of Chemical Technology of Russia (MUCTR) was established in 1935. The area of scientific interests of the department was gradually shifting from research on the synthesis of special products (A.E. Kretov, 1935–1937; P.G. Sergeev, 1937–1938) to the development of technologies for heavy organic synthesis products, including synthesis high-molecular compounds (V.V. Korshak, 1939–1953). The established domestic school of synthesis and production of isocyanates (Yu.A. Strepikheev, 1953–1979) solved the problems of phosgene assimilation in the production of raw materials for polyurethane production and chemical crop protection compounds (CCPS), and was further developed by the founder of the scientific school of carbamic acid derivatives A.L. Chimishkyan (1979–2008). The scientific school of chemistry and technology of organic synthesis and biologically active substances (BAS) has formed at the department. In 1980–2000, production technologies for herbicides, rodenticides, plant growth regulators, and anticoagulants were introduced into industry. At present the department continues both exploratory studies into new CCPS and active pharmaceutical ingredients (API), as well as BAS technologies. The department employs 13 teachers, including 1 Corresponding Member of RAS, 1 Doctor of Sciences (Dr. Sci.), and 7 Candidates of Sciences (Cand. Sci.), 160 students (specialists, bachelors, and masters) and 7 postgraduates are studying, and 5 research groups are functioning.

One of the main areas of focus of the research group of the **Head of DCTOS, Associate Professor, Cand. Chem. Sci. S.V. Popkov** is the synthesis and study of the biological activity of 1-substituted azole derivatives. Under special scrutiny are the synthesis of substituted triazoles and imidazoles and systematic study of their chemical and biological properties and structure–biological activity relationships.

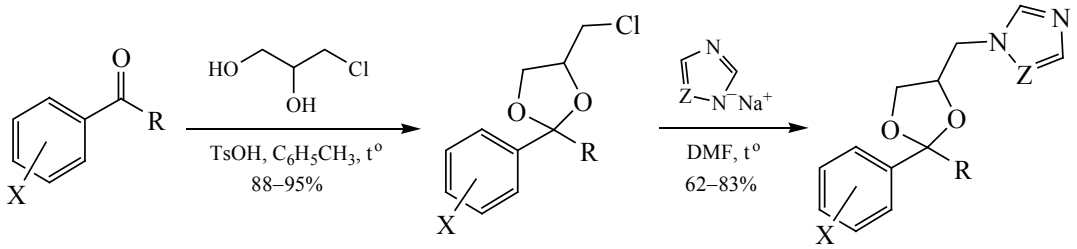
2,2-Disubstituted-6-arylidenecyclohexanones were used to develop effective methods of synthesis of 1-azolylmethylspiro[2.4]octanols and dispiro[2.1.4.3]-dodecanols. The Corey–Chaykovsky methylenation of the starting ketones at the C=C or C=O bond, depending

Scheme 6.1.



R = CH_3, R+R= $(CH_2)_4$, X = Hlg, Alk, Z = N, CH.

Scheme 6.2.



R = Alk, Ar, ArCH=CH; X = Hlg, Alk, CF_3, AlkO; Z= N, CH.

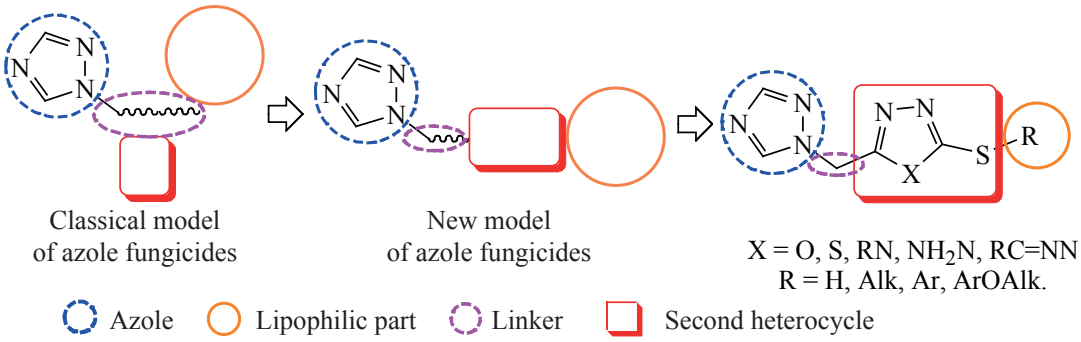
on the reagent used, gave corresponding oxiranes, which, after their ring opening with azoles, transform into the target (azolylmethyl)cyclohexanols (Scheme 6.1) [485]. Compounds of these series exhibit high fungicidal and antimycotic activity and are superior in fungitoxicity to the triadimenol standard. With bis(2,6-arylidene)cyclohexanones as starting materials, a series of 1-azolylmethyldispiro[2.1.2.3]decanols were obtained in a similar way [486].

The condensation of substituted alkanophenones and benzophenones with glycerol chlorohydrin gave 2-aryl-4-chloromethyl-1,3-dioxolanes, which are then used to alkylate sodium triazolate or imidazolate to form 4-azolylmethyl-2-aryl-1,3-dioxolanes (Scheme 6.2) [487]. Azolylmethyldioxolanes exhibit significant fungicidal [3] and antimycobacterial [488] activities, as well as retardant properties [489]. An alternative method for preparing azolylmethyldioxolanes, while in lower yields, is the condensation of 3-azolyl-1,2-propanediols with aceto- and benzophenones [490].

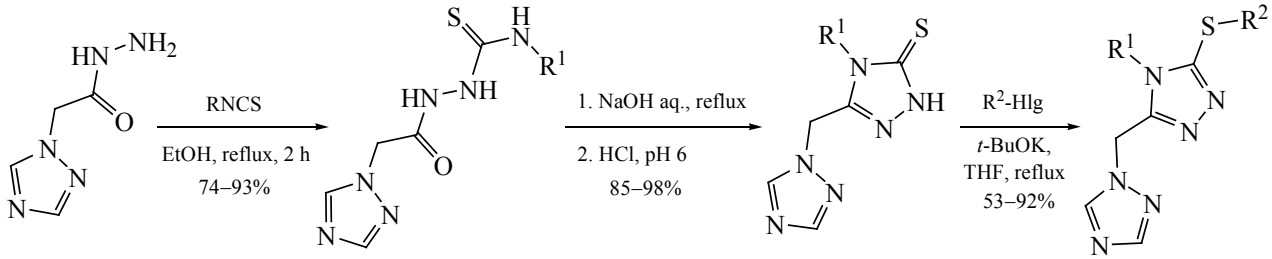
The main direction of the work of **Assistant G.V. Tsaplin** is the synthesis and biological activity of azol-1-ylmethylazoles, and a new approach to the design of azole derivatives with a high fungicidal activity was proposed (Scheme 6.3) [491].

Methods for the synthesis of 5-(azol-1-ylmethyl)-1,2,4-triazole-3-thiones from the key 2-(1,2,4-triazol-1-ylmethyl)acethydrazide by its acylation with isothiocyanates followed by cyclization in an alkaline medium (Scheme 6.4) [492]. The resulting 1,2,4-triazole-

Scheme 6.3.

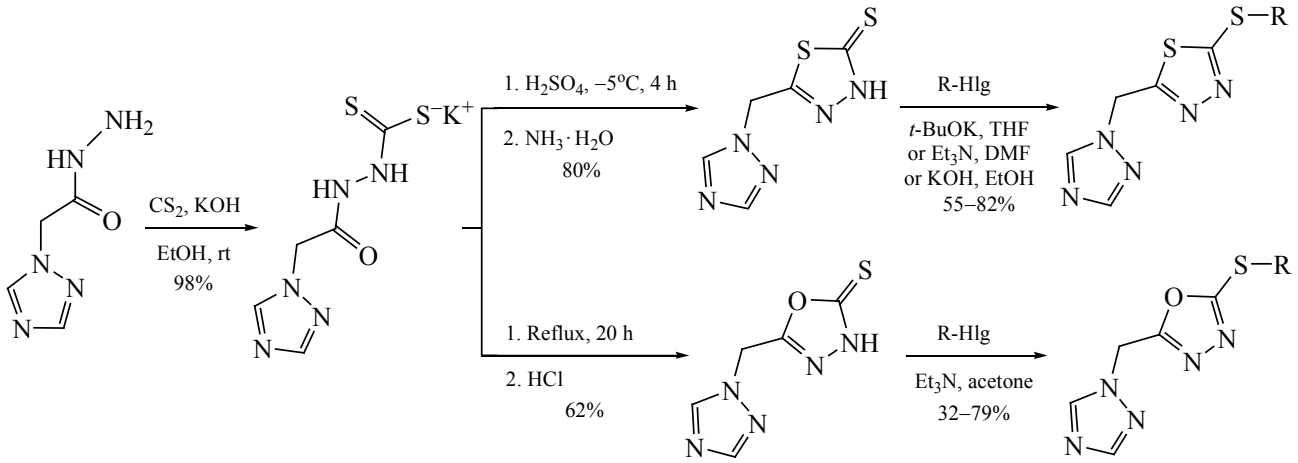


Scheme 6.4.



R^1 = Alk, Ar, HetAlk; R^2 = Alk, ArAlk, ArOAlk.

Scheme 6.5.



R = Alk, ArAlk, ArOAlk.

3-thiones were subjected to further modification, and the final products were found to have a low toxicity [493] and exhibit antioxidant and antihypoxic properties [494].

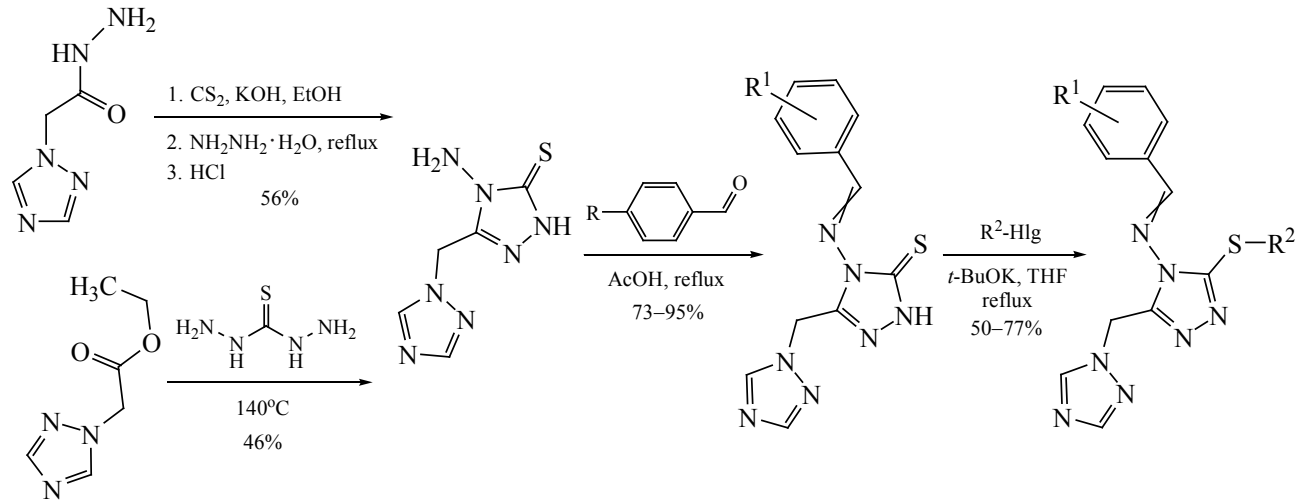
Analogous 5-(azol-1-ylmethyl)-1,3,4-thiadiazole-2-thiones and -1,3,4-oxadiazole-2-thiones (Scheme 6.5) are synthesized by the reaction of 2-(1,2,4-triazol-1-ylmethyl)acethydrazide with carbon disulfide in the presence of a base to form a carbodithionate salt and the subsequent acid- or base-catalyzed cyclization of the latter (Scheme 6.5) [491, 495, 496].

4-Amino-1,2,4-triazole-3-thione was synthesized by the ANRORC-type reaction of hydrazine hydrate with the corresponding 1,3,4-oxadiazole-2-thione [497, 498] or by fusing triazolyl acethydrazide with thiocarbazide. Selective modification at the sulfur atom occurs after an arylidene fragment has been introduced at the exocyclic nitrogen atom of 4-amino-1,2,4-triazole-3-thione (Scheme 6.6).

Both among *S*-substituted 1,2,4-triazoles and among 2-(alkylthio)-5-(1,2,4,-triazol-1-ylmethyl)-1,3,4-thiadiazoles and -1,3,4-oxadiazoles, aryloxyalkane derivatives exhibit the highest fungicidal activity [491, 492, 495, 496], and *S*-cinnamyl-5-(azol-1-ylmethyl)-1,2,4-triazole-3-thiones show anthelmintic properties (Schemes 6.4, 6.5) [499].

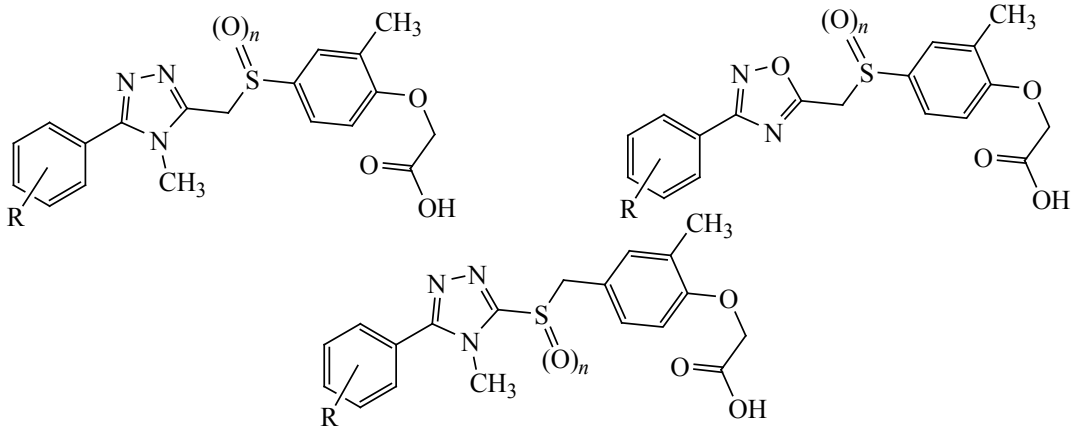
D.V. Minin [500] developed a methodology for searching for new PPARδ/β agonists, analogs of endu-

Scheme 6.6.



R1 = H, Alk, Hlg, NO_2; R2 = Alk, ArAlk, ArOAlk.

Scheme 6.7.



R = Hlg, CF_3, CF_3O; n = 0, 1, 2.

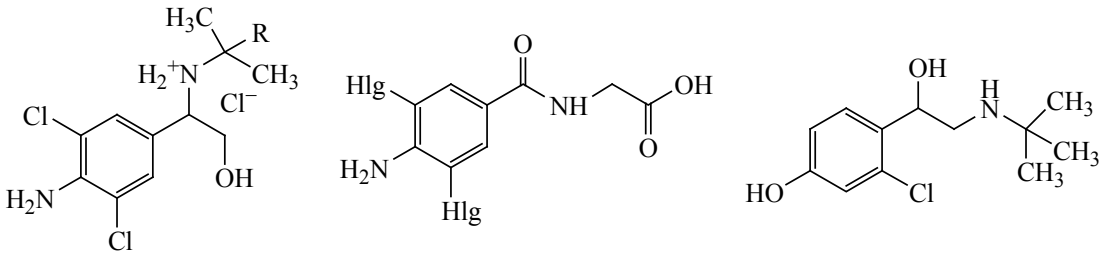
robol [500], in the series of 4-arylazolylmethylthio-2-methylphenoxyacetic acids and their analogs. Based on substituted benzoic acids and benzonitriles, new compounds containing different azole linkers, as well as their oxidation products sulfones were synthesized (Scheme 6.7) [501–503]. The in vitro testing of the synthesized compounds by surface plasmon resonance showed that the most promising potential PPARδ/β agonist was 4-[4-methyl-5-(3,4-dichlorophenyl)-4*H*-1,2,4-triazol-3-yl-methylsulfonyl]-2-methylphenoxyacetic acid.

The research of **M.A. Glushkova** is aimed at searching for new API among β-agonists, developing new synthetic approaches to known drugs tulobuterol [504], brombuterol, clenproperol, and their analogs (Scheme 6.8) [505], preparing their metabolites, and studying pharmacokinetic parameters [506].

The research group of **Dr. Chem. Sci., Professor RAS A.O. Terent'ev** in close cooperation with the Laboratory of Homolytic Reactions of the Zelinskii Institute of Organic Chemistry RAS, conduct studies on the synthesis and biological activity of organic peroxides [507–509], thiocyanates [510, 511], functional derivatives of pyrazolidinone [512, 513], tetrahydroquinoline [514], and bis(dialkylthiocarbamoyl) disulfides [515]. The synthesized compounds exhibit high fungicidal activity and are more potent fungicides than the standards triadimefon and kresoxim-methyl. Efficient synthesis of most of the mentioned classes of fungicidal compounds became possible owing to the procedures of oxidative coupling and peroxidation, developed by Terent'ev's group. The high-yield oxidative coupling of malonyl peroxide with enol esters provides access to monoesters of 2,2-disubstituted malonic acids and β-hydroxy ketones (Scheme 6.9) [516].

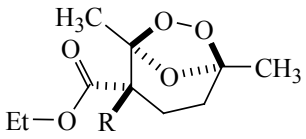
The research group of **Cand. Chem. Sci., Associate Professor V.V. Zakharychev and Cand. Chem. Sci., Associate Professor A.V. Kuzenkov** is focused on the search for biologically active compounds in the series of substituted pyridines. Pyridyl ketone oximes and their *O*-alkyl and *O*-benzyl derivatives were synthesized [517, 518]. Aldonitrones synthesized from nicotinic aldehyde were subjected to the 1,3-cycloaddition reaction with ethyl acrylate or styrenes to obtain 2-aryl-3-(3-pyri-

Scheme 6.8.

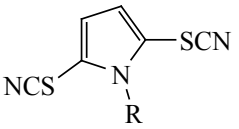


R = H, C_2H_5.

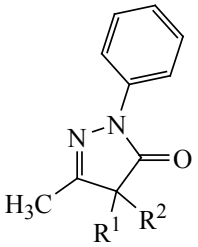
Scheme 6.9.



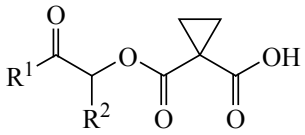
R = Alk, $ArCH_2$.



R = Alk.

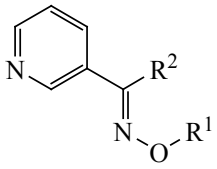


R^1, R^2 = CH_3, NO_2, N_3, Hlg.

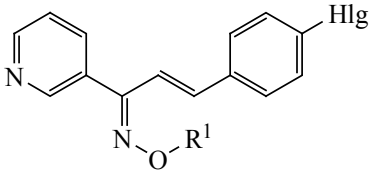


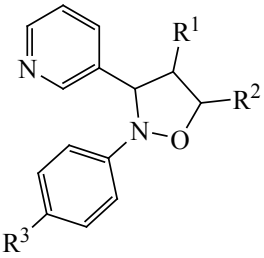
R^1 = Ar, Alk; R^2 = H, Alk.

Scheme 6.10.

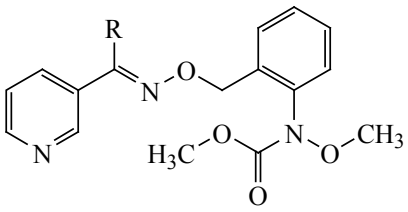


R^1 = Alk, Bn;
R^2 = Ar, Alk.





R^1 = H, EtOOC;
R^2 = H, Ar; R^3 = H, Hlg.



R = H, CH_3, CH_3O, CH_3S.

dyl)isoxazolidines (Scheme 6.10) [519]. Pyridyl-containing compounds exhibit fungicidal activity, and some of them are more active than the standard triadimefon.

Over the past 5 years, **Associate Professor V.V. Zakharychev** has published representative textbooks on chemical crop protection compounds and the chemistry and biological activity of fungicides [520], herbicides [521] and plant growth regulators [522].

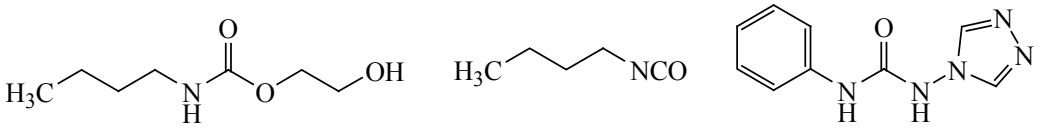
The research group of **Cand. Chem. Sci., Associate Professor S.N. Mantrov** continues research in the field of carbamic acid derivatives, developing phosgene-free syntheses substituted carbamates [523], isocyanates [524] and ureas (Scheme 6.11) [525]. **Cand. Techn. Sci., Assistant R.R. Dashkin** develops technologies for the production of low-tonnage industrial products, including isocyanate production technology with the use of triphosgene [526].

The research of the Department of Chemistry and Technology of Organic Synthesis develops the established scientific school of the chemistry and technology of organic synthesis and biologically active substances in so far as it relates to the search for promising chemical crop protection compounds and active pharmaceutical ingredients and development of import-substituting technologies for biologically active substances.

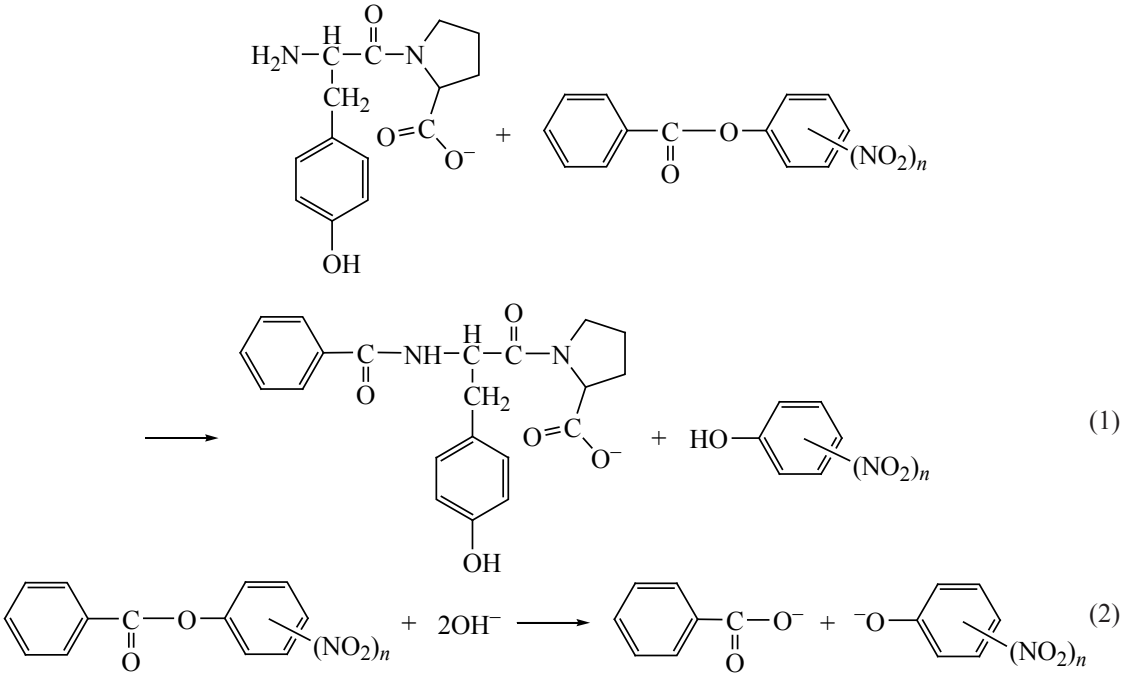
7. DEPARTMENT OF BASIC AND APPLIED CHEMISTRY OF IVANOVO STATE UNIVERSITY

The Department of Basic and Applied Chemistry was created in 2020 on the basis of the Department of Organic and Physical Chemistry as part of the Institute of Mathematics, Information Technology and Natural Sciences of Ivanovo State University (IvSU). The range of scientific areas of the department has expanded significantly and currently includes the kinetics and mechanisms of acyl transfer reactions involving amino acids, dipeptides, amides and hydrazides of aromatic carboxylic and sulfonic acids (**Prof. T.P. Kustova**

Scheme 6.11.



Scheme 7.1.



and Prof. L.B. Kochetova); heterogeneous Pt and Pd catalysts based on activated carbon and carbon nanomaterials: fullerenes, fullerene soot, carbon nanotubes, carbon nanofibers, nanodiamonds, and graphene-like materials for hydrogenation processes: hydrogenation of unsaturated organic compounds and aromatic nitro compounds, hydrodehalogenation of halogenated arenes and carbon tetrachloride, hydrogenation amination of carbonyl compounds with amines, etc. (**Prof. M.V. Klyuev, Assoc. Prof. N.A. Magdalinova**); relationships between structure and physicochemical properties, design of supramolecular mesomorphic materials (**Assoc. Prof. M.S. Fedorov and Assoc. Prof. E.A. Lapykina**); plasma chemical synthesis of water-soluble derivatives of chitosan (**Assoc. Prof. I.K. Naumova**), etc.

The staff of the department in collaboration with the Institute of Chemical Reagents and Highly Pure Chemical Substances, "Kurchatov Institute" National Research Center, have completed a series of works [527, 528] on the reactivity of the L-Tyr-L-Pro dipeptide and its analoge with the carboxyl group reduced to CH_2OH, which have analgesic activity. An experimental study of the kinetics of benzoylation of dipeptides with nitro-activated phenyl benzoates in a water (40 wt %)–1,4-dioxane binary solvent showed that the rate constant of the reaction involving L-Tyr-L-Pro(CH_2OH) is on average 5 times higher than the rate constant of the reaction involving L Tyr-L-Pro (Scheme 7.1) and varies in the range k_{298} = 0.065–2.219 L mol^{-1} s^{-1}.

Comparison of the activation parameters of reaction (1) (Scheme 7.1) with 2,4 dinitrophenyl benzoate and the previously studied reaction with Gly under the same conditions showed that the $\Delta H^{\neq}_{298}$ and $-\Delta S^{\neq}_{298}$ values are close to each other (31–37 kJ mol^{-1} and 134–145 J mol^{-1} K^{-1}, respectively). It was found that the rate of benzoylation of amino acids and dipeptides is determined by the basicity of the amino groups involved in acylation, and a linear correlation was obtained between the log$k$ values of the reactions of amino acids and dipeptides with picryl benzoate and the pK values of their amino groups. This made it possible to predict the rate constants of reactions involving the oligopeptides Tyr-Pro-Phe-Pro-Gly-Pro-Ile (pK = 7.31) and Tyr-Pro-Phe-Val-Glu-Pro-Ile (pK = 7.34): 0.080–0.084 L mol^{-1} s^{-1}.

The mechanisms of acyl transfer reactions of dipeptide have been scarcely studied to date [529]. In [530], computer simulation of the reaction route was performed by calculating the 3D potential energy surface (PES) as a function of the coordinates of the distances between the amine nitrogen atoms of the dipeptides

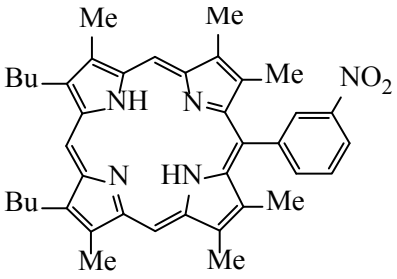
L-Tyr-L-Pro and L-Tyr-L-Pro(CH_2OH) and the ester carbonyl carbon atom, which were varied from 4.0 to 1.0 E, and the angle of attack of the dipeptide molecule on the ester carbonyl reaction center (NCC), which was varied from 90° (axial *p*-attack) to 180°C. As a rule, the bimolecular concerted mechanism S_N2 was predicted judging from the fact that the PESs of the reactions contained a single saddle point corresponding to the transition state of the reaction and a single minimum corresponding to the products. The reaction is initiated by the axial attack of the nucleophile at an angle of 90–100°C, and then, as the reacting molecules approach each other, the angle of the attack significantly increases. The carbonyl reaction center in the transition state (TS) of the reaction tends to adopt a tetrahedral configuration, the sum of the orders of the breaking and forming bonds in it is usually larger than one, implying the formation of a compressed TS. At the same time, the reactions of tyrosylproline with 4-nitrophenylbenzoate proceed by a stepwise mechanism, as evidenced by the presence of two saddle points on the PES of each process, which corresponding to two TSs, and an additional minimum corresponding to the intermediate reaction product. The geometries of both TSs and the intermediate are close to tetrahedral. In the first TS, the formation of an amide bond begins, while the bond with the leaving group is preserved. Then an intermediate is formed, where the bond with the leaving group is loosened, and the amide bond is formed to a slightly greater extent. The second PS precedes the elimination of the leaving group, and the amide bond has already been formed.

Along with dipeptides, the last 5 years the department has been actively studying the benzoylation of individual amino acids and their functional derivatives [531, 532] in aqueous organic media. An experimental study of the kinetics of the reaction of ethyl ester of D,L leucine with 2,4-dinitrophenyl and 2,4,6-trinitrophenyl benzoates binary aqueous organic solvents with ethanol, isopropanol, acetonitrile, and 1,4-dioxane as a nonaqueous component allowed to determine the range of rate constants (k_{298} = 0.011–1.45 L mol $^{-1}$ s $^{-1}$) and the activation barriers of the reactions. It was shown that the reaction rate constant increased significantly as the proportion of water in the binary solvent increased from 20 to 80 wt %, and a compensatory effect from the solvent composition of the solvent was discovered. The results of the kinetic studies showed that aqueous alcohols are the most preferred solvent for the benzoylation of ethyl ester of leucine.

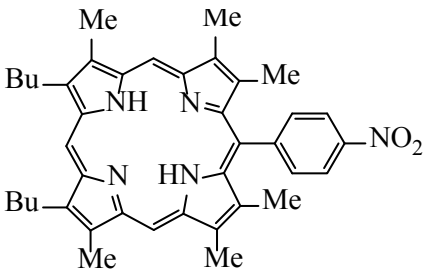
Another line of kinetic research at the department is the sulfonylation of amides and hydrazides of aromatic carboxylic and sulfonic acids [533, 534]. The following reactivity series was obtained in aqueous 1,4-dioxane (ω_{H_2O} = 20 wt %): benzoic acid amide > benzoic acid hydrazide > benzenesulfonic acid hydrazide > benzenesulfonic acid amide > saccharin, and, therewith, the rate constant of the reaction of benzamide with 3-nitrobenzenesulfonyl chloride (k_{298} = 0.039 L mol^{-1} s^{-1}) is higher than that of the reaction with saccharin more than 200 times. The results of computer simulation of the 3D PES of the reaction in the gas phase showed that the process follows a route starting with an axial attack of the nucleophile, which decreases during reaction. The reaction mechanism can be described as the bimolecular concerted nucleophilic substitution S_N2 with the formation of a single activated complex.

One of the areas of focus of the department, developed under the supervision of **Prof. M.V. Klyuev**, involves the development of heterogeneous platinum and palladium catalysts for hydrogenation processes based on activated carbon, which are actively used in industrial synthesis and carbon nanomaterials: fullerenes, fullerene soot, carbon nanotubes, carbon nanofibers, nanodiamonds, and graphene-like materials [535]. Over the past few years, the developed catalysts have been studied in the hydrogenation of a number of unsaturated organic compounds [535, 536], aromatic nitro compounds [535, 536], nitro-substituted macroheterocyclic compounds (Fig. 7.1) [536] and metal complexes (Scheme 7.2) [537], hydrodehalogenation of haloarenes [536] and tetrachloromethane [538], hydrogenative amination of carbonyl compounds with amines or nitro compounds (Scheme 7.3) [536, 539, 540], and reductive cyclization of nitrophenoxy ketones to form 2,3,4,4a,10,10a-hexahydro-1*H*-phenoxazines (Scheme 7.4) [541]. These reactions are important for preparing synthetically and practically important products, including potentially biologically active products [540, 541], and the formation of amino groups in macroheterocyclic compounds makes possible their further modification by alkylation or acylation reactions.

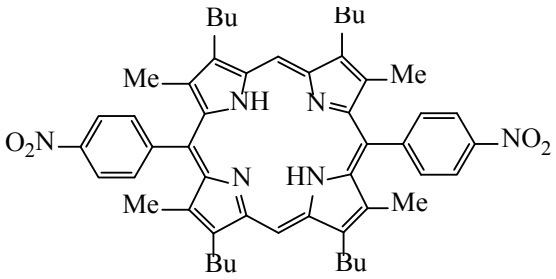
It was found that metal–carbon nanomaterials turned out to be a few times more catalytically active, selective,



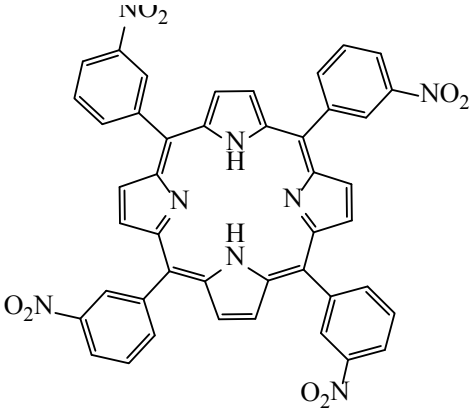
5-(3-Nitrophenyl)-2,3,7,8,12,18-hexamethyl-13,17-di-*n*-butylporphine



5-(4-Nitrophenyl)-2,3,7,8,12,18-hexamethyl-13,17-di-*n*-butylporphine



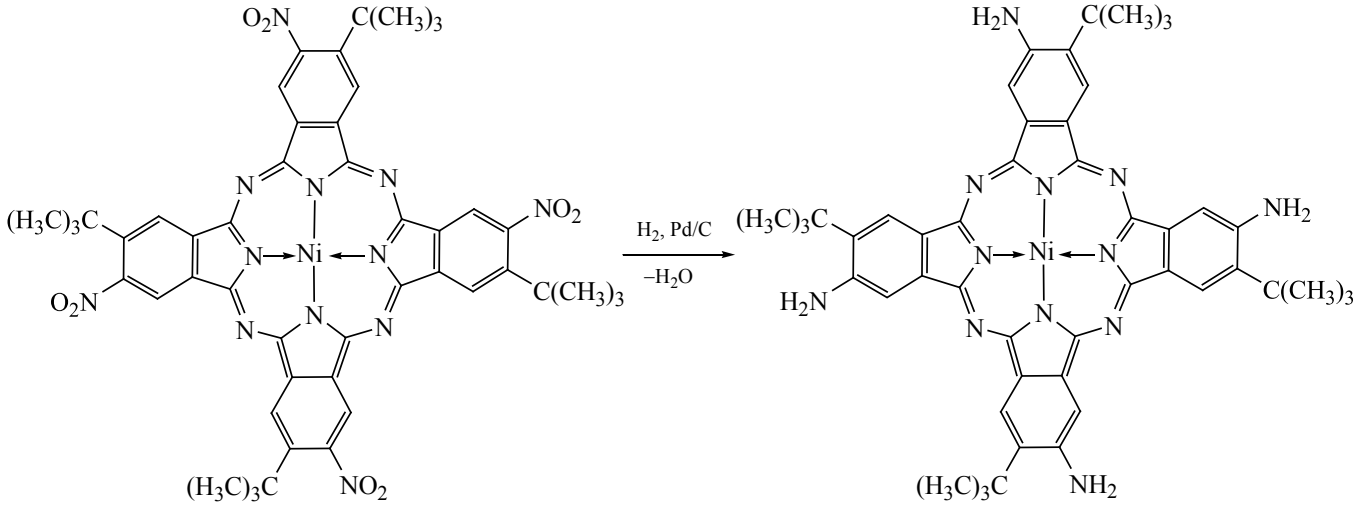
5,15-Bis(4-nitrophenyl)-3,7,13,17-tetramethyl-2,8,12,18-tetra-*n*-butylporphine



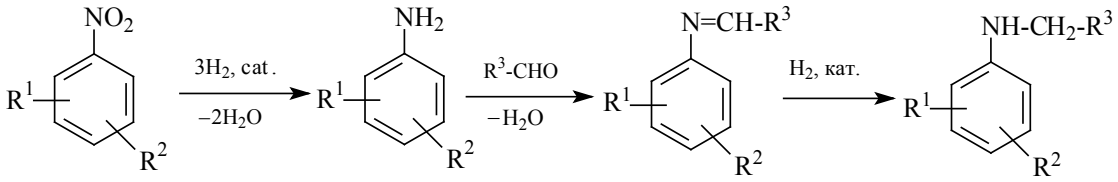
5,10,15,20-Tetrakis(3-nitrophenyl)porphine

Fig. 7.1. Structures of nitro-substituted porphines.

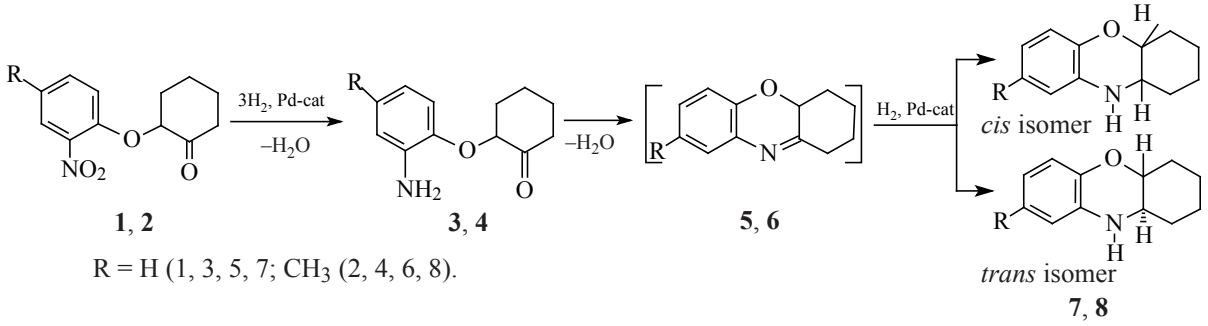
Scheme 7.2.



Scheme 7.3.



Scheme 7.4.



and stable in the studied reactions than commercial Pt/C and Pd/C catalysts [535]. In the hydrogenation of nitrobenzene, carbon nanotube-, nanofiber-, and nanodiamonds-supported platinum catalysts are 1.5–3.8 times more active than the Pt/C catalyst (E-TEK). The same is true of carbon-supported palladium catalysts; in particular, samples based on nanodiamonds showed good performance and found to be 1.5–10 times more potent than commercial 1 wt % Pd/C (E-TEK). In a study of their stability in the hydrogenation of five successive portions of nitrobenzene, the effect of catalyst development was discovered [535]. The TOF activity of palladium-doped nanodiamonds (1 wt % Pd) in the synthesis of 2,3,4,4a,10,10a-hexahydro-1*H*-phenoxazines (the structure was confirmed by NMR; see Scheme 7.4) in quantitative yields was 80 min^{-1}, which is 3 times that of conventional 1 wt % Pd/C (TOF = 26 min^{-1}) [541].

The research on the molecular structure of different classes of organic compounds and the search for structure–property relationships, traditionally carried out at the department under the supervision of **Prof. N.I. Giricheva**, are currently continued in the theoretical and experimental work on the design of supramolecular mesogens. These studies were supported in different years by the Ministry of Science and Higher Education, as well as by the Russian Science Foundation (project manager **Assoc. Prof. M.S. Fedorov**). Knowledge of the features of structure–property relationships and regularities of variation in a property over the series of compounds under study allows one to propose the principles of design and the most promising compounds for specific practical applications. The process of self-assembly of molecules into mesomorphic supramolecular complexes is complex and multifactorial; therefore, to study it, an integrated approach combining modern methods of quantum-chemical modeling of structure and properties, as well as many experimental methods is applied.

The works carried out over the past few years have revealed relationships between the structure and mesomorphic properties for a number of supramolecular

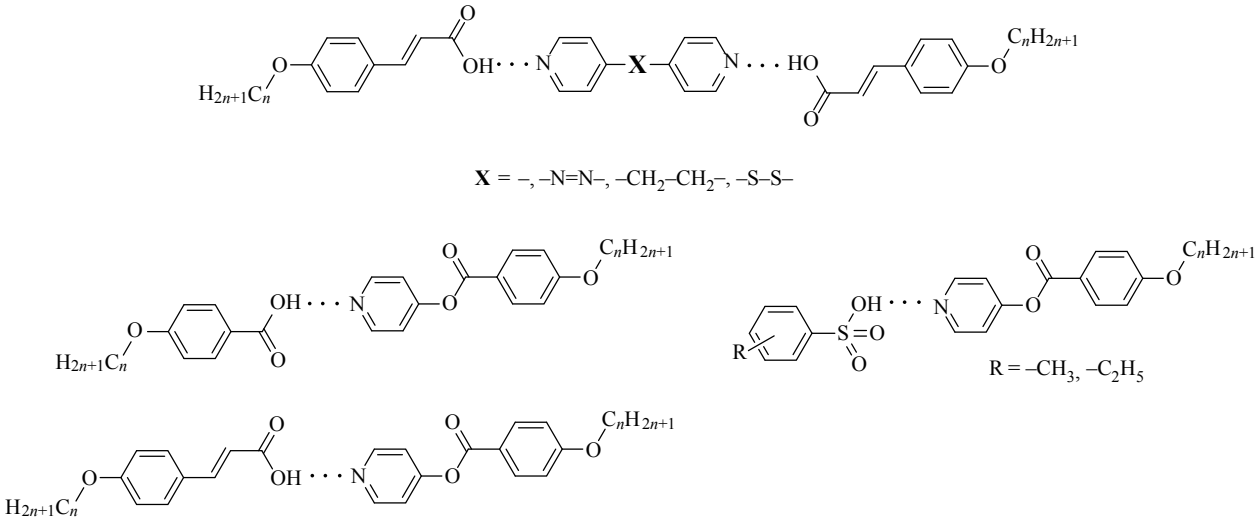


Fig. 7.2. Structural formulas of some studied mesomorphic H-complexes.

complexes based on aromatic carboxylic and sulfonic acids (Fig. 7.2). For example, it has been shown that the structural rigidity and geometric shape of the central molecule significantly affect both the geometry and mesomorphic properties of the H-complexes formed in the systems containing 4-alkyloxycinnamic acids and 4,4'-bipyridine and its analogs with different bridging groups(component ratio 2 : 1, respectively). Complexes of the more structurally rigid 4,4'-bipyridine and 4,4'-azopyridine have the highest thermal stability of the mesophase [542, 543]. At the same time, with increasing length of the alkyloxy substituent in the acid molecule, the probability of inducing a nematic mesophase decreases.

Complexes of alkoxy-substituted benzoic and cinnamic acids with anisotropic pyridine derivatives (pyridin-4-yl 4-alkyloxybenzoates, etc.) were obtained. The changes in the vibrational spectra, induced by the destruction of cyclic acid dimers and the formation of their H-complexes with pyridine derivatives were studied in detail. For example, it was shown that the structural units of the 1 : 1 systems based on 4-dodecyloxybenzoic acid (A) and pyridin-4-yl 4-(dodecyloxy)benzoates (B) are H-complexes of the A···B type, while in the 2A : 1B system, instead of stoichiometric A···A···B and A···B···A complexes, a mixture of A···B complexes and cyclic acid dimers formed during self-assembly were detected [544]. The relationship between structural non-rigidity and mesomorphic properties was also studied for similar systems. Comparison of the structural and dynamic nonrigidity of cyclic dimers of aromatic carboxylic acids and their H-complexes with pyridine derivatives showed that the structural nonrigidity of the central fragment of the complex strongly increases both the vibration amplitudes of the core and the terminal groups of the substituents and decreases the crystal–liquid crystal transition temperature [545].

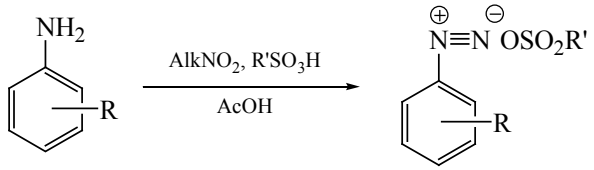
Evidence for the ability of sulfonic acids to form H-complexes with anisotropic pyridine derivatives, which exhibit mesomorphic properties, was obtained for the first time [546, 547]. Even nonmesogenic acids are capable of inducing a mesophase in complexes with anisotropic pyridine derivatives (Fig. 7.2). The main difference of sulfonic acids from carboxylic is that the formation of the intermolecular H-bond between the sulfonyl group and the pyridine fragment is accompanied by proton transfer, as evidenced by the results of analysis of the vibrational spectra. In addition, due to the specific features of the geometry of the sulfonyl group, the resulting H-complexes can be not rod-shaped and have a higher structural flexibility compared to the H-complexes of carboxylic acids.

Since 2022, the department has been developing a new scientific direction in partnership with the staff members of the Krestov Institute of Solution Chemistry, Russian Academy of Sciences, specifically, the development of new biocompatible and biodegradable materials for medicine and agriculture. A number of works on the modification of chitosan, aimed at producing its water-soluble fractions with bactericidal and phytostimulating properties, have been carried out. In 2022, Khlyustova et al. [548] explored the modification of chitosan by underwater AC discharge and DC glow discharge at atmospheric pressure is considered. Water-soluble fractions of chitosan were obtained by gas discharge treatment for 5–20 min of a 1% chitosan solution in acetic acid, whose concentration was varied from 0.5 to 2%. It was found that after 20-min treatment, the molecular weight of chitosan with the initial molecular weight of 195 kDa and deacetylation degree of 0.82 decreased to 15-40 kDa.

Interesting results were obtained in [549] in the study on the one-step synthesis of multicomponent polymer nanocomposites containing zinc oxide and copper oxide nanoparticles based on chitosan and polyvinyl alcohol using underwater pulsed discharge plasma. Direct initiation of the discharge between metal electrodes in the polymer solution allows reagent-free production of metal oxide nanoparticles with an average diameter of about 30 nm. The inclusion of metal nanoparticles in chitosan films was confirmed by X-ray structural analysis. An analysis of the TEM images showed that the nanoparticles were uniformly dispersed in the polymer matrix. The obtained samples were found to exhibit antimicrobial activity against *Escherichia coli*, *Staphylococcus aureus*, and *Bacillus subtilis*, with the highest antimicrobial effect shown by the polymer film containing 0.35% Cu_2O and 3.41% ZnO nanoparticles. In [550, 551], chitosan dispersions treated with AC discharge between graphite electrodes immersed in a liquid were tested; the total concentration of water-soluble and insoluble fractions of chitosan was 0.2%.

The biological activity of low-molecular-weight water-soluble products obtained under various chitosan processing conditions was assessed in terms of their

Scheme 8.1.



R' = Ts, Tf.

effect on the germination of seeds of a number of agricultural crops (barley, wheat, flax, peas, cucumbers, and tomatoes), the rate of early plant development, as well as the ability to suppress the growth of pathogenic microflora. It was found that the synthesized chitooligosaccharides are not inferior to the commercial growth stimulator Epin-extra in the phytostimulating activity. Thus, the germination of peas in Petri dishes showed 89% germination against 84% germination in control (ordinary water), and the degree of seed swelling increased. It was found that extracts from dried greens of cucumbers and peas, which levels, indicating more active photosynthesis.

8. NEW METHODS AND REAGENTS FOR THE SYNTHESIS OF BIOLOGICALLY ACTIVE SUBSTANCES AND MATERIALS FOR MEDICAL PURPOSES (RESEARCH AT THE KIZHNER SCIENTIFIC AND EDUCATIONAL CENTER)

The Kizhner Scientific and Educational Center (Kizhner SEC) became the successor (2017) of the Department of Organic Chemistry, one of the oldest departments of Tomsk Polytechnic University founded in 1901 by Academician N.M. Kizhner. In the subsequent years, the department obtained some priority results in aromatic iodination, discovered new reagents for the oxidation of multiple bonds to 1,2-diketones, and developed convenient and effective methods for the diazotization–iododeamination of aromatic and heteroaromatic amines [552].

At present, the staff of Kizhner SEC continues research into the chemistry of diazonium compounds [553]. A wide range of arenediazonium sulfonates (tosylates, triflates) have been obtained and studied. This is a unique class of aromatic diazonium salts that are stable on storage, safe to work with, readily soluble in water and organic media of various polarities, and exhibit high activity in reactions typical of diazonium salts (Scheme 8.1) [553–555].

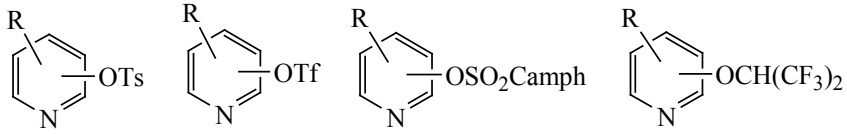
The diazotization of aminopyridines and aminoquinolines in the presence of sulfonic acids leads to extremely unstable diazonium salts, which rapidly transform into sulfonic acid esters. Based on this finding, a general approach to the synthesis of pyridyl sulfonates (tosylates **1**, triflates **2**, and camphorsulfonates **3**), valuable intermediate products of organic synthesis (Scheme 8.2) [553, 556]. It was shown that the diazotization of aminopyridines in hexafluoroisopropyl alcohol yields (hexafluoroisopropoxy)pyridines **4** (Scheme 8.2) [557]. Anilines are practically inert under these conditions.

It was first established that heating pyridyl triflates **2** in DMF allows the substitution of the triflate group, resulting in the formation of (*N*,*N*-dimethylamino)-pyridines. The reaction is accelerated by MW irradiation. These results were used to develop a one-pot synthesis of 2- and 4-(*N*,*N*-dimethylamino)pyridines **5** from commercially available aminopyridines (Scheme 8.3).

The method provides good yields of target products and is an alternative to the existing methods of synthesis of synthesis of *N*,*N*-dimethylpyridin-4-amine (DMAP), a catalyst widely used in organic synthesis [558].

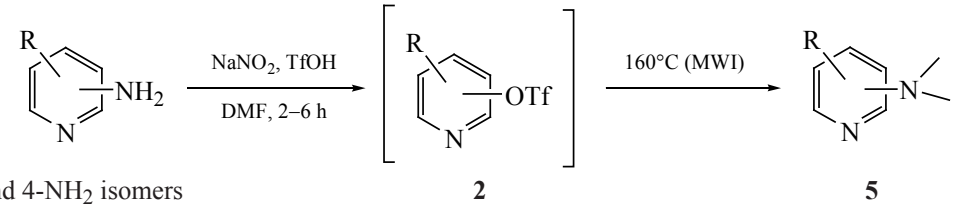
Research in the field of the chemistry of diazonium compounds was also aimed at finding new approaches to

Scheme 8.2.



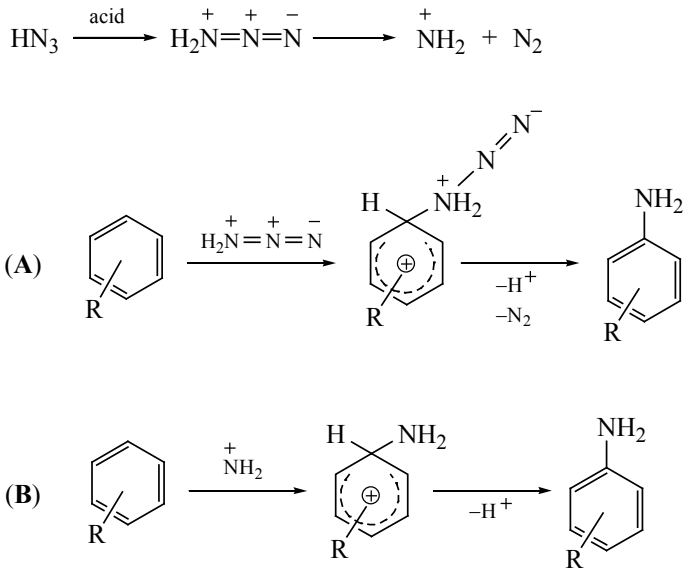
1 **2** **3** **4**

Scheme 8.3.



2- and 4-NH_2 isomers **2** **5**

Scheme 8.4.

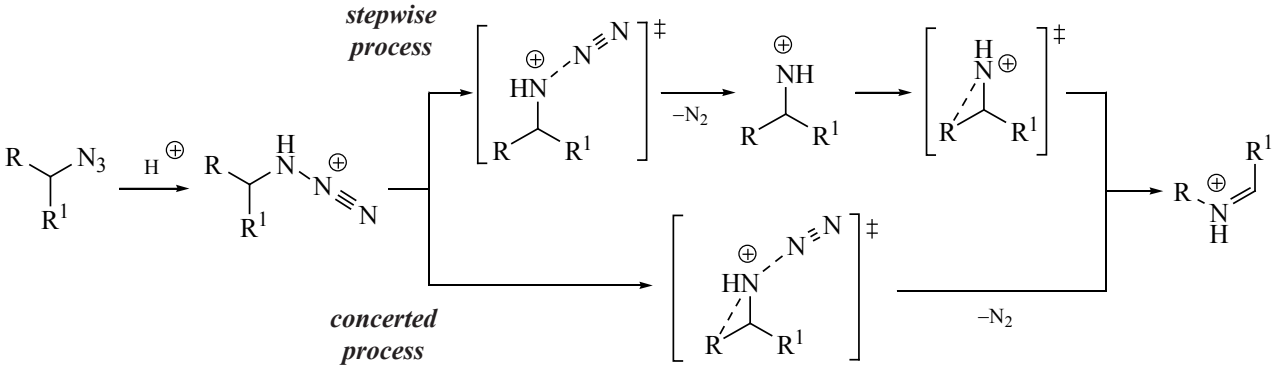


the implementation of intermolecular and intramolecular electrophilic aromatic amination reactions using aminodiazonium cations $R_2N–N_2^+$ as a special type of diazonium compounds. In particular, the mechanisms of $R_2N–N_2^+$ (protonated hydrazoic acid) generation and possible routes of its reactions with aromatic compounds were studied by experimental and theoretical methods [559]. It was established that of the two possible reaction routes (**A** and **B**) of electrophilic amination of arenes (Scheme 8.4), route **A** is more probable. The effect of substituents in RC_6H_5 on the energies and structural parameters of the transition states of electrophilic amination was also elucidated [559].

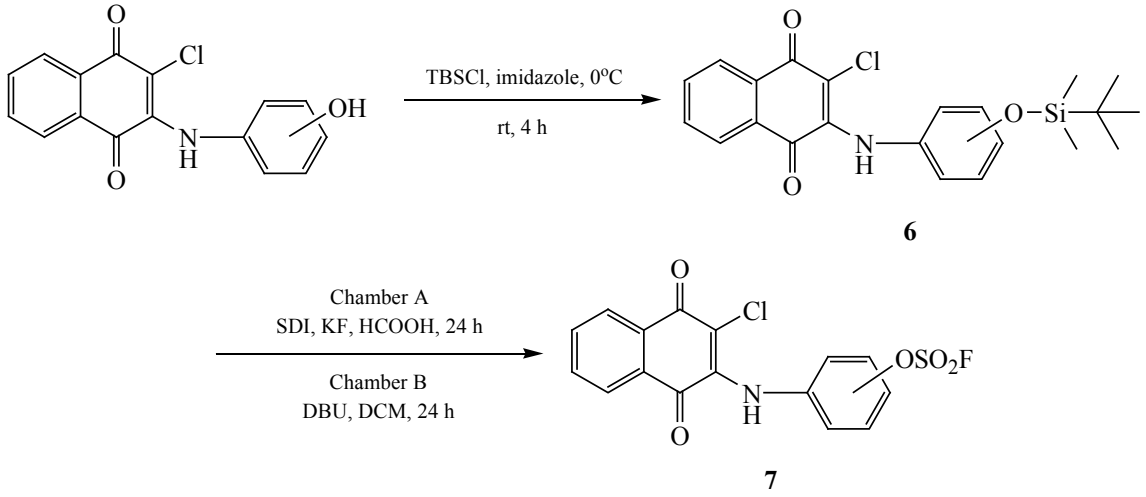
Stankevich et al. [560] made use of B3LYP/aug-cc-pvdz and MP2/aug-cc-pvtz calculations to simulate all stages of the intramolecular electrophilic amination of azides $Ar(Het)CH(Alk)N_3$ initiated by their protonation. The calculations showed that synchronous rearrangement is a much more favorable route (Scheme 8.5).

Recently Danilenko et al. [561] have studied the features of the SuFEx (Sulfur Fluoride Exchange)

Scheme 8.5.



Scheme 8.6.



6

7

click reaction, which allows the synthesis of valuable sulfur-containing organic compounds. In particular, new fluorosulfate phenol derivatives **7** based on 1,4-naphthoquinone were obtained by this reaction. It was found that the best substrates for preparing the target fluorosulfates are silyl ethers **6** derived from these phenols (Scheme 8.6).

Previously unknown fluorosulfate derivatives of 2-(hydroxyphenyl)benzoxazoles were obtained. It was found that increasing the pressure of gaseous SO_2F_2 increases the selectivity of the SuFEx reaction in favor of the target fluorosulfate by reducing the amount of the by-product sulfate with two benzoxazole rings (Scheme 8.7). The presilylation of the hydroxyl group in this case adversely affects the fluorosulfate yield.

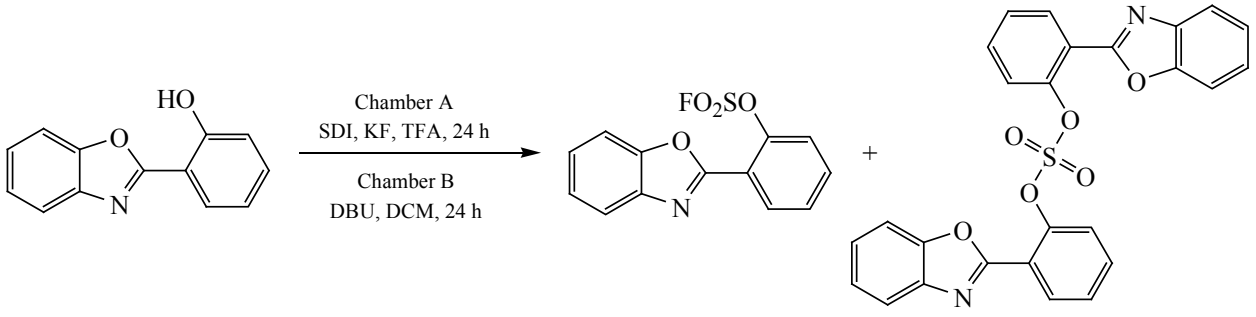
In 2020, Kizhner SEC set up the Molecular Modeling and Synthesis of Biologically Active Compounds laboratory. The laboratory continues research in the chemistry and technology of medicinal substances, initiated in the middle of the past century by Prof. L.P. Kulev [552].

The work on the synthesis of linear and cyclic derivatives of ureas as anticonvulsants was given a new impetus. Thus, the enantiomeric composition of 1-phenyl(3-chlorophenyl)-methylurea in the racemic drug Galodif **8** was determined for the first time by NMR and chiral HPLC [562]. The (R–) and (S+) diastereomers of *N*-[(3-chlorophenyl)(phenyl)methyl]-camphor-sulfonamides **9** were obtained and identified as potential precursors of Galodif in asymmetric syntheses (Scheme 8.8).

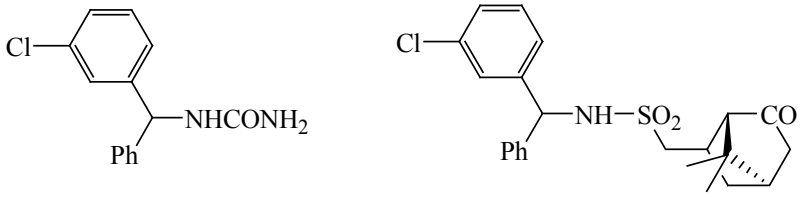
The mechanism of the pharmacological action of Galodif **8** is unknown; presumably, the activity of this drug is underlain by interaction with the γ-aminobutyric acid A ($GABA_A$) receptor. The stttrength of interaction of Galodif with the benzodiazepine site of $GABA_A$ was assessed using molecular docking. More than 10 different 3D conformations with potential energies close to the theoretical minimum were obtained [563].

The water-soluble 4-oxo-4-{3-[phenyl(3-chlorophenyl)methyl]ureido}butanoic acid (**9**), a suitable candidate

Scheme 8.7.

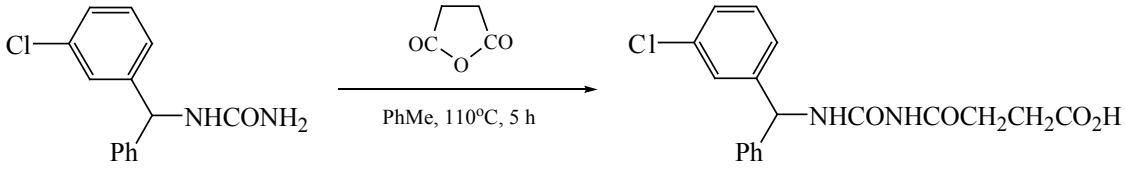


Scheme 8.8.



8 **9**

Scheme 8.9.



8 **9**

for the development of a water-soluble dosage form of Galodif (Scheme 8.9), was obtained for the first time [564]. Practical voltammetric methods for the analysis of dosage forms of Galodif were developed [565].

The laboratory is actively working on the development of JNK kinase inhibitors. A number of new oximes based on 11*H*-indeno[1,2-*b*]quinoxalin-11-one **10** and quinoxaline **11** (Scheme 8.10) were synthesized and studied, and their physicochemical and biological properties were studied [566–573].

The developed procedures allow the facile synthesis of various oximes with substituents both in the heterocyclic moiety and at the oxygen atom in high yields without the use of expensive reagents. The luminescence of tryptanthrin derivatives with a modified oxime group was studied by experimental and quantum-chemical methods. Some tryptanthrin and 11*H*-indeno[1,2-*b*]quinoxaline derivatives exhibit high inhibitory activities with respect to enzymes of the JNK family and high anti-inflammatory activities, which gives grounds to consider them as perspective candidates for application as anti-inflammatory, anti-ischemic, and antihypertensive agents.

Previously unknown derivatives of 1,4-benzodiazepin-2-one (BD) **12–15**, which showed a broad spectrum of biological activity, were synthesized. Thus, it was established that 3-acyloxy derivatives **12** exhibit affinity for central benzodiazepine receptors (CBDR), as well as and hypnosedative and anxiolytic properties (Scheme 8.11) [574]. It was established that the derivatives containing an *ortho*-chlorin group in the 5-phenyl substituent have the highest affinity; furthermore, the hydrocarbon chain length in the acyl residue affects the strength of the affinity.

The first obtained 3-phthalimidoacyloxy derivatives **13** and **14** (Scheme 8.12) [575] show, along with a high antihypoxic activity, anticonvulsant and antitoxic properties, and also improve long-term memory and learning ability. It is important to note that derivatives **13** and **14** are low-toxic: LD50 > 550 mg/kg.

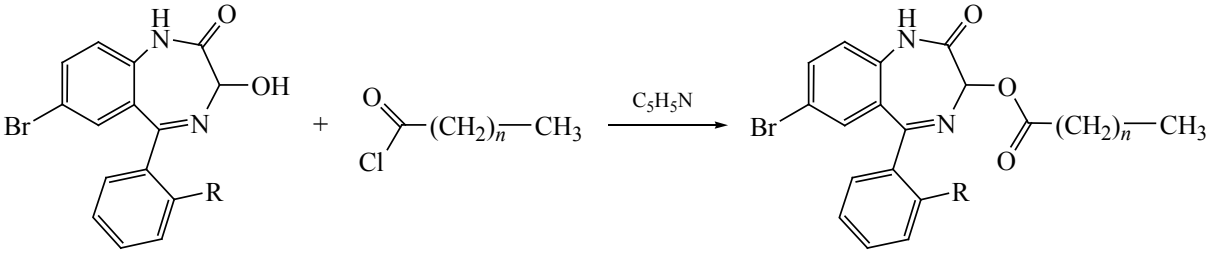
It was found that previously unknown 3-arylamino derivatives **15** [576] at low doses (2.07–2.21 μM) have a

Scheme 8.10.

10 **11**

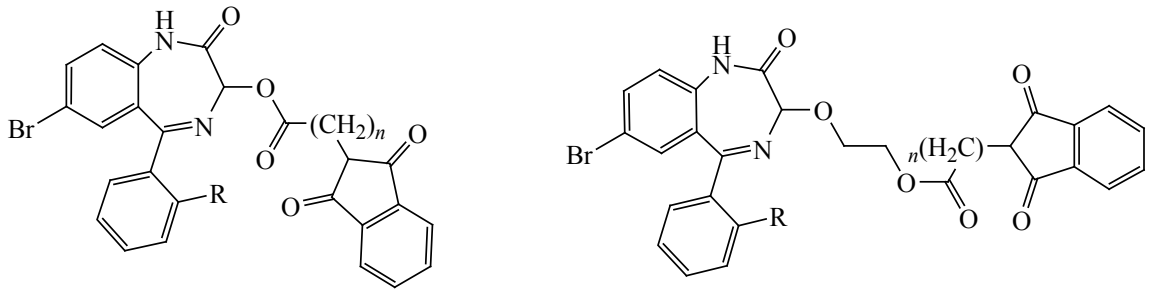
11*H*-Indeno[1,2-*b*]quinoxalin-11-one

Scheme 8.11.



R = H, Cl; n = 0–10. **12**

Scheme 8.12.



13 **14**

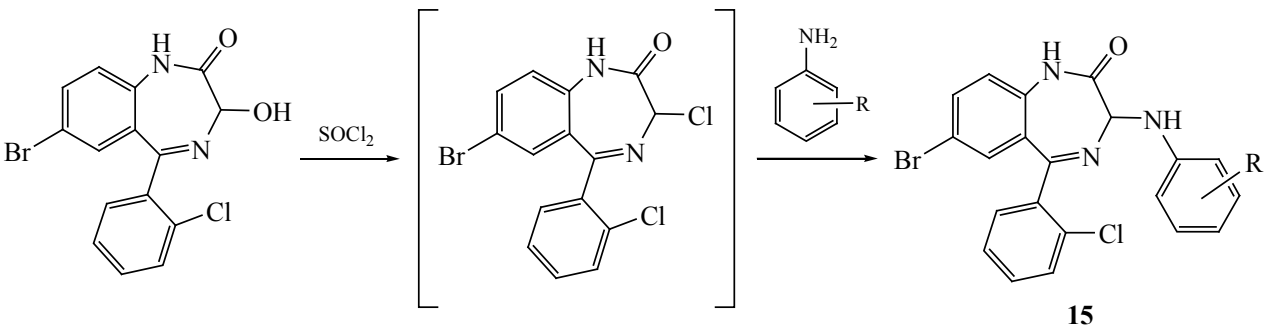
significant effect on appetite, exhibiting both orexigenic and anorexigenic properties (Scheme 8.13).

In collaboration with the Weinberg Scientific and Educational Center, Tomsk Polytechnic University, and some biomedical organizations, Kizhner SEC developed new composite materials for use as implants and means of targeted drug delivery.

A method of surface modification of poly(lactic acid) (PLA)-based medical devices was proposed and implemented for the first time. This method involves pretreatment with a "good/bad" solvent mixture and allows immobilization of 5-amino-2-(benzo[*d*]oxazol-2-yl)phenol or *N*-{2-(2-hydroxyphenyl)benzo[*d*]oxazol-5-yl}acetamide as fluorescent labels, as well as polyacrylic acid [577], hyaluronic acid, and gelatin [578–580] on the surface of PLA films. A method for assessing the effectiveness of this immobilization was developed [581].

Composite materials based on poly(e-caprolactone) (PCL) filled with L-arginine [582], a JNK kinase inhibitor [583–585], and Doxorubicin [586] were obtained. Thus, new PCL matrices containing naphthoquinone derivatives ensure prolonged release of the drug from the matrix; at the same time, increasing drug concentration increases the cytotoxicity of the matrices in relation to the MonoMac-6 cell line [585]. Thus, the obtained scaffolds can be used as medical materials with anticancer and other useful properties.

Scheme 8.13.



15

Currently, new medical materials based on a copolymer of polycaprolactone and poly(vinylpyrrolidone) were fabricated by electrospinning in hexafluoropropanol as a solvent [586, 587].

Scientific research at the Kizhner Scientific and Educational Center was supported by grants from the Russian Foundation for Basic Research, Russian Science Foundation, the Ministry of Science and Higher Education of the Russian Federation through state assignments, and TPU grants within the framework of the 5-100 and Priority 20230 programs.

The work was carried out under the state assignment of the Ministry of Science and Higher Education of the Russian Federation (topic: Science; no. FSWW-2023-0008).

9. DEPARTMENT OF ORGANIC CHEMISTRY OF LOBACHEVSKY NIZHNY NOVGOROD STATE UNIVERSITY

Over the past few years, the Department of Organic Chemistry of the Faculty of Chemistry of Lobachevsky Nizhny Novgorod State University has been actively developing a few scientific areas in the field of medicinal chemistry, synthetic organic chemistry, and the chemistry of polymer and organoelement compounds.

The lines of research of the department in the field of medicinal chemistry include (a) the chemistry of conjugates with enzymatically cleavable linker for targeted delivery of natural porphyrins for photodynamic therapy (PDT) and imaging, as effective agents for combined anticancer therapy [588]; (b) the design and synthesis of analogs of natural alkaloids, specifically heterocyclic colchicinoids, which exhibit antitumor, antifibrotic, antiviral, and anti-inflammatory properties, and systems for their targeted delivery [589]; and (c) the synthesis of oligopeptide complexes of iron carbonyl, capable of releasing therapeutic amounts of carbon monoxide under the action of enzymes (ET-CORMs). In addition, the study of the structures and molecular dynamics of BET proteins made it possible to create the first selective inhibitors of BET protein bromodomains involved in the epigenetic mechanisms of chromatin modification and regulating oncogene expression [590]. Inhibitors of the PI3K/Akt/mTOR signaling pathway [591] and PROTAC agents targeting the degradation of oncogenic proteins [592] were also created.

Porphyrin conjugates for photodynamic therapy. A concept for creating multifunctional conjugates for targeted photodynamic therapy, which selectively accumulate in tumor cells due to interaction with specific growth factor receptors, was put forward. A systematic analysis of this concept is presented in our reviews [588, 593, 594]. The proposed conjugates consist of a chlorin photosensitizer, a natural dihydroporphyrin (highlighted in green in the scheme), a 4-arylaminoquinazoline ligand of the EGFR/VEGFR growth factor receptor (red), and their connecting linker (Scheme 9.1, compounds **1** and **2**). Unfortunately, these compounds are insufficiently soluble for full-fledged biological testing [595]. The introduction of hydrophilizing quaternary ammonium or carbohydrate fragments (compounds **3–8**) into the conjugates made it possible to solve the problem of water solubility [596–599]. It is known that carbohydrate conjugates ensure targeted drug delivery to target cells due to interaction with tumor- and tissue-specific receptors [600].

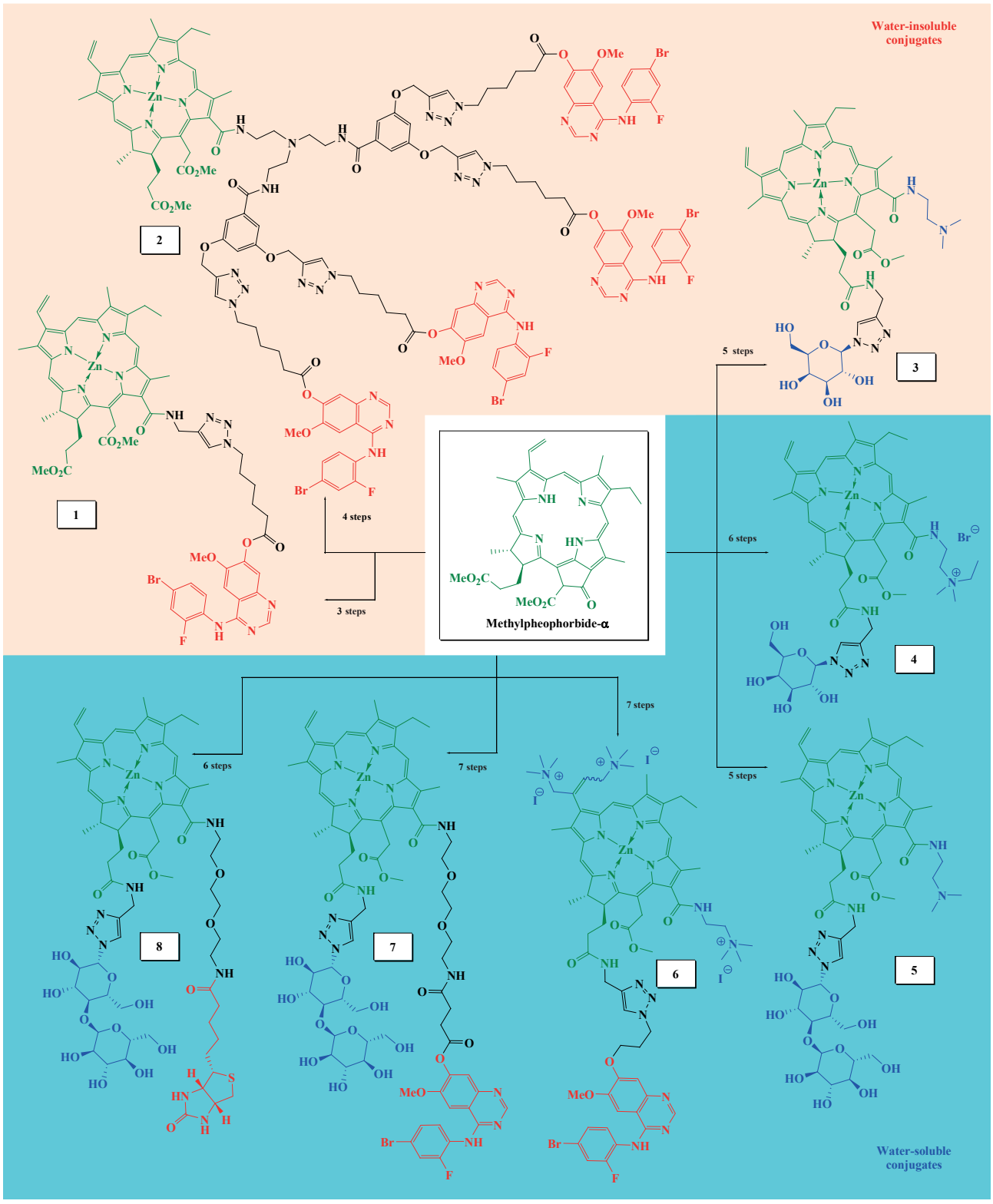
It was found that the photodynamic activity of compounds **4** and **5** is ~50 times higher compared to their dark cytotoxicity: IC_{50} (dark) ~20 μM; IC_{50} (light) ~0.4 μM (irradiation 20 J/cm^2) [596]. For derivatives **6** and **7**, which contain a 4-arylaminoquinazoline vector to growth factor receptors, the phototherapeutic index increases to ~360. These conjugates have a significant photoactivity: IC_{50}(light) ~0.2 μM (irradiation 20 J/cm^2), and, therewith, they effectively inhibit tumor growth in mice without essential side effects [597, 598].

A photocleavable conjugate **9**, which, upon irradiation with light, should release a free synthetic porphyrin **10** photosensitizer and the chemotherapeutic agent combretastatin A-4 **11**, which undergoes photoisomerization and converts into a therapeutically active *Z* form (the principle of action is shown in Scheme 9.2) [601]. It was shown that the acetylenic fragment in conjugate **9** prevents its photoinduced degradation.

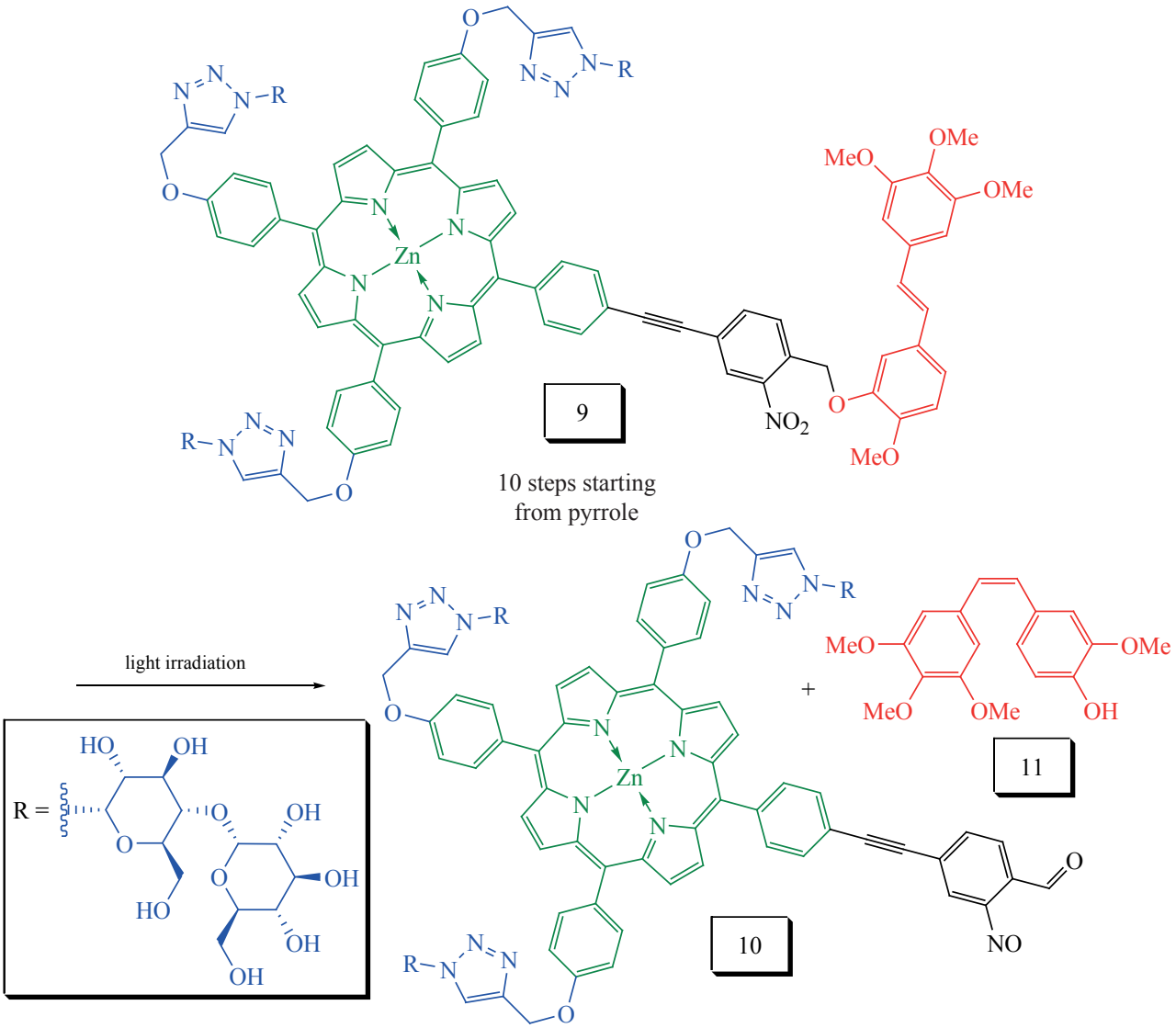
A μ-oxo-Fe^{3+} complex of chlorin-e_6 containing lipoic acid in the side chain was synthesized. This complex can be used as a sensor for the detection and quantitative determination of NO [602].

Therapeutic agents based on colchicine alkaloids. Colchicine (Scheme 9.3) is an alkaloid with a wide range of physiological activity, used in clinical practice to treat gout, Behcet's disease, familial Mediterranean fever, and some other diseases. Colchicine has a high antitumor potential, but its high systemic toxicity prevents its use as an antimitotic drug. Our research is focused on the

Scheme 9.1.


Water-insoluble conjugates
Methylpheophorbide-α
4 steps
3 steps
5 steps
6 steps
7 steps
Water-soluble conjugates

Scheme 9.2.

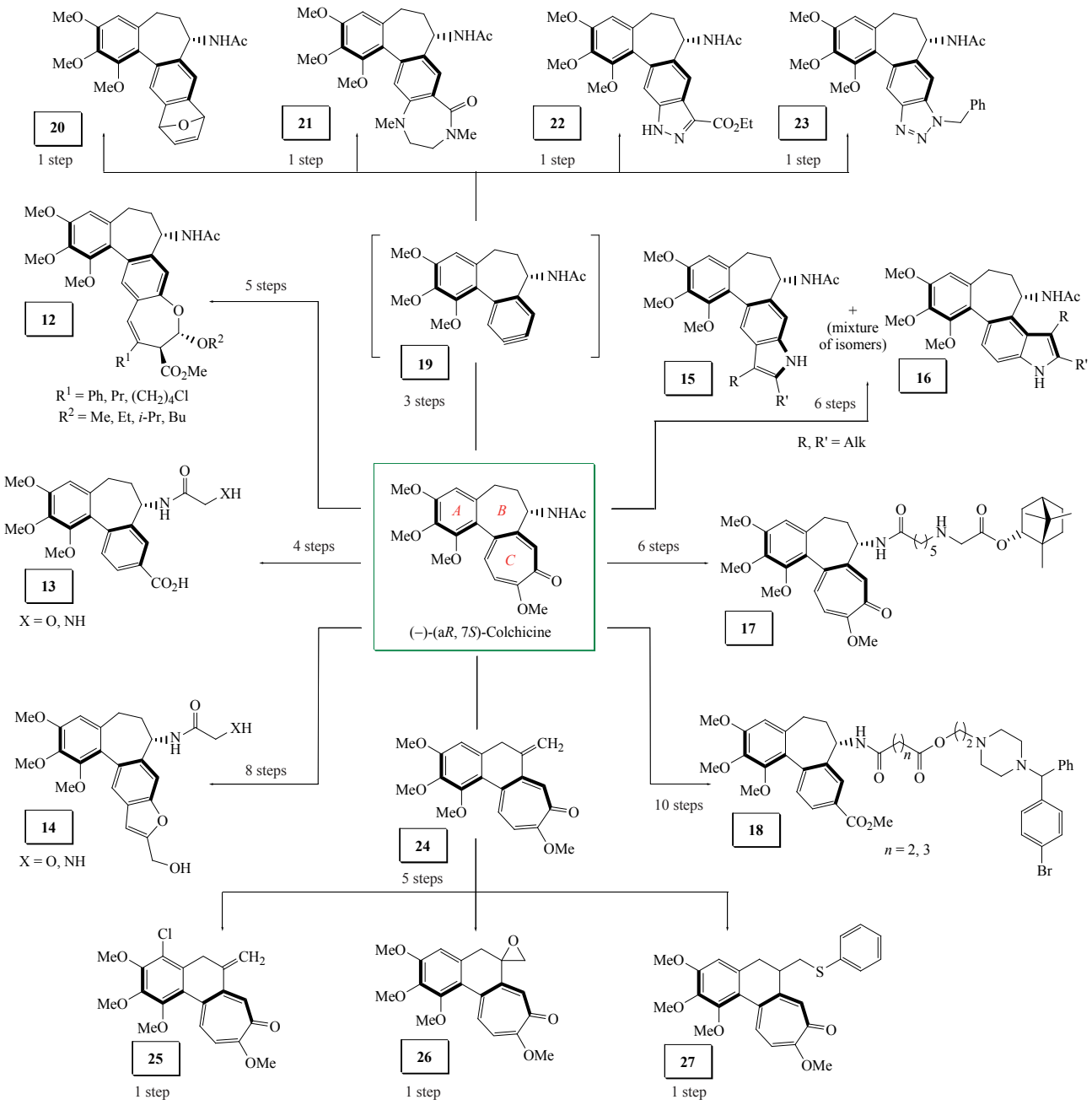


modification of the colchicine core and the synthesis of conjugates of the modified colchicines, which led to a family of colchicinoids with diverse bioactivities.

The colchicine molecule is a convenient chiral building block: the modification of its A, B, and C rings allows the synthesis of different types of bioactive polycyclic compounds with the preservation of the central and axial chirality of the original natural compound. For example, dihydrobenzoxepines **12** (Scheme 9.3) were prepared in 5 steps via gold-catalyzed cyclization; the products demonstrated moderate cytotoxicity [603]. Bifunctional allocolchicinoids **13** and furanoallocolchicinoids **14** were synthesized in 4 and 8 steps, respectively [604, 605], and some of these compounds demonstrated antitumor activity at low nanomolar concentrations. The Fischer indole synthesis was used to obtain pairs of isomeric indoloallocolchicinoids **15** and **16** from colchicine in 6 steps [606]. Indoloallocolchicinoids with structural formula **15** were also synthesized by a Pd-catalyzed intramolecular cascade reaction [607]. The conjugation of colchicine with terpenoids (for example, compound **17**) gave compounds with antiviral activity [608], and compound **18**, which contains an analog of cetirizine, was synthesized as potential anti-inflammatory and antiallergic agent [609].

A universal synthetic approach to heterocyclic colchicinoids, involving the intermediate formation of aryne **19** in 3 steps from colchicine, was developed. It

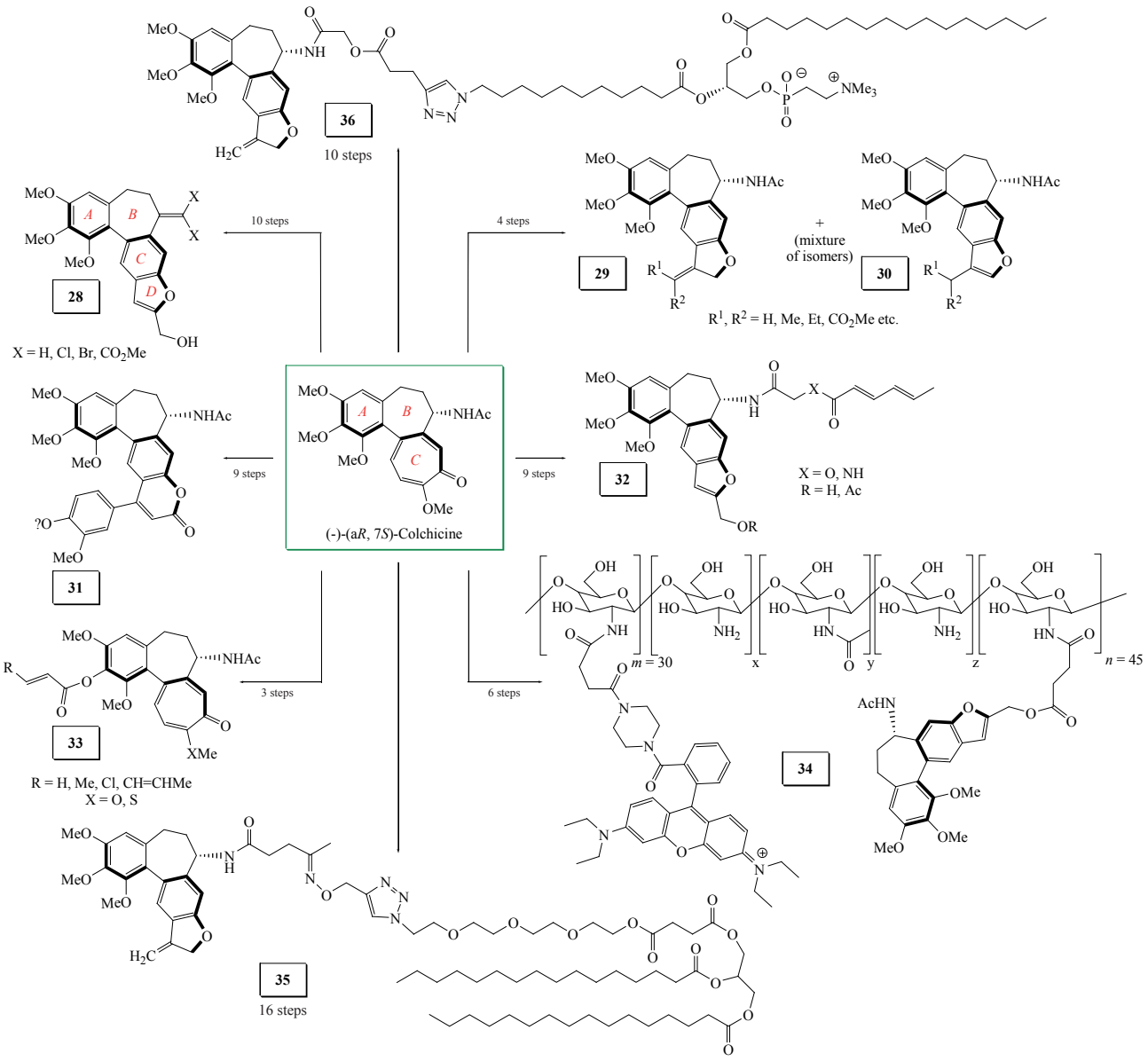
Scheme 9.3.



should be noted that aryne **19** is the first example of an aryne featuring a combination of the central and axial chirality. Aryne **19** undergoes [3+2]- or [4+2]-cycloaddition reactions with furans, azides, and diazo derivatives, or insertion reactions with cyclic ureas, which leads to nonracemic tetracyclic heterocycles **20–23** [610].

Derivative **24** was synthesized from colchicine in 5 steps with the Demyanov rearrangement as a key step. Compound **24** contains a Michael acceptor fragment, which allows it to covalently bind to protein targets [611]. As shown by X-ray diffraction analysis, colchicinoid **24** is effectively integrated into the colchicine site of the tubulin protein. Derivative **24** inhibits the growth of resistant tumors at low nanomolar concentrations and exhibits a synergistic effect, when used in combination with vincristine. Compounds **25–27** were synthesized from derivative **24** in one step. Furanoallocolchicinoids **28** [612] and derivatives **29** containing an *exo*-double

Scheme 9.4.



bond in the D ring demonstrate antiproliferative activity at pico-molar concentrations (Scheme 9.4). Compounds **29** in acidic media tend to isomerize into furans **30** [613].

Based on colchicine, colchicine coumarins **31** [613] were synthesized, as well as derivatives of colchicine and thiocolchicine containing Michael acceptor fragments in the B (compounds **32**) [614] and A rings (compounds **33**) [615].

Various delivery systems were successfully created to enhance the selectivity of colchicine derivatives. A furanoallocolchicine conjugate with chitosan functionalized with rhodamine dye **34** was obtained in 6 steps and used to determine the biodistribution of colchicinoid-containing nanoparticles in organs and tissues in animal models [616]. It was established that chitosan nanoparticles of colchicinoids inhibit tumor growth in vivo more effectively compared to the intact forms of colchicinoids and, at the same time, exhibit no gastro- and cardiotoxicity. Lipid pH-sensitive prodrug conjugate **35** was synthesized from colchicine and solketal in 16 stages [617]. Its liposomal forms effectively release a therapeutic agent structurally similar to compound **29** at a pH of 4.5–5.9, which corresponds to

acidification of the tumor microenvironment, while the liposomal forms of derivative **35** are ~10^5 times less toxic than the intact therapeutic agent. Chiral phospholipid **36** capable of being easily incorporated into therapeutic nanosized natural phosphatidylcholine liposomes (100–110 nm) was synthesized from natural colchicine [618]. It was shown that lipid prodrug conjugate **36** effectively releases therapeutic colchicinoid type **29** on the surface of the liposomal bilayer, under the action of PL-2 phospholipases generated by tumor tissues [618].

Enzymatically Triggered CO-Releasing Molecules. The design and synthesis of a new type of enzymatically triggered CO-releasing molecules (ET-CORMs) (Scheme 9.5) has been developed. In conjugate **37** shown in Scheme 9.5, the iron tricarbonyl fragment (highlighted in red) is separated from the tetrapeptide specific to the tumor serine protease plasmin-specific tetrapeptide (highlighted in blue) by a self-immolative linker (highlighted in blue) [619].

When plasmin interacts with the conjugate, the peptide bond between lysine and the self-immolative linker is broken, which destroys the conjugate, and a cyclic urea and the enol form of the iron cyclohexadiene complex are formed. Having tautomerized to the ketone form, the enol is destroyed, releasing Fe(III) ions and Fe(II) oxide. Carbon monoxide is an endogenous molecule that fulfills signaling functions at low (ppm) concentrations and also exhibits anti-inflammatory, antitumor, antiapoptotic, anticoagulant, and antioxidant properties.

New synthetic aspects in the chemistry of Eschenmoser salts. A cascade procedure for the conversion of substituted styrenes into conjugated enals in reactions with iminium cations in the presence of the $ZnCl_2/LiCl/H_2O$ system (Scheme 9.6) was developed [620]. The procedure was tested for the tolerance to different styrene substrates and different protective groups.

According to DFT calculations, NMR experiments, and deuterium-labeled starting compounds, the reactions proceed via the electrophilic addition and hydride transfer steps involving iminium cations, and, therewith, the $LiCl/ZnCl_2/H_2O$ system activates the iminium electrophile. The key step is the isomerization of the iminium intermediate, presumably via $ZnCl_2$-stabilized azomethine ylides [620].

Chemistry of polymers and biocompatible materials. The copolymerization of alkyl methacrylate and styrene in the presence of radical inhibitors was studied: the Blatter radical terminates one polymer chain (Scheme 9.7A) [621], *p*-quinones terminate two polymer chains (Scheme 9.7B) [622–624]; and 4-alkynylcoumarins are incorporated into the polymer chain similarly to *p*-quinones (Scheme 9.7C) [625].

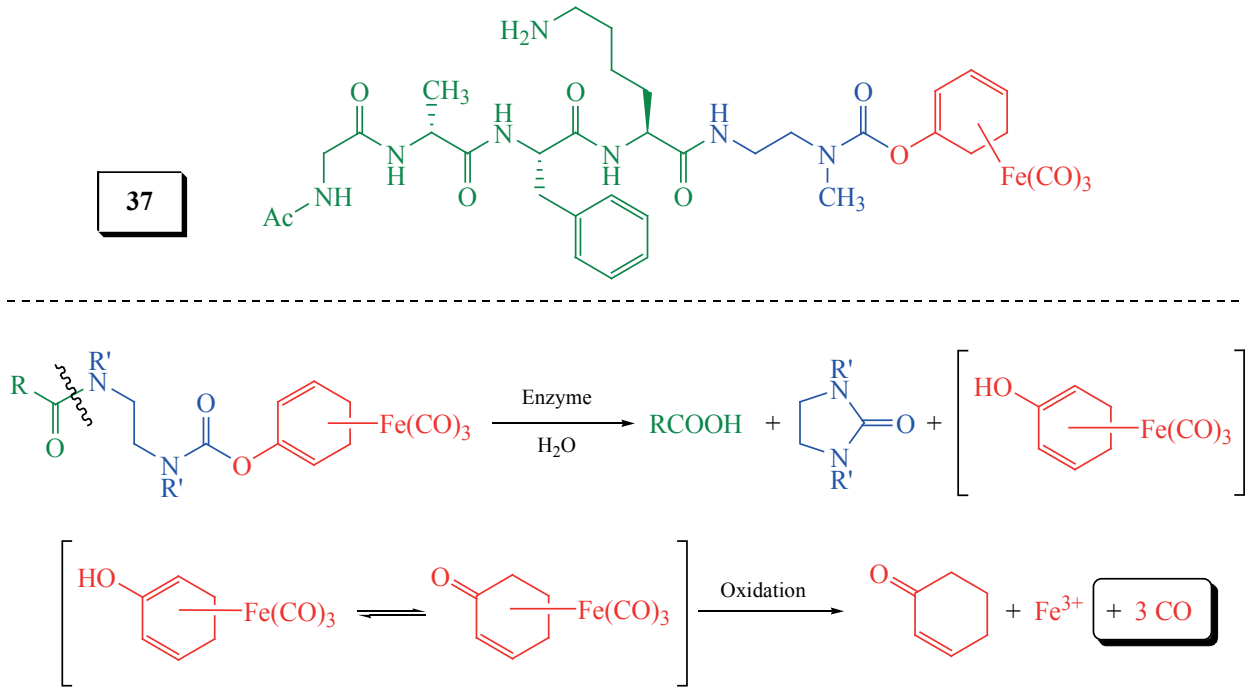
Under polymerization conditions, the macromolecules formed according to Schemes 9.7A and 9.7B can initiate polymerization by the reversible inhibition mechanism, which allows polymerization without preliminary separation of the monomer from the inhibitor. With alkynylcoumarins, no signs of controlled polymerization are observed. Poly(methyl methacrylate), which incorporates covalently bound alkynylcoumarin into its structure, has a green fluorescence persisting in a laboratory sample of organic glass for 3 years.

Biocompatible materials were obtained by grafting synthetic polymers, such as poly(methyl methacrylate) and polyacrylamide, to collagen or gelatin in the presence of boron alkyls [626, 627]. This approach results in the formation of a grafted cross-linked copolymer, which is especially important for the creation of materials for regenerative medicine.

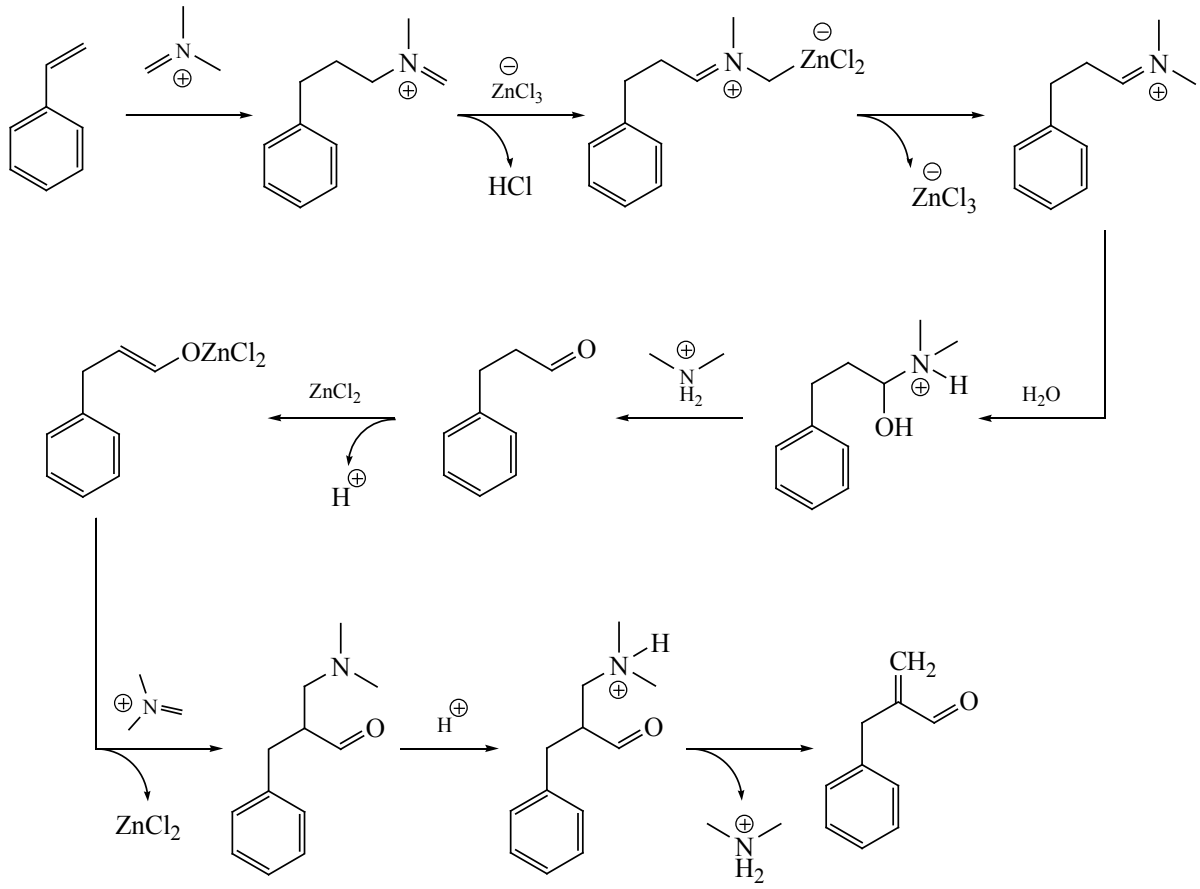
Chemistry of organic compounds of Group V elements. New organoelement compounds of antimony and bismuth containing fragments of unsaturated carboxylic acids were synthesized. These products were used to obtain organometallic copolymers [628–631], which found application in components of UV resistors, organic glasses with special properties (absorption of nuclear and X-ray radiation), and also exhibit bactericidal and fungicidal activity.

The work was financially supported by the Russian Science Foundation (project no. 21-73-10230) and the Research Laboratory of Chemistry of Natural Compounds and Their Synthetic Analogs, created by the state assignment at the Technoplatform 2035 Scientific and Educational Center (project no. FSWR-2024-0002).

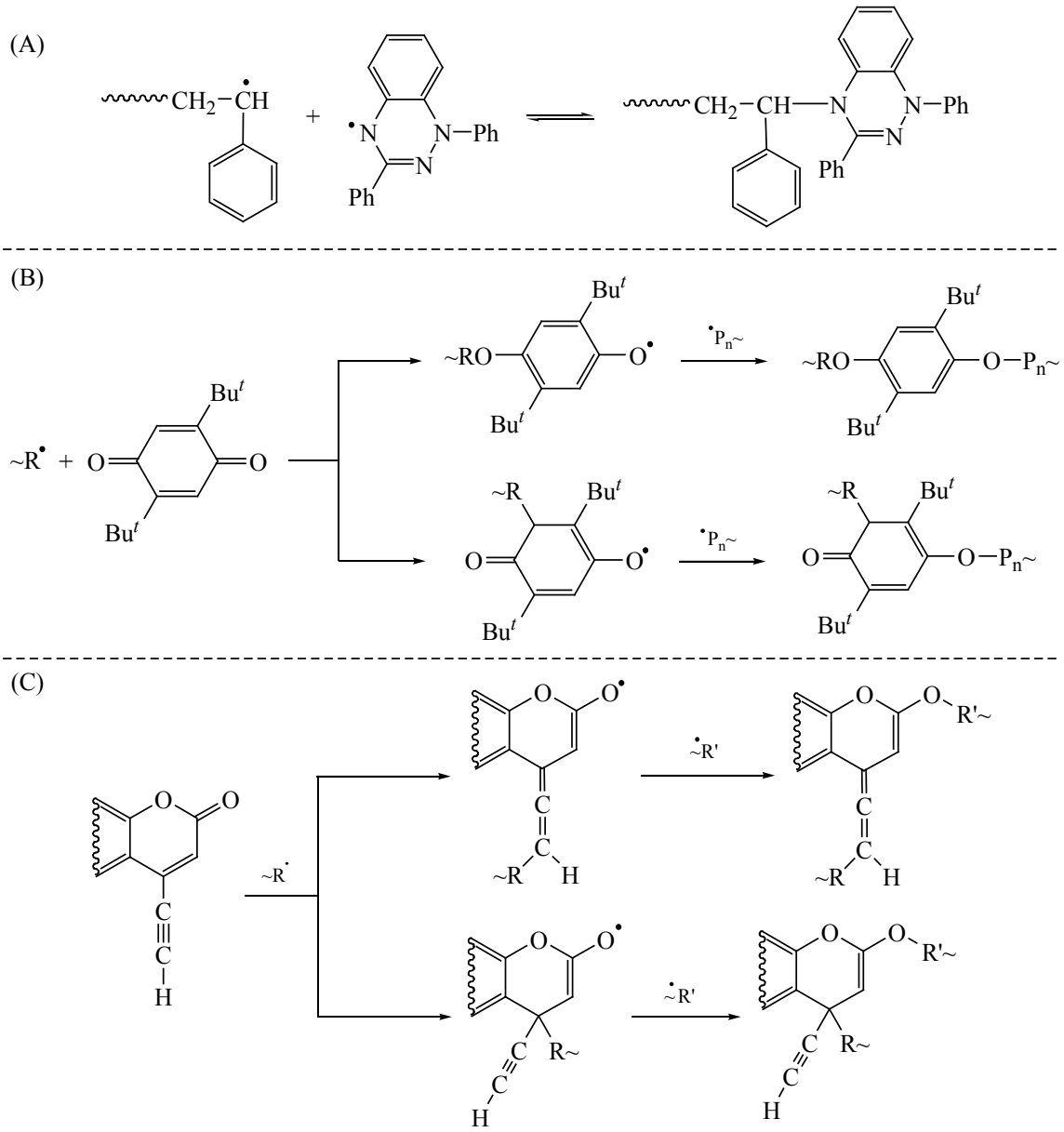
Scheme 9.5.



Scheme 9.6.



Scheme 9.7.



10. DEVELOPMENT OF FUNDAMENTAL APPROACHES TO THE CREATION OF LIBRARIES OF NEW N,O,S-CONTAINING HYBRID HETEROCYCLES AND THEIR FUNCTIONAL DERIVATIVES AT THE DEPARTMENT OF ORGANIC AND BIOORGANIC CHEMISTRY OF CHERNYSHEVSKII SARATOV STATE UNIVERSITY

The department has continued research into the scientific problems of the chemistry of N,O-heterocycles with linearly bound and fused, including pharmacophoric fragments.

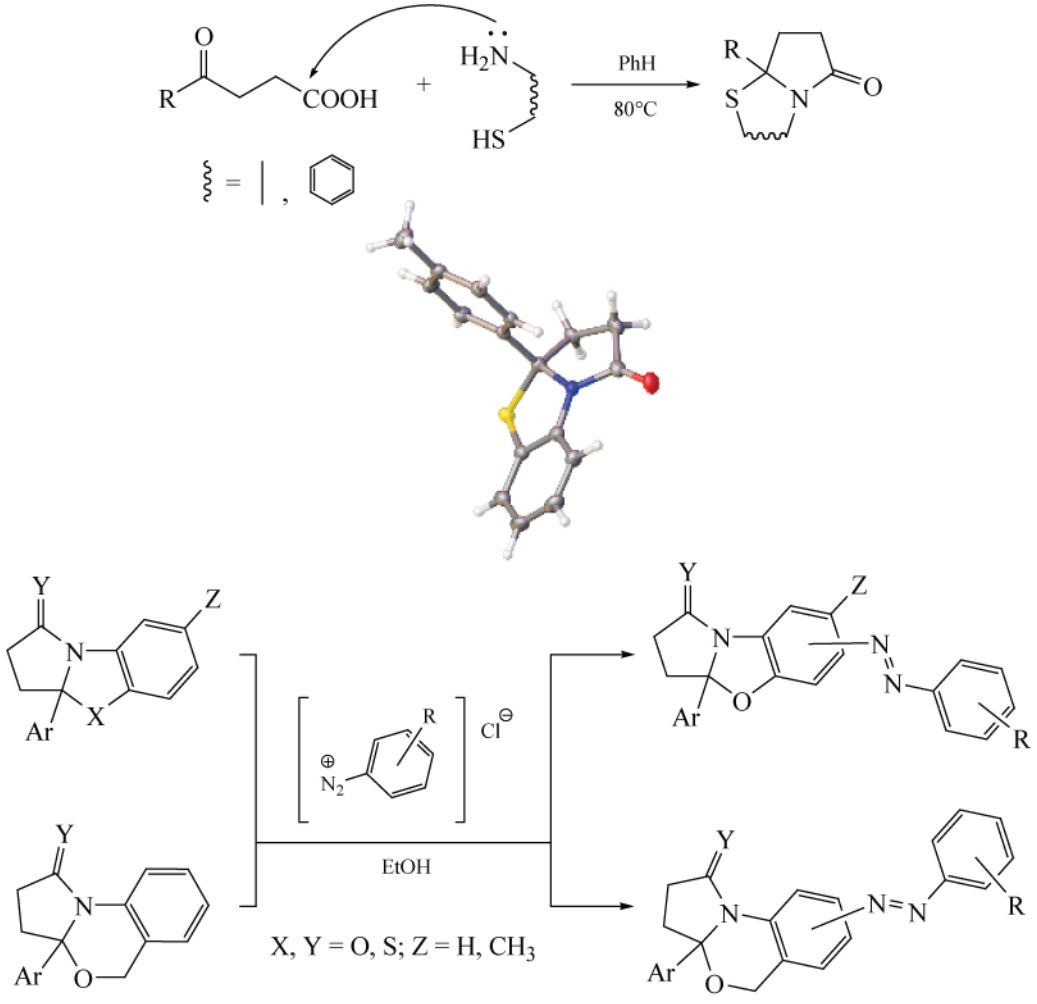
Systematic research based on various dicarbonyl structures allowed the library of heterocyclic compounds to be expanded to include both bi- and tricyclic fused systems with different modes of ring fusion and different combinations of heteroatoms and ring sizes, depending on what dinucleophiles (N,O or N,S) was reacted (Scheme 10.1). The stereochemistry of imidazoimidazole(oxazole)(thi)ones, imidazopyrimidine(oxazine)-(thi)ones, and imidazodiazepine(oxazepine)(thi)ones with different degrees of substitution, their relative reactivity, intramolecular interactions, tautomerism, azo coupling reactions, multicomponent transformations,

and complex formation were studied (**Prof. A.Yu. Egorova and co-workers**). Fundamental data on the reactivity of tetrahydropyrrolooxazolones, tetrahydropyrrolooxazinones, dihydrobenzopyrrolooxazolones, and dihydrobenzopyrrolooxazines toward Lawesson's selective thionating reagent were obtained for the first time under conditions of one-pot synthesis of thione derivatives of the studied heterocyclic systems [632–636].

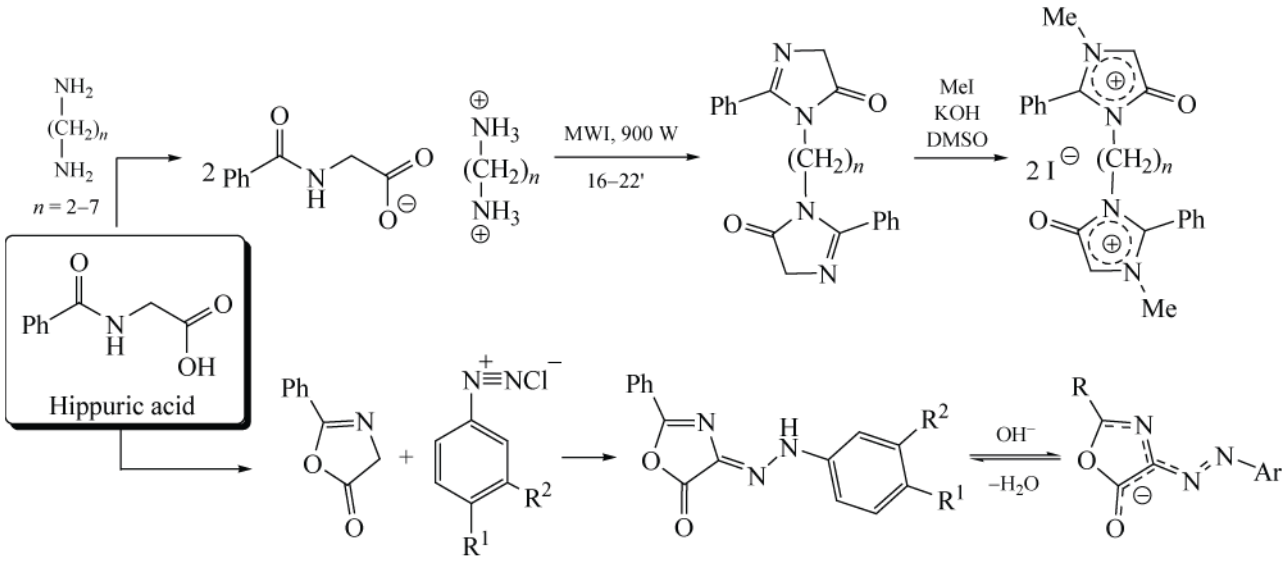
The effect of the synthesis conditions and the nature of the starting reagents on the possibility to implement new directions of heterocyclization in reactions of benzoylglycine with terminal aliphatic diamines was investigated (**Assoc. Prof. V.S. Grinev and co-workers**). It was found that the reactions of hippuric acid with aliphatic diamines gives salts, specifically alkane-1,*N*-diammonium dihippurates, in quantitative yields (Scheme 10.2). Under MW irradiation, bis(phenyldihydroimidazolones) are formed; conformational analysis was performed to establish the contributions of individual molecules. The quaternization of 3,3'-(alkanediyl)bis(2-phenyl-3,5-dihydro-4*H*-imidazol-4-ones) with methyl iodide in the KOH/DMSO system was performed [637–639].

A representative library of oxazol-5(4*H*)-one arylhydrazones was synthesized in order to study their optical properties and biological activity. The X-ray diffraction analysis of some representatives of the series showed that these hydrazones are present in the *Z* form. A method for the targeted creation of diversely formed hybrid systems has been proposed (**Prof. O.V. Fedotova and co-workers**) using multicomponent reactions of "platform systems" of 4-hydroxy-2*H*-chromen-2-one, 3-acetoacetyl-2*H*-chromen-2-one, 1,3-indanedione, 3,4-dihydronaphthalen-1(2*H*)-one and aromatic aldehydes in their combined presence (Scheme 10.3) [640, 641].

Scheme 10.1.



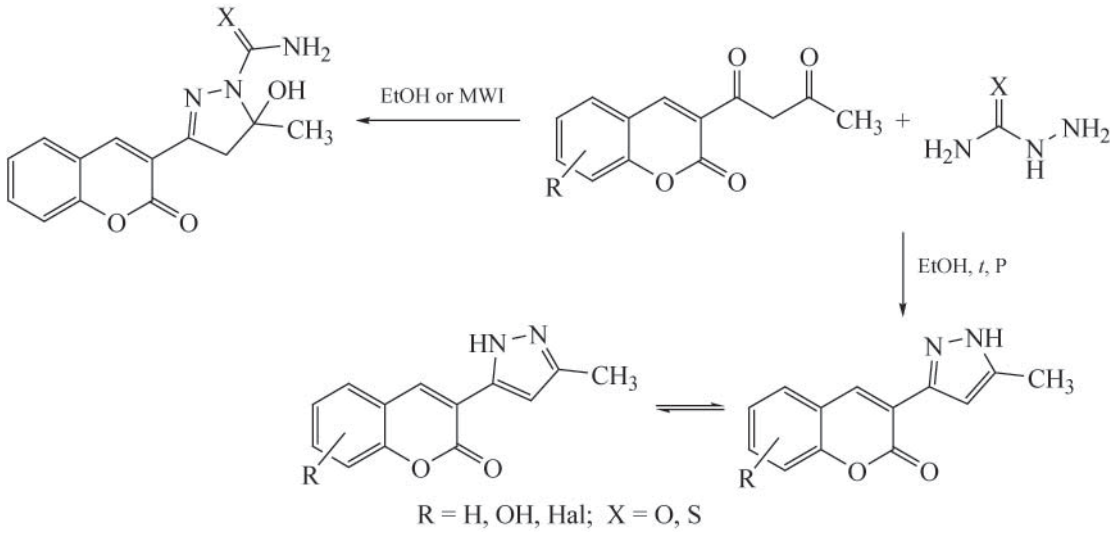
Scheme 10.2.



New data in the chemistry of furanones and their S,N-hetero analogs were obtained (**Prof. A.Yu. Egorova and co-workers**). Optimal conditions of the synthesis of aryl(hetaryl)aminomethylidene-3*H*-furan-2-ones by the one-pot condensation of furanones, orthoester, and aromatic and heteroaromatic amines were found. Thio analogs of these furanones were also obtained. Methods for transforming the studied compounds into new heterocyclic systems were explored (Scheme 10.4) [642–646].

Further impetus was given to research on the application of the cycloaddition reaction in the synthesis of spiro-fused pyrrolidines and pyrrolizidines (**Prof. V.V. Sorokin and co-workers**). Approaches to the regio- and stereoselective synthesis of new representatives of the mentioned series by the 1,3-dipolar

Scheme 10.3.



cycloaddition of azomethine ylides to dipolarophiles, for which alkenes with pronounced electronic effects, were developed (Scheme 10.5). The reactions of benzylidene malononitriles with ylides derived from isatin and proline showed some specifics: they formed a mixture of isomeric pyrrolizidines capable for interconversion by, among others, the retro-Mannich reaction [647]. Three-component reactions of carbonyl compounds, malononitrile, and *N*,*N*-binucleophiles leading to new pyrido[1,2-*a*]pyrimidinecarbonitriles and spiropyrazolinecarbonitriles have been actively studied [648, 649].

Research in the chemistry of chromene, quinoline, quinazoline, and pyrimidine derivatives containing linearly bound and fused fragments has been conducted (**Prof. A.P. Kriven'ko and co-workers**). were used to obtain A series of new 4,8-C-substituted 2-aminochromene(quinoline)-3-carbonitriles were synthesized by two- and three-component condensations of carbonyl compounds with C– and N–nucleophiles (malononitrile, ammonium acetate, and cyanoguanidine) under various conditions, and their reactions involving the amino group (N–acylation), vicinal amino and cyano groups (annulation of the pyrimidine ring), and chromene fragment (O–N recyclization), as well as quaternization (for pyridyl-substituted systems) were studied (Scheme 10.6). Three-component condensations were used to obtain previously unknown thiazolopyrimidinecarboxylates, chromenecarboxylates, and aminoquinolinecarbonitriles, and the latter were used to obtain pyrimidine ring annulation products: pyrimidoquinolinones. The annulation was carried out using graphene oxide as a recycled heterogeneous nanocatalyst [650–652].

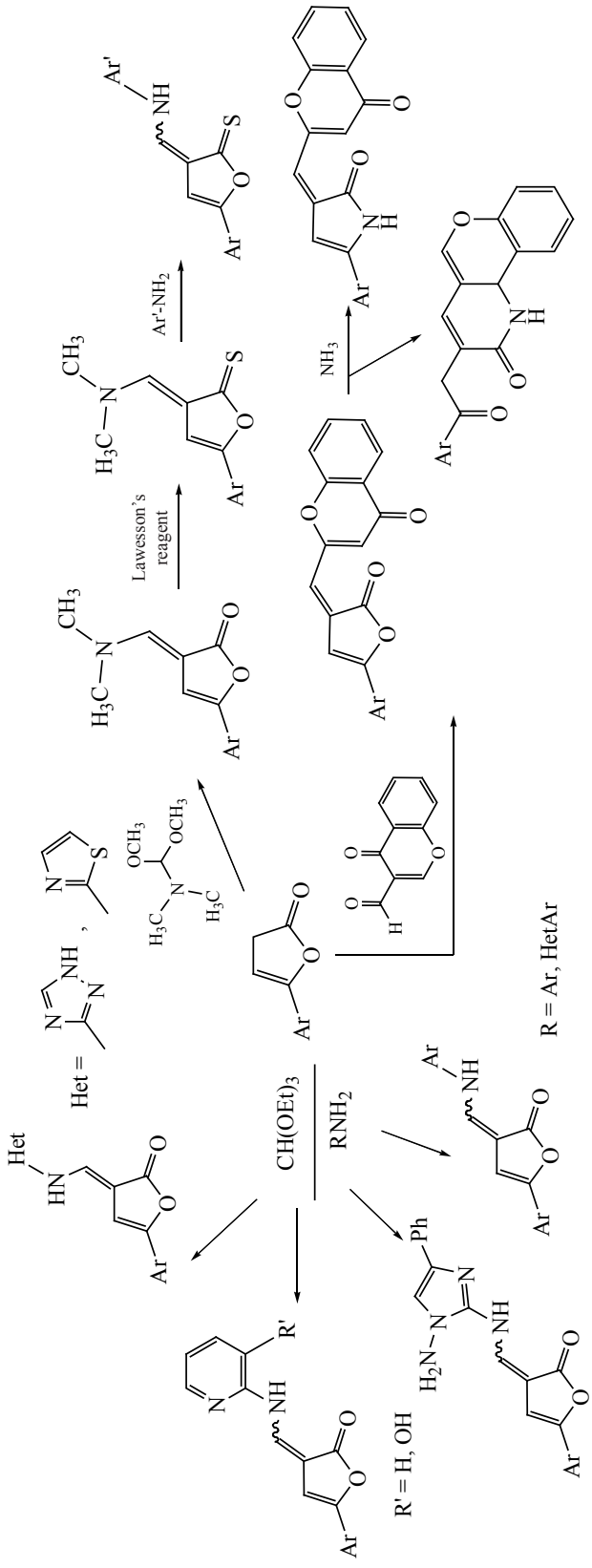
A series of 4,8-aryl(hetaryl)-substituted 2-(cyanmino)octahydroquinazolines was synthesized based on carbonyl compounds and cyanoguanidine. The subsequent oxidation of the synthesized compounds involved selective dehydrogenation of the tetrahydropyrimidine ring transformation of the cyanimino group to form 2-aminocarbamoyl- and 2-nitrosotetrahydroquinazolines (Scheme 10.7).

In collaboration with the Institute of Biochemistry and Physiology of Microorganisms RAS (**Prof. L.Yu. Matora and Prof. S.Yu. Shchegolev and coworkers**), the new compounds are tested for different types of biological activity: antimicrobial activity, cellular respiration inhibition of tumor tissues of various genesis, as well as growth stimulation to plants of agricultural importance [653–659]. The effect of the coexistence of bacteria of the genus *Azospirillum* in purposefully created ectosymbiotic associations with economically important plants in an in vitro culture on the physiological, biochemical, and serological characteristics of reisolates and their ability to colonize and stimulate plant growth.

11. DEPARTMENT OF ORGANIC AND ANALYTICAL CHEMISTRY OF YAROSLAVL STATE TECHNICAL UNIVERSITY

One of the research areas of the department is associated with the synthesis of phenylcycloalkanecarboxylic

Scheme 10.4.



acids and their derivatives. The phenylcycloalkane fragment is synthesized by the alkylation of benzene with cycloalkene-1,2-dicarboxylic acids and their functional derivatives (Scheme 11.1).

It was shown that the stereochemical result of the reaction depends on the order of addition of the reagents. On this basis, methods for the synthesis of phenylcycloalkanedicarboxylic acids and their separation to obtain the target diastereomer were developed. A mechanism of this phenomenon was proposed [660].

A series of carboxylic acid amides containing imide, cyclohexene, and norbornene rings and fragments of natural amino acids was synthesized. The synthesis conditions were optimized to obtain high yields of the target products. The results of biotesting allowed a number of the synthesized compounds to be recommended for testing as potential plant growth regulators [661].

New optically active, heat-resistant polyamideimides soluble in different organic solvents were obtained starting from 4-nitrophenylcycloalkane-1,2-dicarboxylic and natural aminocarboxylic acids [662].

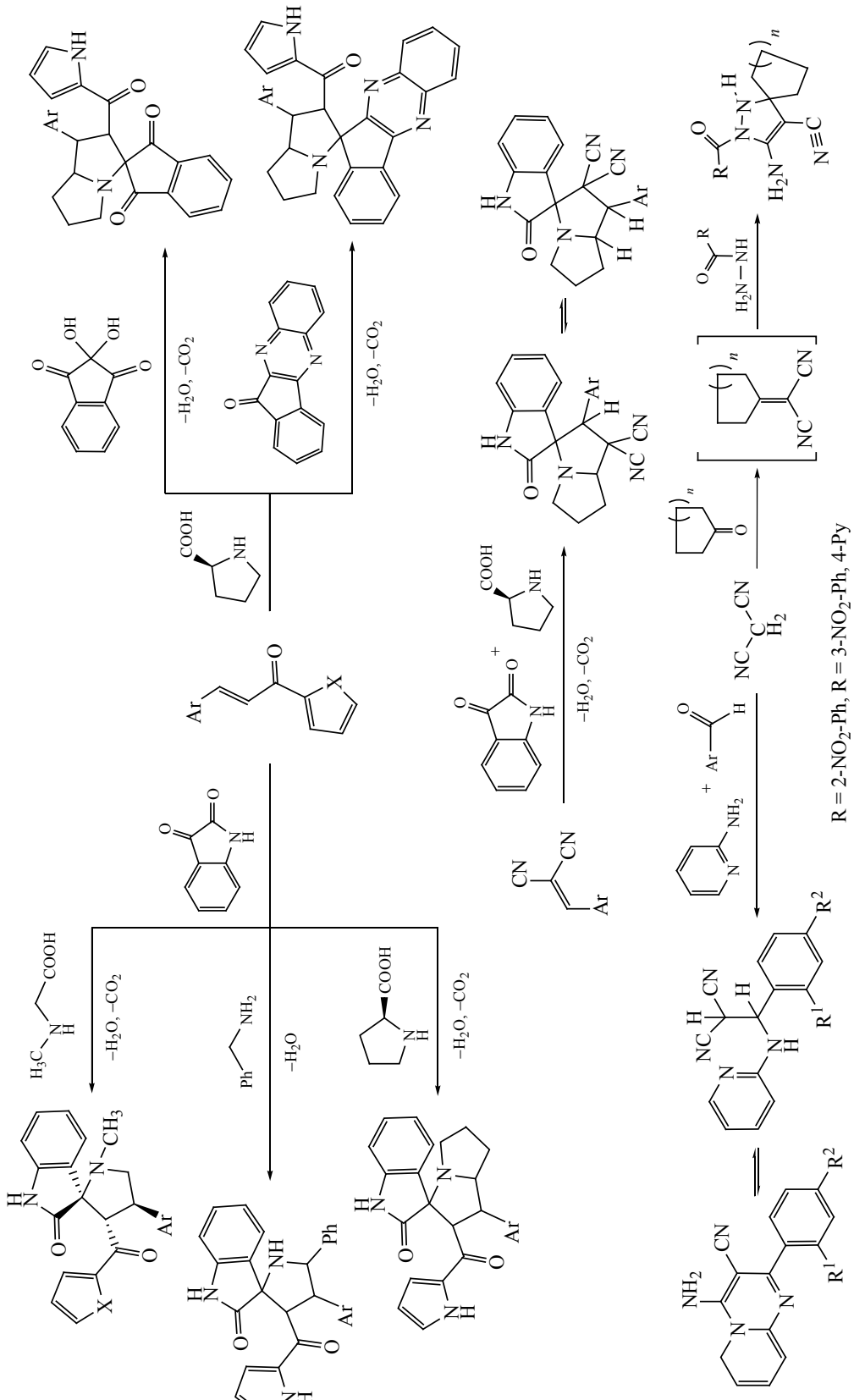
Methods for the synthesis of functional derivatives of phenylcycloalkanepolycarboxylic acids were developed [663, 664, 665].

A quantum-chemical study of the Friedel–Crafts alkylation of benzene with cyclohex-4-ene-1,2-dicarboxylic acid was performed. The results of the quantum-chemical study and experiment show that the alkylation of benzene with cyclohex-4-ene-1,2-dicarboxylic acid followed by the functionalization of the product are regio- and stereoselective reactions [666].

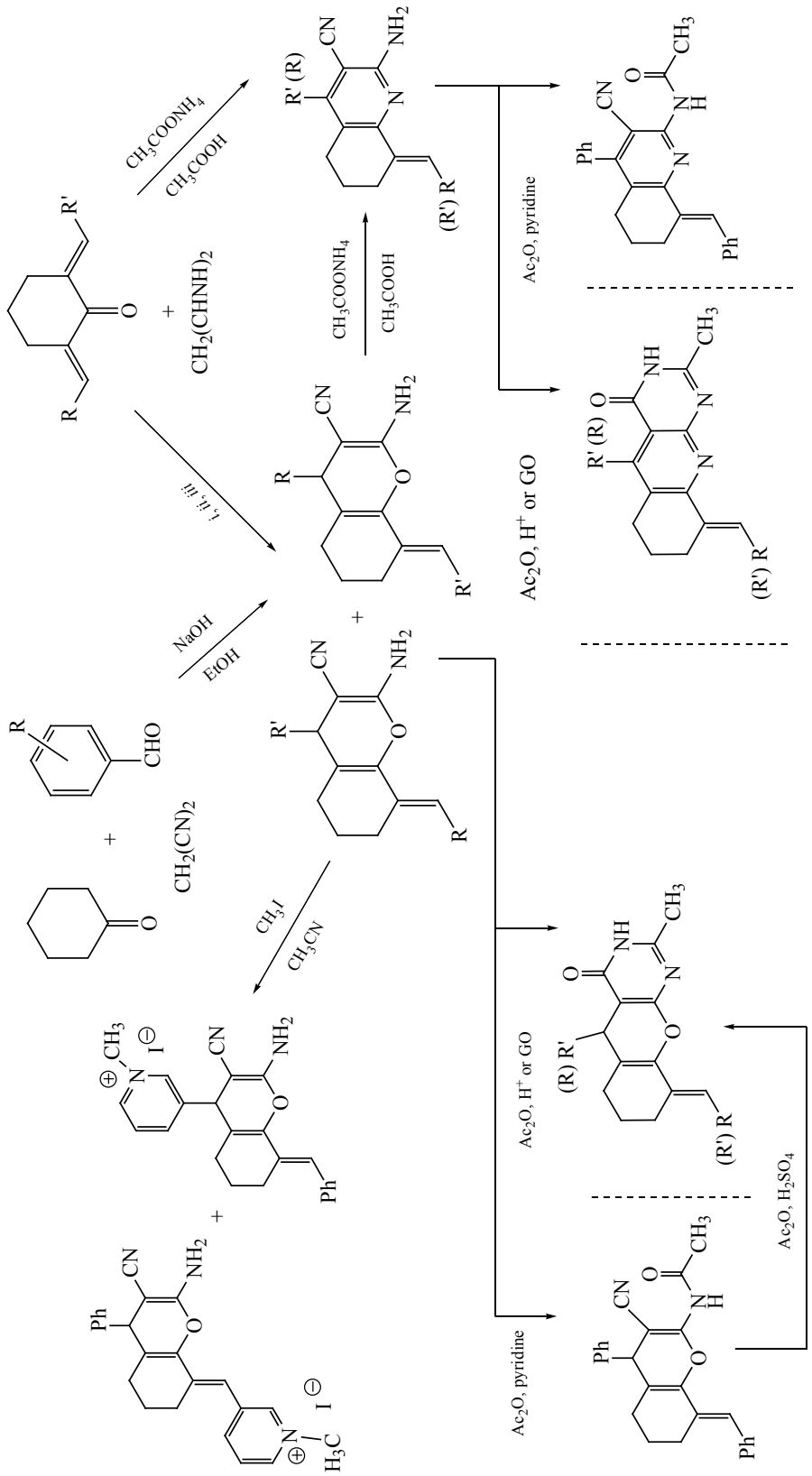
Another line of research is the synthesis and functionalization of 3,5-disubstituted-1,2,4-oxadiazoles. As the key reaction of the synthesis, the reaction of amidoximes with carboxylic acid derivatives was chosen (Scheme 11.2).

An efficient one-pot method for the synthesis of 3,5-disubstituted-1,2,4-oxadiazoles by the reactions of amidoximes with carboxylic acid anhydrides or chlorides, catalyzed by the NaOH/DMSO system, was developed [667]. It was shown that the reaction proceeds through the formation of *O*-acylamidoximes, which was confirmed experimentally and by quantum-chemical calculations [668].

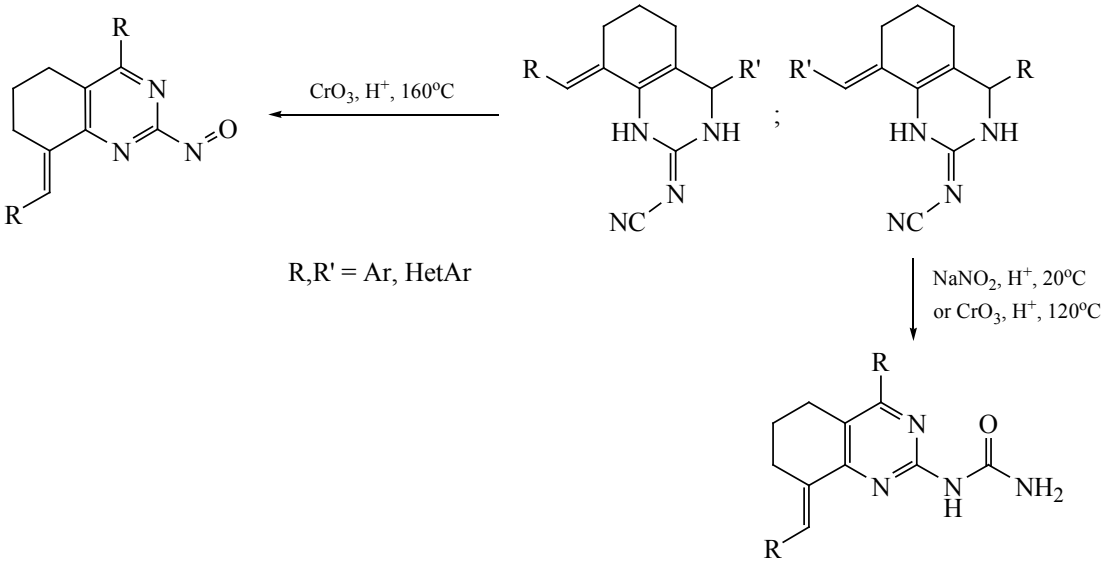
Scheme 10.5.



Scheme 10.6.



Scheme 10.7.



3,5-Disubstituted-1,2,4-oxadiazoles were synthesized by the reaction of amidoximes with carboxylic acids or their esters under high pressure (10 kbar) and in the absence of other reagents or catalysts. Both aliphatic and aromatic carboxylic acids enter into the reaction. The reaction is complicated by the concurrent condensation of two amidoxime molecules to form 3,5-bis-1,2,4-oxadiazole [669].

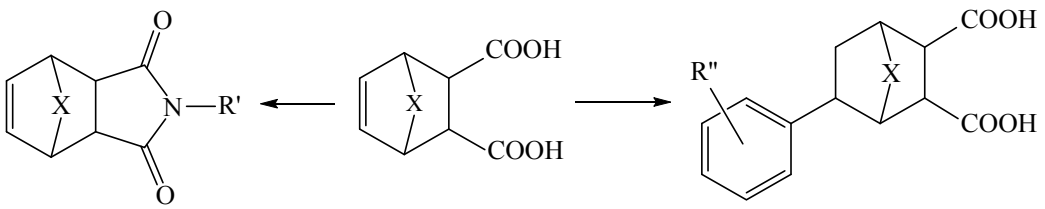
5-Alkenyl- and 5-styryl-1,2,4-oxadiazoles were prepared by the cyclodehydration of *O*-acylamidoximes at room temperature in the superbasic KOH/DMSO medium. A one-pot synthetic approach to a wide range of related structures was developed. A series of new compounds were obtained that showed moderate antimictobial activity against *E. coli* and *S. aureus* bacterial and *C. albicans* fungal strains [670–672].

3,5-Disubstituted 1,2,4-oxadiazoles containing an alkenyl fragment were used as the starting materials to synthesize *N*-phthalimidoaziridines with pyridine and 1,2,4-oxadiazole rings. Thermolysis of these compounds was carried out in the presence of dipolarophiles, and a number of new representatives of the *N*-phthalimidoaziridine and *N*-phthalimidopyrrolidine series were synthesized. It is worth noting that the newly synthesized compounds contain a few pharmacophoric fragments (aziridine, oxadiazole, pyridine, and phthalimide) [673].

A promising area of research at the department is the chemistry of thiopyrans. Having developed an efficient one-pot synthesis (thionation, hetero Diels–Alder reaction) of 3,4-dihydro-2*H*-thiopyran derivatives [674, 675], we began a systematic study of their synthetic potential. The main results of this study are presented in Scheme 11.3.

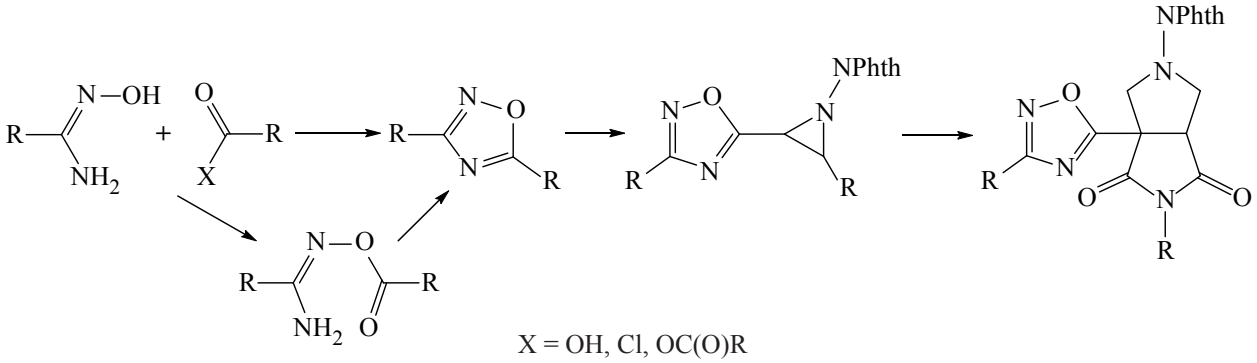
It was found that the reaction of α,β-unsaturated ketones and Lawesson's reagent with maleic, itaconic, or 5-norbornene-2,3-dicarboxylic acids leads to the corresponding 3,4-dihydro-2*H*-thiopyran anhydrides.

Scheme 11.1.



X = none, CH_2
R' = Alk, CycloAlk, AlkC(O)COOH; R" = NO_2, NH_2, COOH

Scheme 11.2.

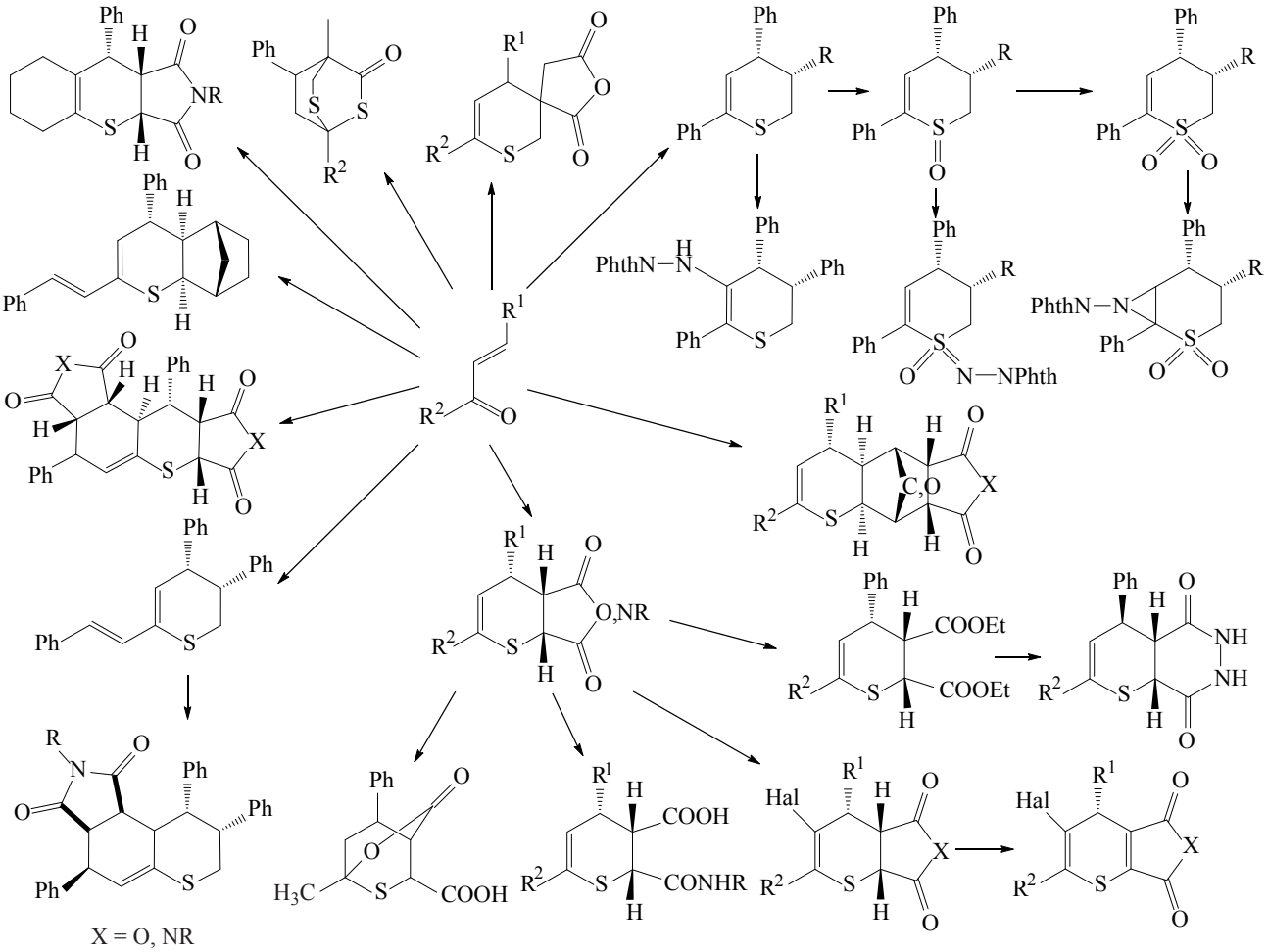


X = OH, Cl, OC(O)R
R = Alk, Alken, Ar, Het

The reaction of methacrylic acid, Lawesson's reagent, and chalcones gives 4-methyl-1,8-diphenyl-2,6-dithiabicyclo[2.2.2]octan-3-one. The selectivity of the acylation of amines with the synthesized anhydrides was studied [676].

Merkulova et al. [677] reported the results of a study of the halogenation of 3,4-dihydro-2*H*-thiopyrans. It was found that the bromination of 3,4-dihydro-2*H*-thiopyran derivatives forms bromo-substituted 3,4-dihydro-2*H*-thiopyrans or 4*H*-thiopyrans, depending on the reaction

Scheme 11.3.



X = O, NR

conditions and the structure of the starting heterocycles.

The behavior of 3,4-dihydro-2*H*-thiopyrans and their *S*-oxides and *S*,*S*-dioxides in oxidative phthalimidoaziridination reactions was studied to find out that the reactions provide products of the enamine, sulfoxyimine, and aziridine structures, respectively [678].

A facile synthesis of new polycyclic thiopyrans by a three-step domino reaction of dibenzylideneacetone with maleic acid derivatives, involving thionation and heteroaddition (HAD) and cycloaddition (CAD) Diels–Alder reactions was developed. It was established that the reactions with styrene and norbornene as dienophiles result in the exclusive formation of HDA reaction products capable of reacting with maleic acid derivatives under more severe conditions [679].

A logical continuation of this series of works was the extend the one-pot approach to the synthesis of 2*H*-thiopyrans from β-aminoenones [680].

12. DEPARTMENT OF ORGANIC AND BOOMOLECULAR CHEMISTRY OF URAL FEDERAL UNIVERSITY. MODERN SYNTHETIC APPROACHES TO AZAHETEROCYCLES. STUDY OF APPLIED PROPERTIES

The department is one of the oldest ones created in the Soviet period. In 2014, the department celebrated its 90th anniversary, and since 2015 it has been called the Department of Organic and Biomolecular Chemistry. Today, the department's faculty members includes 2 academicians of RAS, 1 corresponding member of RAS, 1 professor of RAS, 4 professors, and 10 associate professors.

The department traditionally focuses on research in heterocyclic chemistry. The works deal with such basic issues as the reactivity, structure, and properties of heterocycles, and also with the potential applications of heterocycles, for example, in the field of biomolecular chemistry, via searching for structure–biological activity relationships, and also in the field of materials chemistry, via studying the complexing and sensory properties of the synthesized compounds in terms of new drug design, as well as the luminescent characteristics of the products. Along with the mentioned areas, new lines of research, associated with mechanochemical, photochemical, and other methods of green chemistry, are being developed.

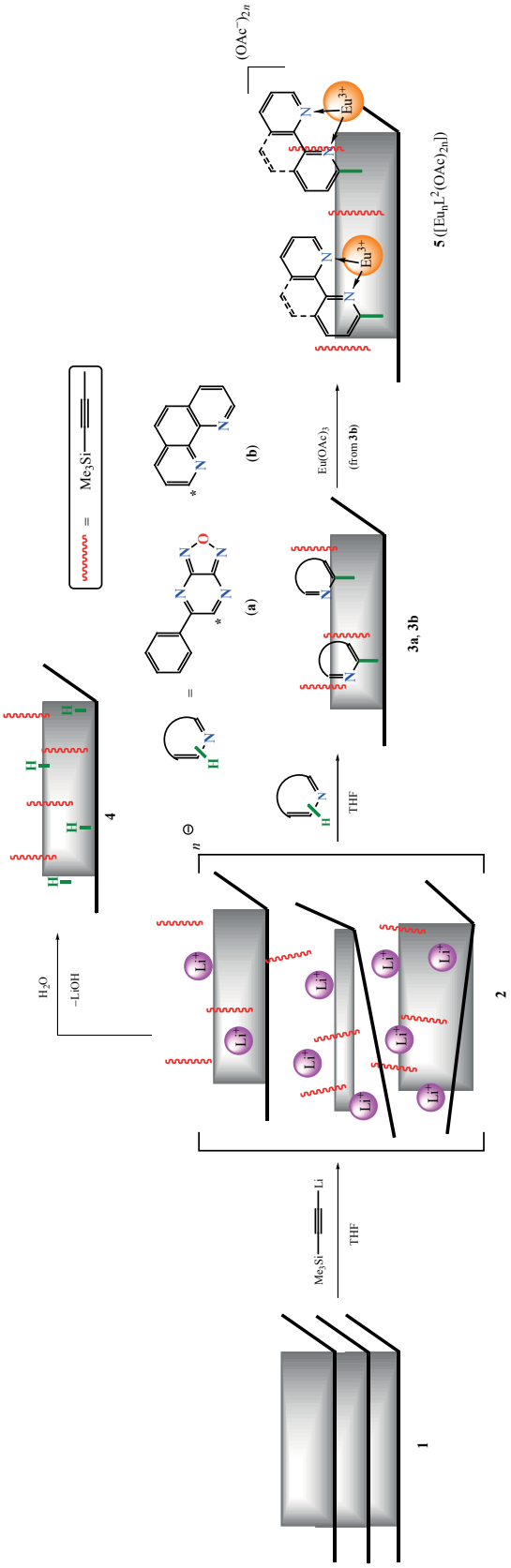
Among green methods, the priority is given to direct C–H functionalization (S_N^H reactions) of heterocycles, initiated by Academician O.N. Chupakhin and further developed by Academician V.N. Charushin, Corresponding Member V.L. Rusinov, Professors M.V. Varaksin, G.V. Zyryanov, and I.A. Utepova and other employees. Recently, there works have acquired an obvious practical focus.

Thus, for the first time, graphene was modified with fragments of aza-heterocyclic ligands by a two-step procedure involving the reductive alkynylation of commercially available three-layer graphene **1** (few-layer graphene, FLG) with (trimethylsilyl)ethynyl lithium and the subsequent reaction of the resulting graphenide **2** with phenanthridine or [1,2,5]oxadiazolo[3,4-*b*]pyrazine to obtain azinyl-modified graphenes **3a** and **3b** (Scheme 12.1). It should also be noted that the reaction of graphenide **2** with water gave ethynyl-substituted graphene **4**, and ligand **3b** (L2) reacted with $Eu(OAc)_3$ to form complex $[Eu_nL^2(OAc)_{2n}]$ **5**, whose structure was confirmed by X-ray photoelectron spectroscopy [681].

An approach to the direct C(sp^2)–H functionalization of phenanthridine with a 1*H*-benzotriazole residue was proposed, carried out in the presence of Selectfluor reagent and leading to the formation of a new C(sp^2)–N bond (Scheme 12.2). The approach involves a one-step synthesis of α-(1*H*-benzotriazol-1-yl)phenanthridine **6**, which is a valuable synthon for further chemical transformations [682].

A method of the direct C–H functionalization of 8-azapurines **7** with C-nucleophiles (Scheme 12.3) to form 4-heteroaryl-substituted 2-aryl-2*H*-benzo[4,5]-imidazo[1,2-*a*][1,2,3]triazolo[4,5-*e*]pyrimidine fluorophores **8** with planarized intramolecular charge-transfer (PLICT) state characteristics. Fluorophores **8** all showed high luminescence quantum yields (up to 60%) and large Stokes shifts (up to 7459 cm^{-1}). In addition, compounds **8** were found to exhibit a positive solvatochromic effect, as well as pronounced aggregation-induced emission (AIE) properties. This behavior was further confirmed by time-resolved fluorescence lifetime measurements and DFT geometry optimization. One of the compounds **8** demonstrated a well-defined acidochromism toward trifluoroacetic acid via a visible color change of its solution from yellow green to orange, which returned to the original yellow-

Scheme 12.1.



green color upon addition of triethylamine (TEA), as well as photoluminescence quenching with a Stern–Volmer constant of 38 M^{-1} [683].

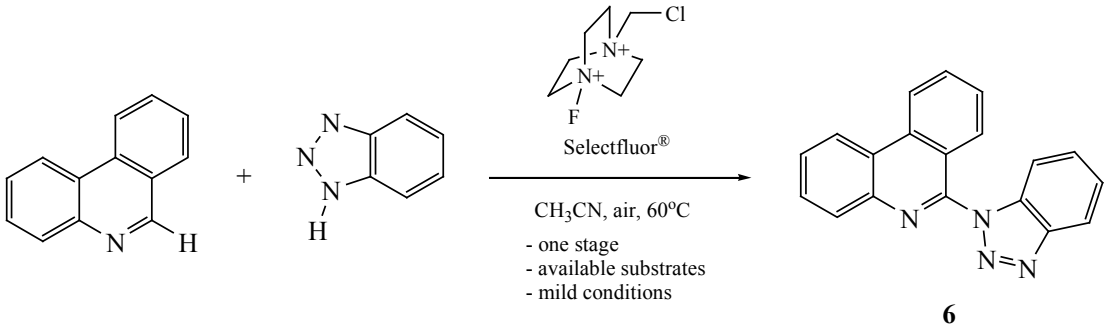
Along with aromatic azaheterocycles, the direct C–H functionalization reaction was extended to a series of non-aromatic systems. Thus, a procedure was developed of the direct cross-dehydrogenative C–H/N–H coupling in a series of cyclic 2*H*-imidazole-1-oxides **9** in the presence of the I_2–*tert*-butyl hydroperoxide (TBHP) reagent system to form heterocyclic derivatives **10** in yields of up to 97% (Scheme 12.4). The radical nature of the process was confirmed by EPR experiments. The reaction involves the N-iodination of the cyclic amine with the homolysis of the N–I bond in the resulting intermediate, followed by the amination of the nitrone fragment via a nitrogen-centered radical [684].

A convenient synthetic approach to previously unknown phosphonium salts **11** containing a 2*H*-imidazole fragment was developed. The approach is based on the direct nucleophilic C(sp^2)–H functionalization of 2*H*-imidazole-1-oxides **9**, where triphenylphosphine acts a nucleophile and the N-oxide fragment of the heterocyclic substrate, as an auxiliary group, whose elimination during reaction facilitates hydrogen cleavage from the C–H bond (Scheme 12.4) [685].

The synthetic strategy based on reactions of 2*H*-imidazole-1-oxides **9** with thiophenols in the absence of acetyl chloride was successfully applied as a convenient tool for the preparation of a number of new azaheterocyclic molecules, including water-soluble hydrochloride forms. The nucleophilic substitution of hydrogen in nonaromatic azaheterocyclic substrates by the addition–elimination (S_N^H AE) mechanism under the optimized conditions gave a series of arylthiolated 2*H*-imidazoles **12** in yields up to 90% (Scheme 12.4). The developed synthetic scheme opens a way to the preparation of azaheterocyclic molecular systems of interest for medicinal chemistry and materials science [686].

The nucleophilic hydrogen substitution (S_N^H) strategy was first applied to the metal-free C–H/C–H coupling reactions of 4*H*-imidazole 3-oxides **13** with indoles. As a result, a series of new bifunctional azaheterocyclic derivatives **14** were obtained in yields of up to 95% (Scheme 12.5). The developed compounds may be of particular interest for medicinal chemistry, especially in the targeted design of candidate small molecules as drugs against neurodegenerative diseases.

Scheme 12.2.



6

To this end, in silico molecular docking experiments were performed to assess the possibility of binding of the synthesized small azaheterocyclic molecules **14** to selected biotargets (BACE1, BChE, CK1δ, AChE) associated with the pathogenesis of neurodegenerative diseases [687].

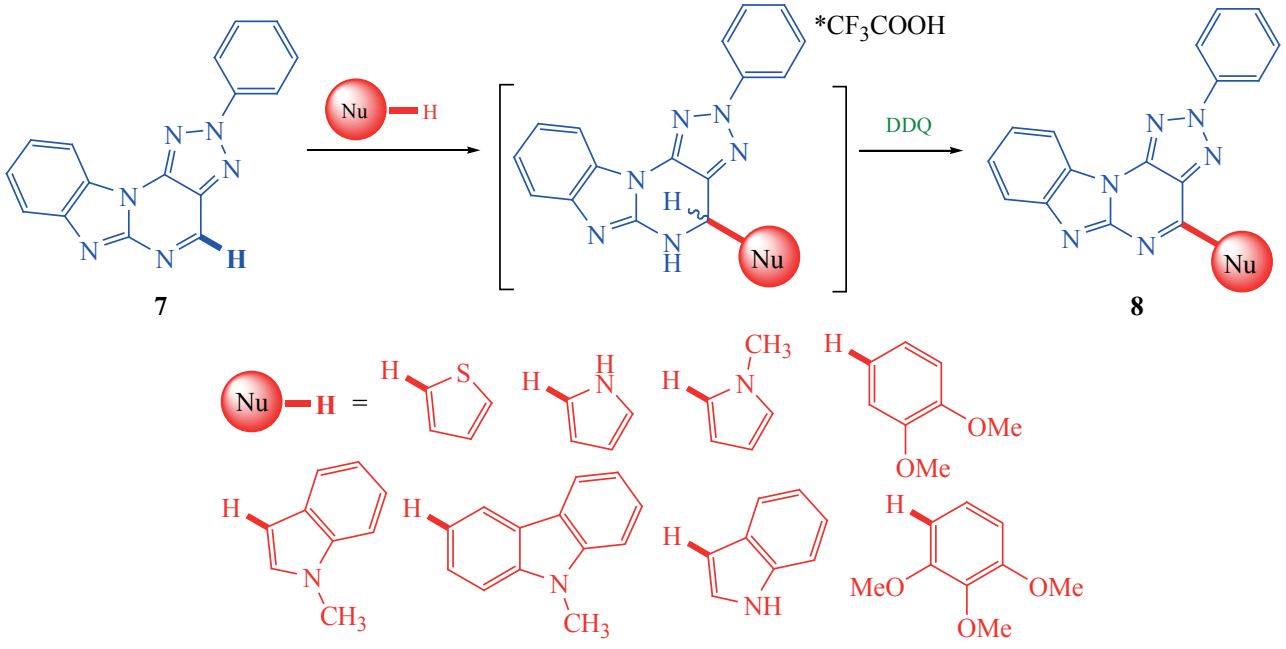
The line of research focused on the *ipso*-substitution of the cyano group in the series of 5-cyano-1,2,4-triazines **15** prepared by S_N^H reactions from 1,2,4-triazine-4-oxides deserves separate mentioning. Thus, under the action of N-nucleophiles in the absence of a solvent, *ipso*-amination products 5-amino-1,2,4-triazines **16** were obtained and then transformed into α-aminopyridines **17** via the aza-Diels–Alder reactions with 2,5-norbornadiene (Scheme 12.6) [688–691]. It is interesting that the reactions with 2-aminooxazoles gave no *ipso*-amination products, and the aza-Diels–Alder reaction was the only reaction pathway [692–695]; this reaction was used as a convenient method of synthesis of **18** and **19**, as well as 2,2'-bipyridines **20** as analogs of natural antibiotics [696] and promising fluorophores [697].

12.1. Photoactivated reactions

In terms of atom economy and green chemistry, photoactivated processes are the most promising for synthetic organic chemistry and materials chemistry. The Department of Organic and Biomolecular Chemistry is also involved in research on such processes.

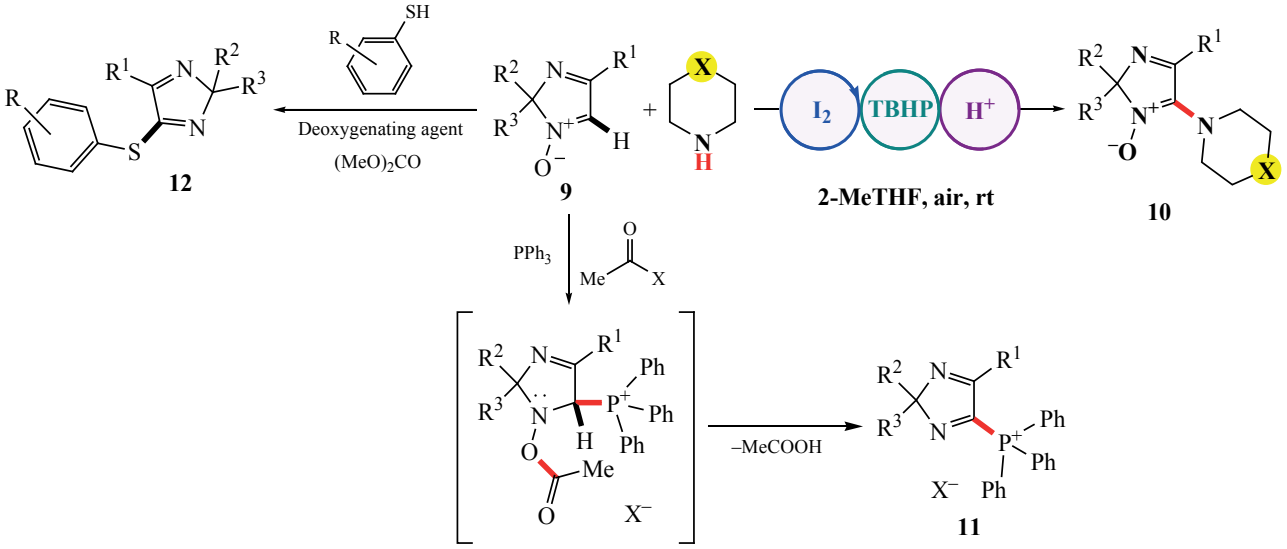
Thus, the oxidative C–H/C–H coupling of azines with dipyrrylmethanes **21** was performed in aerobic conditions under photoirradiation (λ = 425 nm) in the presence of the TiO_2 photocatalyst and atmospheric

Scheme 12.3.



7 **8**

Scheme 12.4.



oxygen was accomplished (Scheme 12.7). These reactions formed mono- or disubstituted products **22**, depending on the nature of the substrate and reaction conditions [698].

The regioselective O-acylation of the C(sp^3)–H position in a small azaheterocycle, aryl-2*H*-azirine with (diacetoxy)iodobenzene, promoted by visible light (blue LED, 34 W) and in the presence of the Rose Bengal photoredox catalyst was accomplished for the first time. The convenience of the reaction is that it occurs under aerobic conditions at room temperature. The transformation occurs by a radical mechanism, which was confirmed by experiments with free radical scavengers, as well as in the dark. The protocol is also scalable to the gram level (Scheme 12.8) [699].

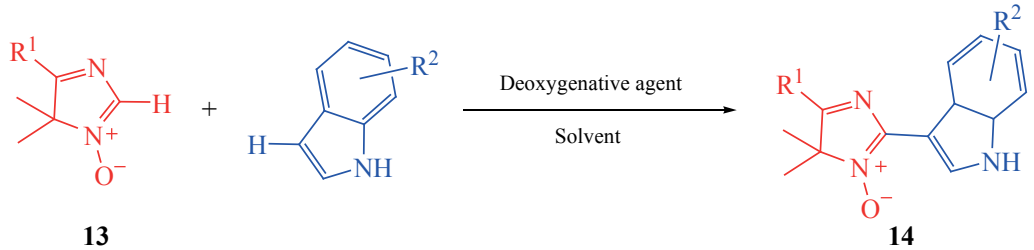
An efficient protocol for the synthesis of 2-phenacyl-substituted tetrahydrofurans or α-hydroxyalkyl ketones **27** from ethers by the reactions of synthons **26** with vinyl azides under photoirradiation in the presence of Rose Bengal, a radical initiator, and *tert*-butyl hydroperoxide, was developed. The process is initiated by radical addition to the vinyl azide, as demonstrated by control experiments, which then triggers a cascade fragmentation mechanism due to the loss of a nitrogen molecule, and the stabilized radical ultimately yields α-hydroxyalkyl ketones (Scheme 12.9) [700].

The reactions of 1,4-quinones with vinyl azide under similar conditions resulted in the formation of 1-oxa-4-azaspirooxazolines in yields of up to 92%. The reaction involves the spiroannulation of vinyl azide at the C=O double bond rather than at the C=C double bond to form target products **28** (Scheme 12.10) [701].

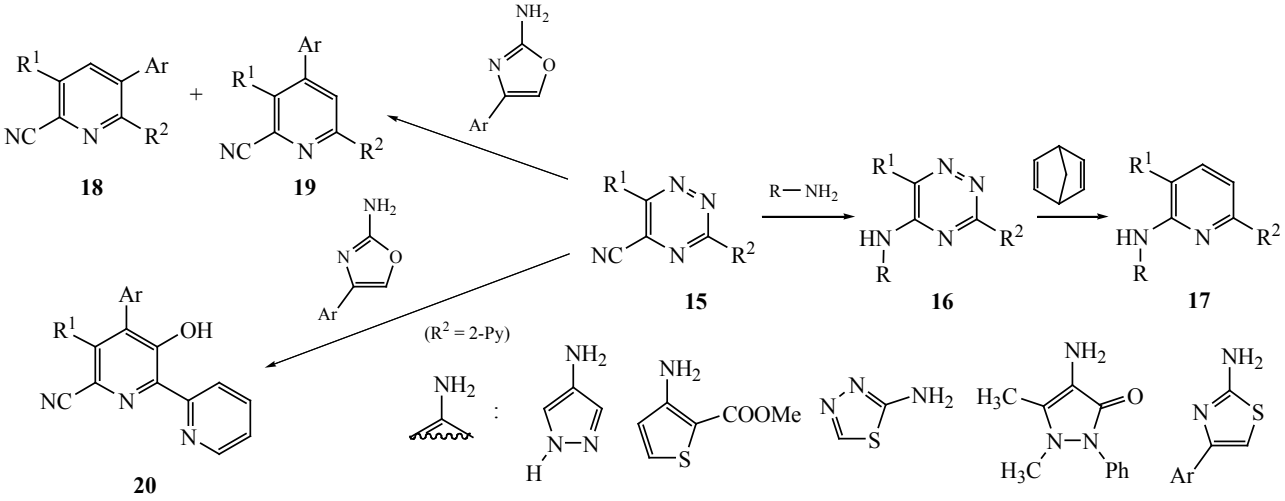
12.2. Photoactive azaheterocycles: luminophores and chemosensors

A convenient synthetic approach to promising fluorophores based on N(2)-fluoroaryl-1,2,3-triazole **29**, which are considered as valuable functional blocks for the design of organic materials emitting blue light, was developed. The protocol includes five synthetic steps with yields of 60–99% at each. It was established

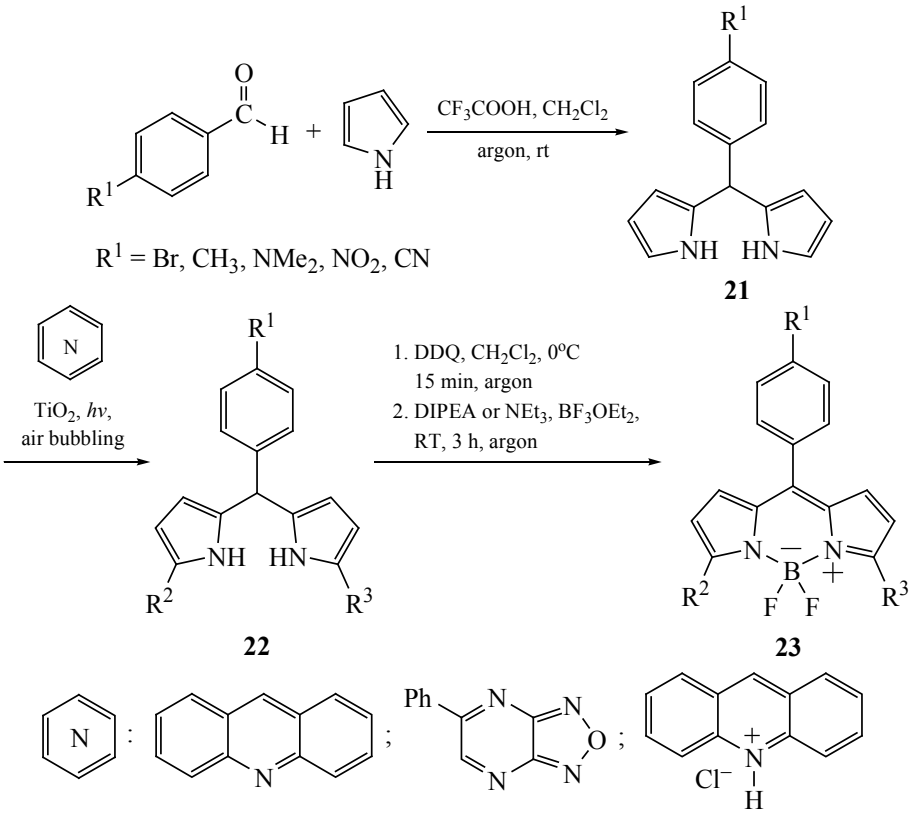
Scheme 12.5.



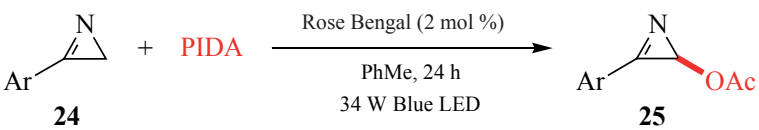
Scheme 12.6.



Scheme 12.7.



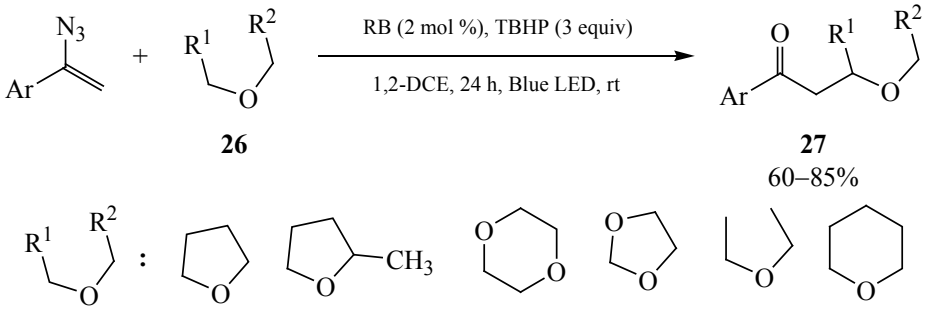
Scheme 12.8.



58–85%

24, **25**, Ar = Ph, *o*-Tol, *m*-Tol, *p*-Tol, 2,4-$Me_2C_6H_3$, 2,4,6-$Me_3C_6H_2$, 4-*t*Bu-C_6H_4, 2-ClC_6H_4, 3-ClC_6H_4, 4-ClC_6H_4, 2,6-$Cl_2C_6H_3$, 3-BrC_6H_4, 4-BrC_6H_4, 3-FC_6H_4, 4-$CF_3C_6H_4$, 4-PhC_6H_4, 1-naphthyl, 2-naphthyl

Scheme 12.9.



26 **27**

60–85%

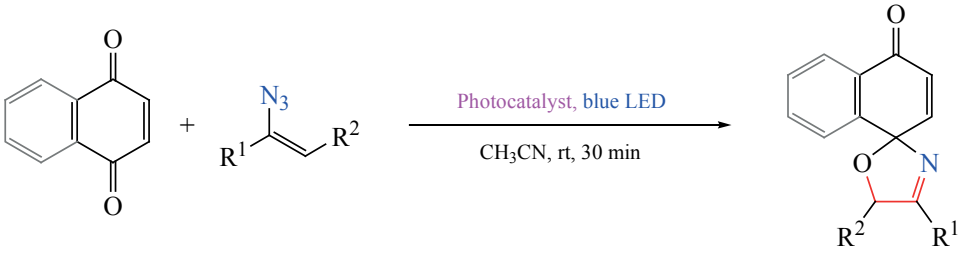
27, Ar = Ph, *m*-Tol, *p*-Tol, 4-FC_6H_4, 3-ClC_6H_4, 4-ClC_6H_4, 3-BrC_6H_4, 4-BrC_6H_4, 4-$CF_3C_6H_4$, 4-PhC_6H_4, 2-naphthyl

that the synthesized photoactive 2-fluoroaryl-5-aryl-4-methyl-1,2,3-triazoles **29** emit blue-violet light at wavelengths of 330–440 nm with an absolute quantum yield of > 99% in solvents of different polarities. Density functional theory and CIS calculations were used to study the features of the mechanism of electron transfer in the excited states of N(2)-fluoroaryl-1,2,3-triazoles **29** in comparison with the respective data for their 1,2,3-triazole 1-oxide analogs (Scheme 12.11) [702].

Studies in the field of push–pull fluorophores of the quinazoline and quinoxaline series were performed. A series of D–p–A chromophores based on 2-aryl/thienyl-4-(morpholin-4-yl)quinazolines, 2-aryl/thienyl-4-cyanoquinazolines **30**, or quinazolin-4(3*H*)-ones **31** were obtained. An analysis of the effect of the nature of the donor and acceptor fragments, as well as the nature and length of the p-spacer on the photophysical properties of the synthesized chromophores was carried out. It was found that 4-(morpholin-4-yl)quinazolines **30** exhibit halochromic properties, when trifluoroacetic acid is added to their toluene solutions [703, 704]. Derivatives of 3-aryl-[1,2,4]triazolo[4,3-*c*]quinazoline **32** containing an amino-[1,1']-biphenyl fragment in position 5 of the heterocyclic ring were synthesized (Scheme 12.12), and the photophysical properties of the resulting fluorophores were analyzed [705].

2,4-Diarylquinazoline D-p-A fluorophores **33** and their 2-azinyl analogs **34** were developed. The effect of the nature of the donor fragment and the substituent in position 2 of the quinazoline core on the position of the absorption and emission maxima and the quantum yield was studied. Spectral changes upon the addition of metal cations to solutions of some of quinazolines **33** and **34**, as well as their solvatochromic and nonlinear optical properties were noticed [706, 707]. Polycyclic compounds **35** were synthesized by the Rh(III)-catalyzed annulation of diphenylacetylene to 2-(thiophen-2-yl)-quinazolin-4(3*H*)-one. It was shown that in the case of 2-phenylquinazolin-4(3*H*)-one, the same conditions favor the formation of benzonaphthyridine derivative **36** as a result of alcoholysis of the amide group and double annulation of phenylacetylene (Scheme 12.13). Derivatives **35** showed aggregation-induced emission in aqueous acetonitrile; with benzonaphthyridine derivative

Scheme 12.10.

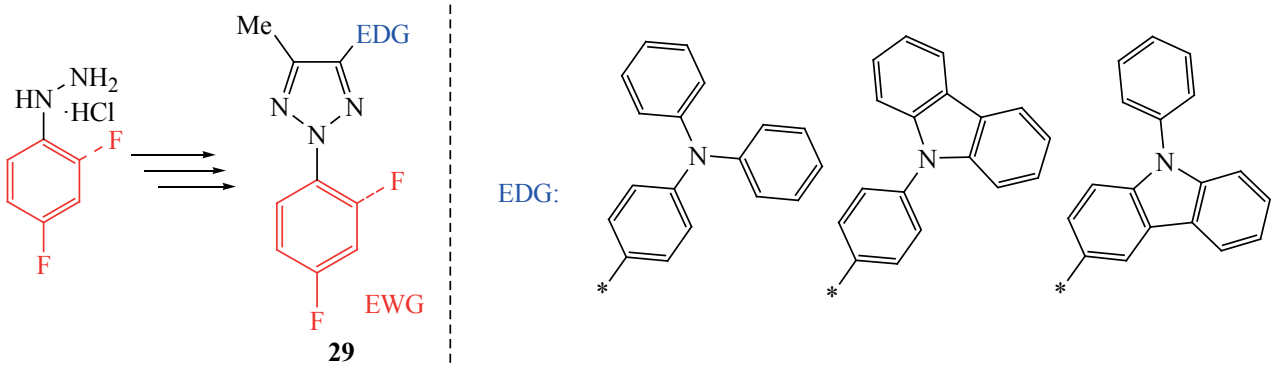


28

68–92%

28, R^1 = Ph, *p*-Tol, 2,4,6-$Me_3C_6H_2$, 4-$MeOC_6H_4$, 3-FC_6H_4, 2-ClC_6H_4, 3-ClC_6H_4, 4-ClC_6H_4, 4-BrC_6H_4, 4-PhC_6H_4, 4-$CF_3C_6H_4$, 1-naphthyl, PhC(O); R^1 = H, CH_3, Ph

Scheme 12.11.

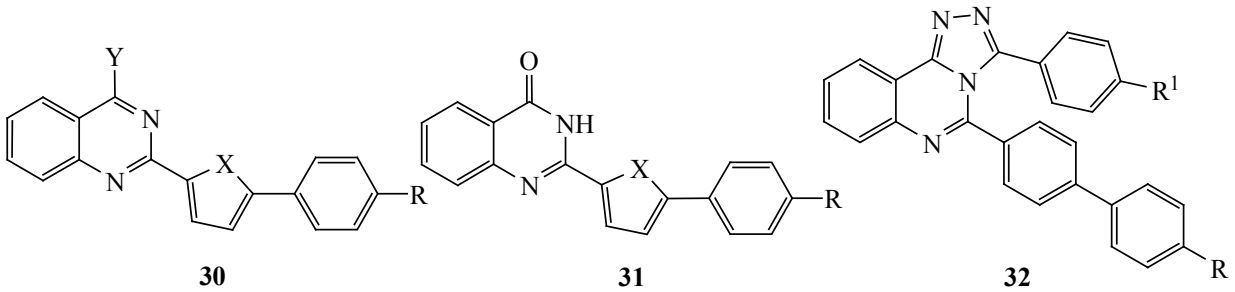


36, luminescence enhancement in the presence of Fe^{3+} cations was observed [708].

V-Shaped D-p-A-p-D chromophores **37–39** were synthesized based on 2,3-bis(aryl-thiophen-2-yl)quinoxaline and its dibenzo derivative (Scheme 12.14). A good sensitivity of chromophores of this class to nitro compounds of different natures was demonstrated [709–711].

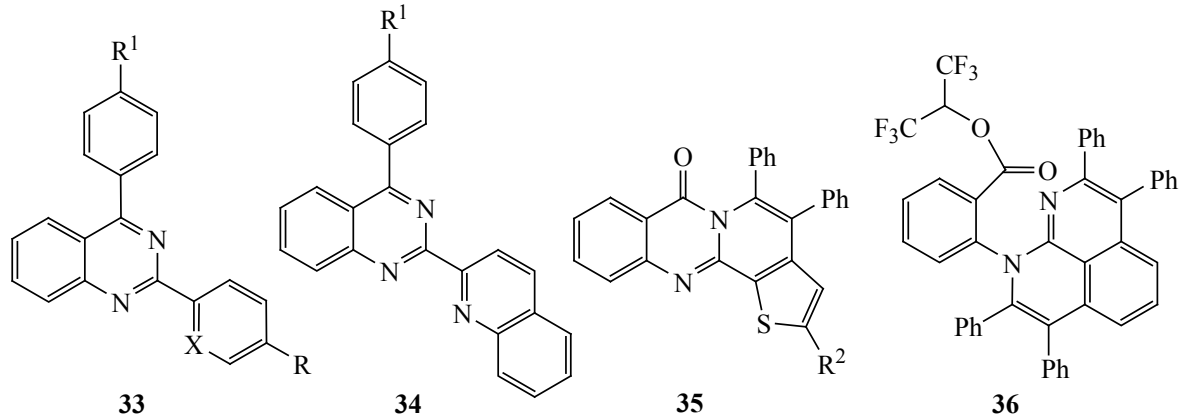
A series of 2-(2-hydroxyphenyl)quinazolin-4(3*H*)-ones **40** were synthesized, the effect of the nature

Scheme 12.12.



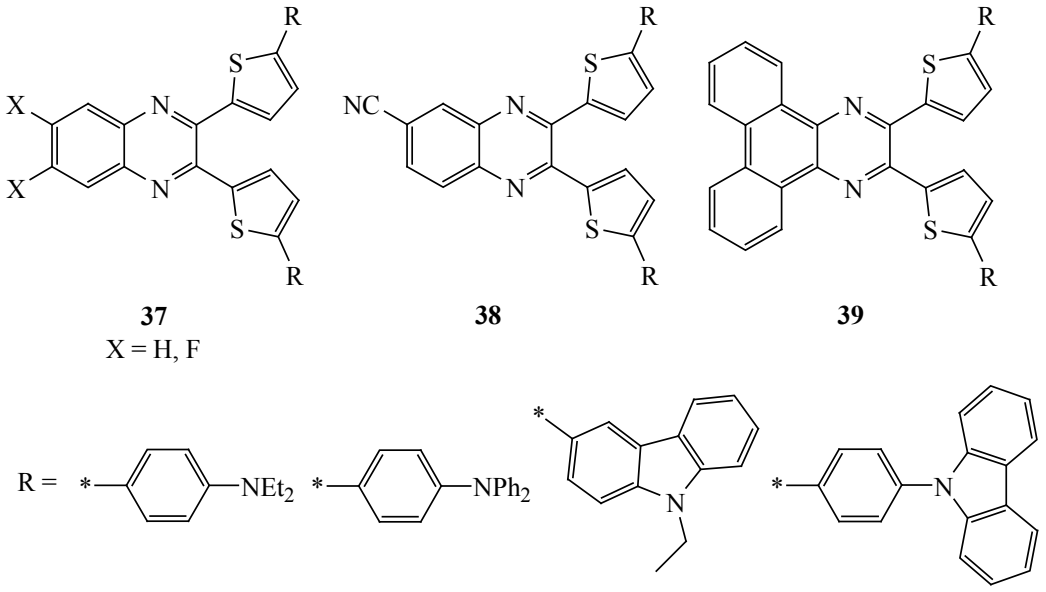
X = S, CH=CH; Y = morpholin-4-yl, CN;
R = NEt_2, NPh_2, 9*H*-carbazol-9-yl; R^1 = CH_3, OCH_3, CF_3

Scheme 12.13.



X = CH, N; R = CN, CF_3;
R^1 = NEt_2, NPh_2, 9*H*-carbazol-9-yl;
R^2 = H, Br, *n*-C_6H_{13}, 4-$NPh_2C_6H_4$

Scheme 12.14.

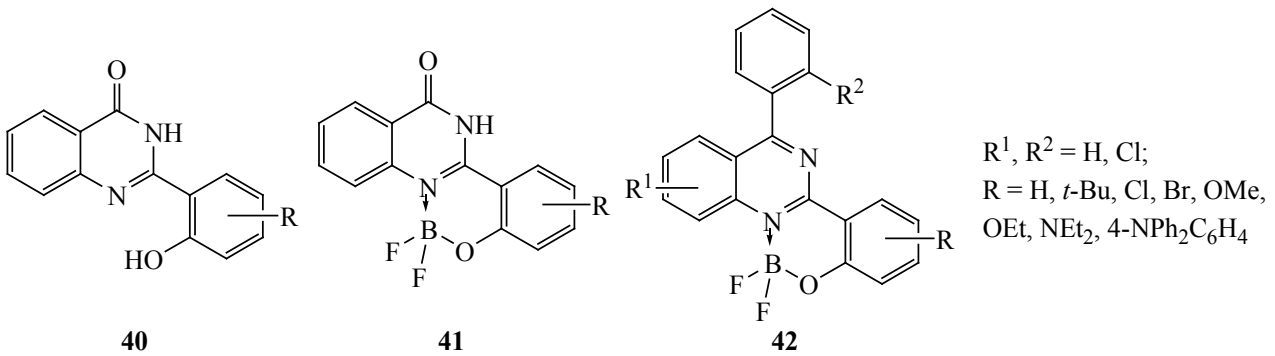


and position of substituent R on the photoinduced intramolecular proton transfer, as well as aggregation-induced emission enhancement were studied. The synthesis of BF_2 complexes **41** and **42** based on ligands **40** and 4-arylquinazoline analogs was performed (Scheme 12.15); the synthesized complexes showed strong emission in solution and in powder, as well as large Stokes shifts [712, 713].

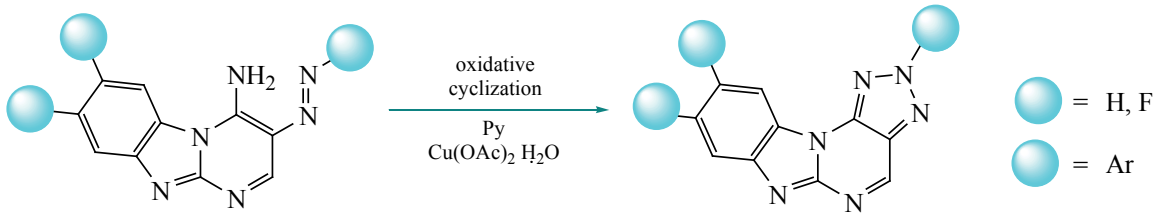
A highly efficient synthetic approach to a new class of polycyclic 8-azapurines, benzo[4,5]imidazo[1,2-*a*][1,2,3]triazolo[4,5-*e*]pyrimidines **44** was developed. The procedure involves the condensation of aminobenzimidazoles with 3-oxo-2-phenylazopropionitrile to form 3-(arylazo)benzo[4,5]imidazo[1,2-*a*]-pyrimidin-4-amines **43** and the subsequent oxidative cyclization of the latter, catalyzed by Cu(II) acetate, to afford target products **44**. The resulting 8-azapurines show good quantum yields (up to 60%) with absorption maxima at 379–399 nm and emission maxima at 471–505 nm (Scheme 12.16) [714].

A series of new luminophores, 4-(aryl)-benzo[4,5]-imidazo[1,2-*a*]pyrimidine-3-carbonitriles **45**, were synthesized from benzimidazole-2-arylimines via a combination of Povarov (aza-Diels–Alder) and oxidation reactions, and the photophysical properties of the synthesized compounds were studied. Fluorophores **45** demonstrated absorption with λ_{max} up to 421 nm and emission with λ_{max} up to 567 nm. In all cases, a positive emission solvatochromism with the Stokes shifts ranging from 120 to 180 nm was recorded. Some fluorophores **45** exhibited aggregation-induced emission (AIE) and mechanoluminescent properties, as well as fluorescence in the solid and film states (Scheme 12.17) [715].

Scheme 12.15.



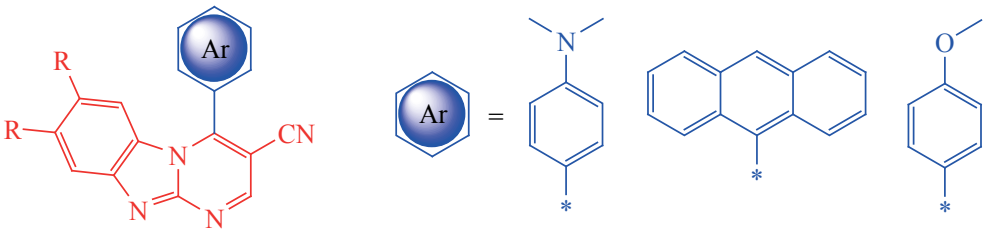
Scheme 12.16.



43 **44**

73–84%

Scheme 12.17.



45

R = H, F

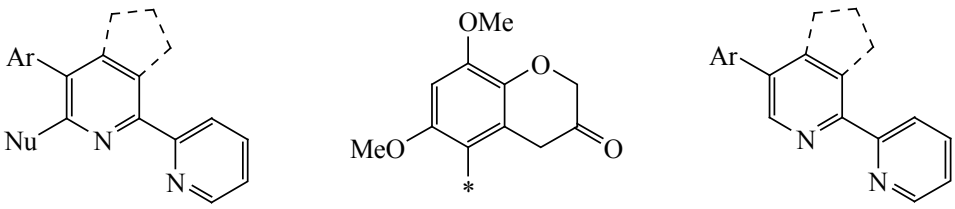
Combinations of S_N^H/S_N^{ipso} and aza-Diels–Alder reactions or Pd-catalyzed cross-coupling/aza-Diels–Alder reactions were used to obtain a series of 2,2'-bipyridines **46** and **47** as promising push–pull/AIE fluorophores (Scheme 12.18) [716–721].

12.3. Mechanochemical synthesis

Mechanochemical processes also seem quite promising for the development of green methods. The main advantage of the mechanochemical method is that it works at room temperature in the absence of a solvent and is applicable to a wide range of reactions. Thus, the fact that 1,3-dipolar azide–alkyne cycloaddition (AAC) reactions can be carried out under mechanochemical conditions in the absence of copper catalysts was demonstrated by the synthesis of 1,4-disubstituted-1,2,3-triazoles **48** and **49**, including the antiepileptic drug Rufinamide **50a** and its analogs, for example, chlorinated Rufinamide **50b** [722], as well as by the construction of PEG-linked bola-type bis-1,2,3-triazole-containing fluorescent chemosensors **51** showed their efficiency for the detection of nitro explosives, including the difficult-to-detect penaterithritol tetranitrate (PETN), with quenching constants of up to 10^4 M^{-1}. It should be noted that chemosensors **51** can also be obtained by the azide–alkyne cycloaddition reaction in solution in the presence of copper(I) salts (Scheme 12.19) [723].

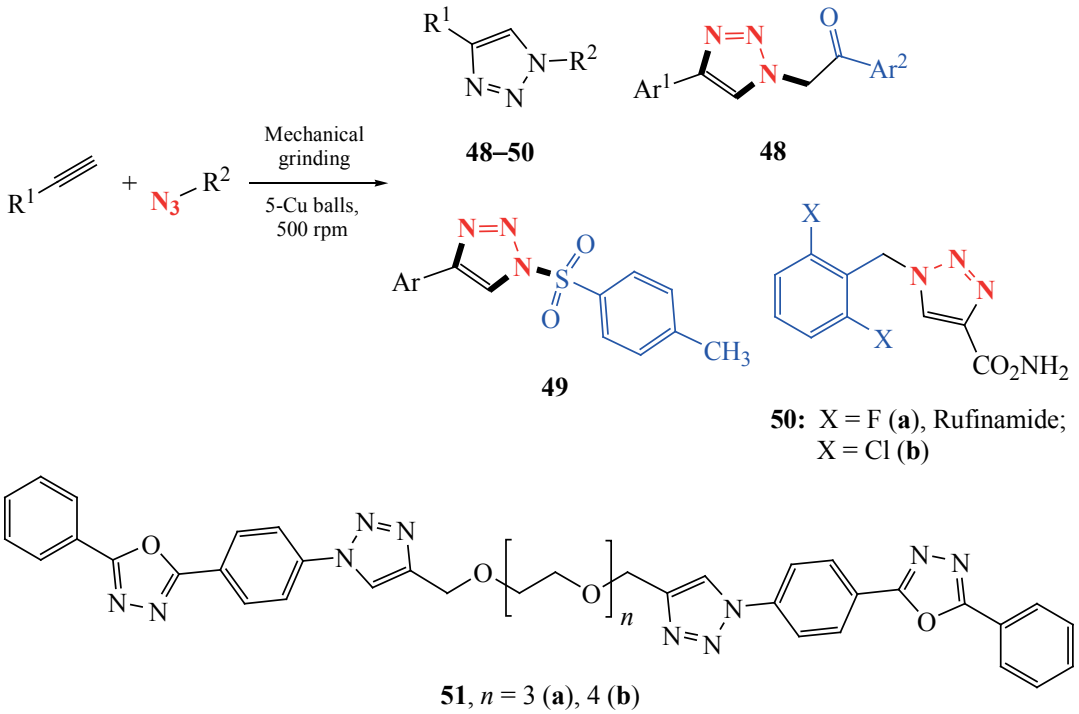
The efficiency of mechanochemical synthesis for the construction of heterocycles by two-component reactions was also demonstrated, as was evidenced by the synthesis of imidizolines by the reaction of aziridines with nitriles in the presence of $HClO_4$ [724], the synthesis of coumarins or annulated pyrano[2,3-*f*] and [3,2-*f*]indoles by the Pechmann reaction between phenols with β-keto esters under conditions of acid catalysis [725], as well as the multicomponent construction of pyrroles by the $FeCl_3$–catalyzed reactions between indoles, phenylglyoxal,

Scheme 12.18.



46 Nu = NHAr', S-Ar'', OAlk, **47**

Scheme 12.19.



48, Ar^1 = Ph, thiophen-2-yl, 4-PhC_6H_4, *p*-Tol, 4-(9*H*-carbazol-9-yl)C_6H_4, 4-$nC_5H_{11}OC_6H_4$; Ar^2 = Ph, thiophen-2-yl, 4-$MeOC_6H_4$, 3,4-$F_2C_6H_3$, naphthyl-2, 6-MeO-naphthyl-2; **49**, Ar = Ph, thiophen-2-yl, 4-PhC_6H_4, 4-(9*H*-carbazol-9-yl)C_6H_4, 4-$nC_5H_{11}OC_6H_4$.

anilines, and dimethyl acetylenedicarboxylate [726]. Finally, mechanochemical synthesis was used to obtain azomethines and azomethine-containing polyureas as chemosensors for visual detection of OH^- and F^- [727] or CN^- anions [728] and as synthons for the preparation of α-substituted azolylamines [729].

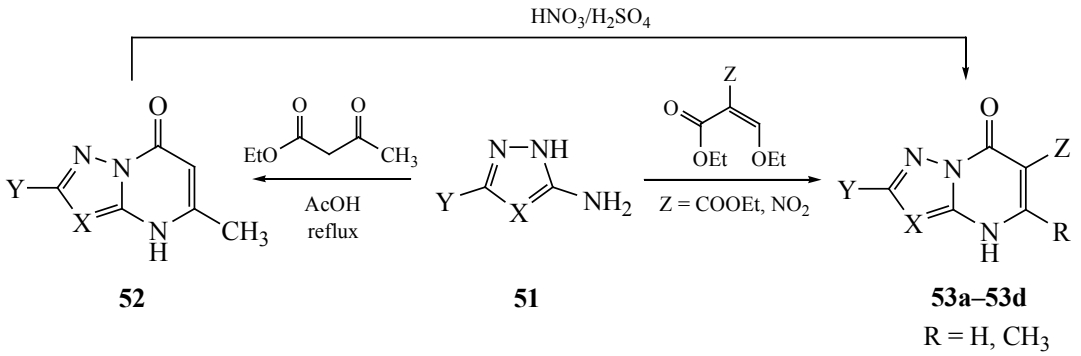
12.4. Synthesis of fused azolazines as candidate drugs

Ural chemists pay special attention to the pharmaceutically important group of polynitrogen bicyclic compounds with a bridging nitrogen atom, specifically azolo[1,5-*a*]pyrimidines, azolo[5,1-*c*]-1,2,4-triazines, or compounds containing these fragments. Since the ring system of these heterocycles is isoelectronic to the purine system, such compounds can be considered as possible substitutes of purine bases. To date, the structures under consideration represent new families of potential antiviral [730], antibacterial, antitumor, anticoagulant [731], and antidiabetic [732, 733] drugs, as well as drugs protecting against Alzheimer's, Parkinson's, and Huntington's diseases [734]. The domestic drug Triazavirin (Riamilovir) is sodium salt of 2-(methylsulfanyl)-6-nitro-1,2,4-triazolo[5,1-*c*]-1,2,4-triazinone dihydrate, which is actively used to treat acute respiratory viral infections, influenza, and coronavirus infections [735].

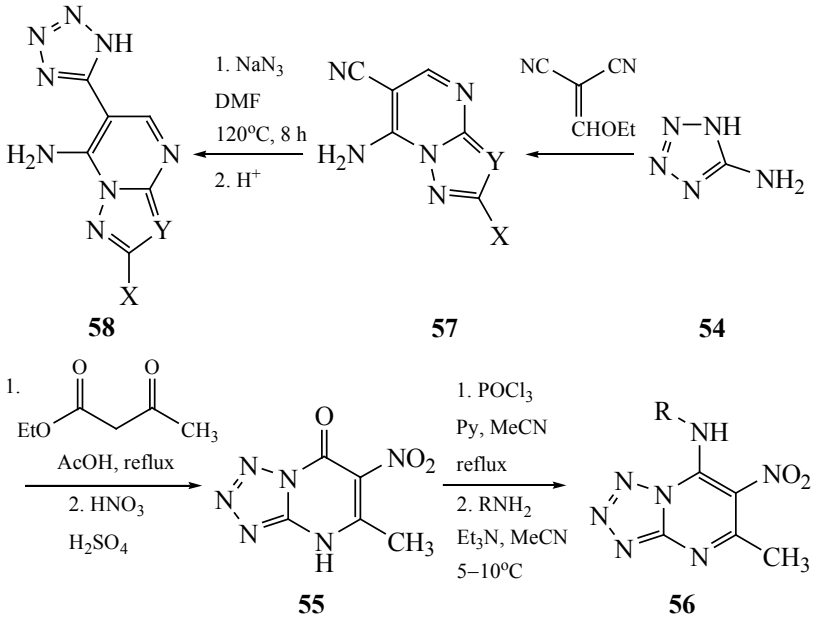
In recent years, we have identified derivatives of the azoloazines under consideration that have demonstrated significant potential in such therapeutic areas as cancer chemotherapy, diabetes, and viral and bacterial diseases, which allows us to expect that such heterocyclic systems hold promise in terms in the search for biologically active compounds. The main method of synthesis of the azolopyrimidine system is the cyclocondensation of aminoazoles with various bielectrophilic synthetic equivalents (Scheme 12.20). Some of the compounds obtained in this way, for example, 2,6-diethoxycarbonyl-1,2,4-triazolo[1,5-*a*]pyrimidin-7-one **53a**, showed pronounced anticoagulant activity [736].

Some of the nitro derivatives obtained in this way, for example, methylsulfanyl **53d** and furyl **53c** heterocycles demonstrated significant antiglycation activity, which can be used to prevent late complications of type 2 diabetes mellitus [733]. Nitro-containing

Scheme 12.20.



Scheme 12.21.



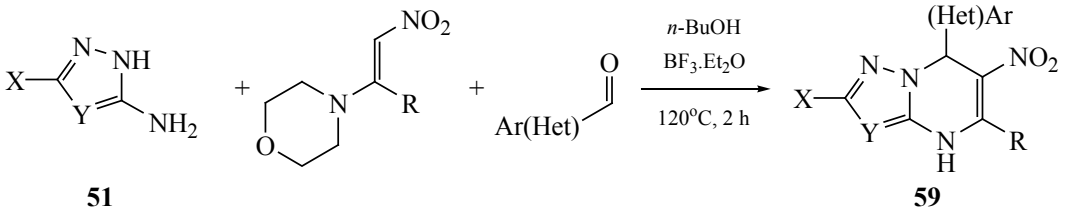
azolopyrimidines exhibit antiviral and strong antiseptic effects [730].

The considered azolo[1,5-*a*]pyrimidines are convenient objects for various modifications (Scheme 12.21). The reaction of aminotetrazole **10** with acetoacetic ester followed by nitration of the resulting heterocycle led to product **11**. The phosphoryl chloride–pyridine–acetonitrile system developed for chlorodeoxygenation turned out to be effective in this case, too: 5-methyl-6-nitro-7-chlorotetrazolopyrimidine was isolated in a yield of 75–85%. The subsequent reactions with different alkylamines made it possible to synthesize a series of derivatives **12**, among which potent anti-inflammatory agents were found. Experiments on in vivo cytokine storm models demonstrated gave evidence for the ability of compound **12a** to reduce thrombosis and inflammation in lung tissue, which can be used to treat COVID-19 complications [737].

The search for methods for the synthesis of azoloazine compounds with potential anticancer activity gave impetus to research into the reactivity of 6-cyano-substituted derivatives **21**. It was shown that the C≡N group can be effectively transformed into a tetrazole ring by treatment with sodium azide in DMF under heating. A series of 6-(tetrazol-5-yl)-7-aminoazolo[1,5-*a*]pyrimidines **22**, among which potent inhibitors of casein kinase 2 with a low micromolar IC_{50} values [738]. Thus, compounds **22** (Y = H, CN, C_6H_5) may be of interest as potential anticancer agents, since it was found that the growth rate of some types of malignant tumors correlates with the expression level of casein kinase 2.

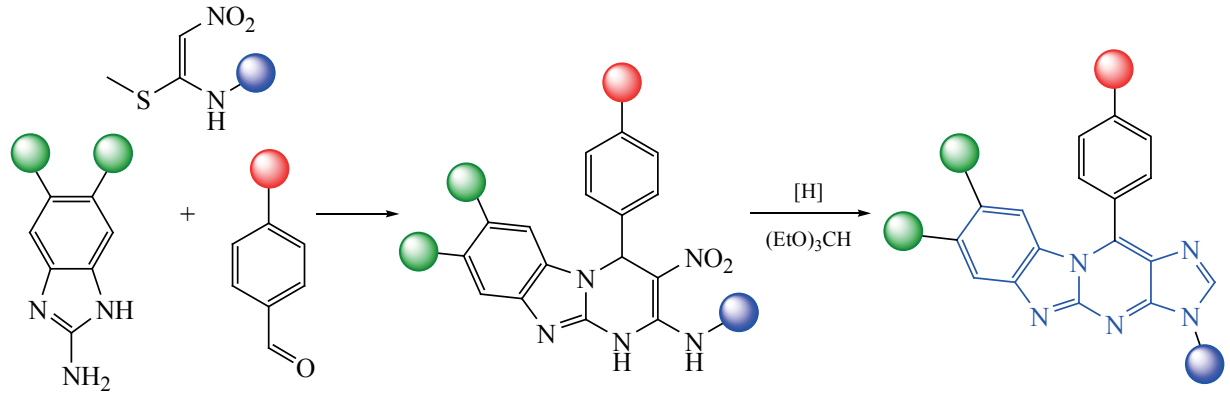
Another synthetic approach to 4,7-dihydroazolo[1,5-

Scheme 12.22.



51 **59**

Scheme 12.23.



60 **61**

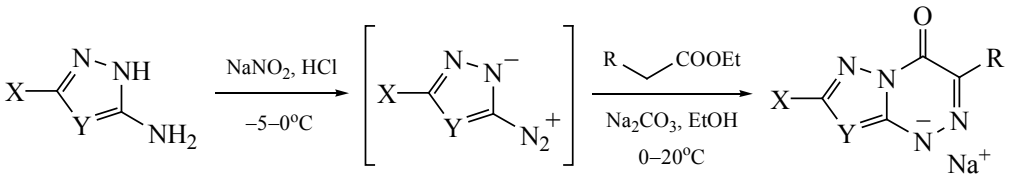
a]pyrimidines involved a three-component condensation of aminoazoles **51**, morpholinonitroethylenes, and (hetero)aromatic aldehydes (Scheme 12.22). The use of fluoroboric acid etherate as a catalyst resulted in the synthesis of a series of derivatives **59**. Among such 6-nitro-7-(het)arylazolopyrimidines **59**, compounds with a high cytotoxic effect on tumor cells were identified [739].

A highly efficient Pot, Atom, and Step Economy (PASE) approach to a new class of polycyclic purine derivatives was also proposed. The strategy involves consecutive metal reduction in acidic media, autoaromatization, and heterocyclization of nitrobenzimidazopyrimidines **60** obtained by three-component condensation (Scheme 12.23). New benz[4,5]imidazo[1,2-*a*]purine derivatives **61** were synthesized in good yields, and their proposed structure was confirmed by X-ray diffraction (XRD) data. The resulting convergent benzimidazopurines combine two relevant frameworks relevant for medicinal chemistry: benzimidazole and purine [740].

The scheme for the addition of an azine ring to an azole ring is also actively used in the synthesis of biologically active azolo[5,1-*c*]-1,2,4-triazines **63** and involves the reaction of diazoazoles **62** with esters of malonic, cyanoacetic, or nitroacetic acids [730, 741] (Scheme 12.24). This reaction underlies the synthesis of the antiviral drug Triazavirin and its analogs [735].

Recent studies have shown that synthesized azolo-[5,1-*c*]-1,2,4-triazines show an antiglycation effect: they inhibit the reaction of non-enzymatic protein glycation (the Maillard reaction), the main cause of the negative impact of diabetes mellitus [732]. This made it possible to develop an effective antiglycation drug (AB-19) based on sodium diethyl 4-oxo-1,4-dihydropyrazolo[5,1-*c*]-

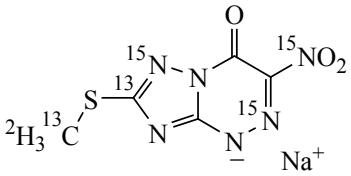
Scheme 12.24.



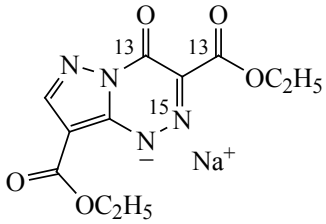
51 **62** **63**

R = NO_2,CN, COOEt; X = H, Alr, Ar, Het, SAlk; Y = N, CH, CCN, CCOOEt, CBr, CNO_2

Scheme 12.25.



64



65

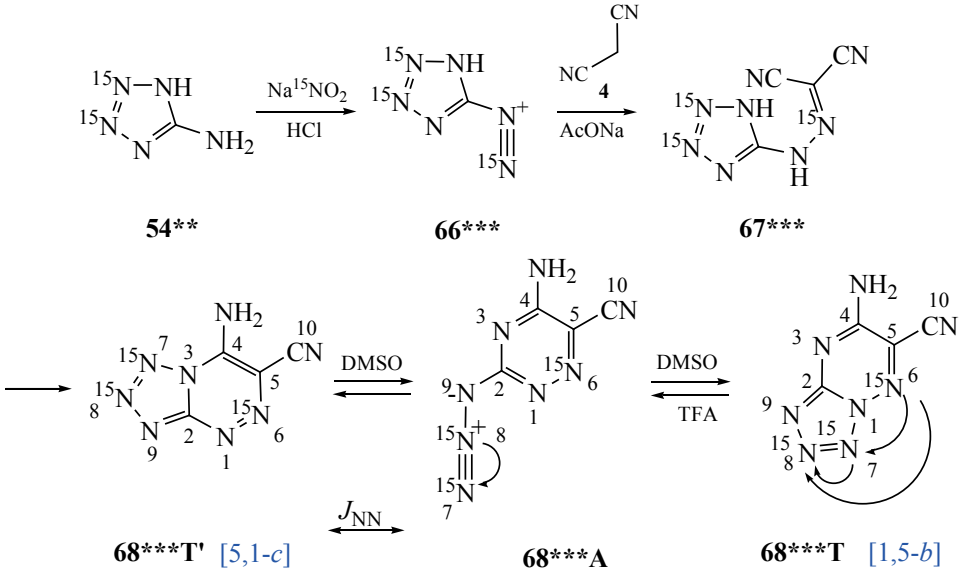
1,2,4-triazine-3,8-dicarboxylate monohydrate [742–745], which is currently prepared for clinical trials.

The most promising way to study the metabolism of azaheterocycles is to use isotopically labeled atoms. To this end, Triazavirin **64** and AB-19 **65** (Scheme 12.25) selectively labeled by ^{2}H, ^{13}C, and ^{15}N were synthesized [746, 747].

Moreover, an analysis of the ^{1}H–^{15}N and ^{13}C–^{15}N coupling constants in the NMR spectra of ^{15}N-labeled samples significantly expands the potential of NMR spectroscopy in research into the structural features and chemical transformations of azines, azoles, azoloazines, and other azaheterocycles [748]. As shown, the introduction of a few ^{15}N labels simultaneously into the azole and azine fragments makes it possible to study the azide–tetrazole equilibrium by the ^{15}N NMR spectroscopy. The potential of this approach was demonstrated using the examples of tetrazolo[1,5-*b*][1,2,4]triazine **68***T** (Scheme 12.26) and tetrazolo[5,1-*b*]quinazoline [749]. For example, an analysis of the ^{15}N–^{15}N coupling constants and chemical shifts of labeled nitrogen atoms provided unambiguous evidence for the structure of the tetrazole form even in cases, when two cyclic isomers are formed. Furthermore, these characteristics were used to fix the azido forms in solution. It is important to note that the described approach was used for the first time to assess the azide–tetrazole equilibrium.

Recently, the usefulness of the ^{13}C isotope for studies of the adamantylation of 1,2,4-triazolo[1,5-*a*]-pyrimidin-7-ones in trifluoroacetic or perfluorovaleric acids was demonstrated [750]. The introduction of a ^{13}C label to the azole ring made it possible to detect the rearrangement of 1,2,4-triazolo[1,5-*a*]pyrimidines into 1,2,4-triazolo[4,3-*a*]pyrimidines, which accompanies the N-adamantylation process. In this case, the main diagnostic structural information comes from the values of long-range ^{13}C–^{13}C coupling constants ($^{2}J_{CC}$ and $^{3}J_{CC}$).

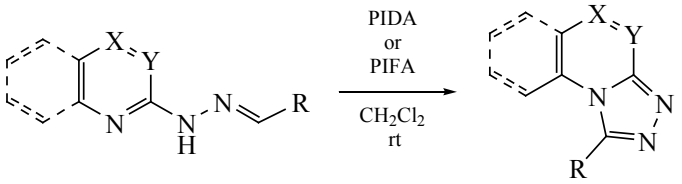
Scheme 12.26.



54** **66***** **67*****

68*T'** [5,1-*c*] **68***A** **68***T** [1,5-*b*]

Scheme 12.27.



69 **70**

X, Y = CH, N

R = 2-thienyl, 3-thienyl, 5-bromo-2-thienyl, ferrocenyl, cymantrenyl

The second way to constructing the azoleazine structure, specifically the build-up of the azole fragment, was used to synthesize 1,2,4-triazolo[4,3-*a*]azines **70** under hypervalent iodine catalysis. The approach is based on the intramolecular cyclization of 2-azinylhydrazones **69** in the presence of (diacetoxy)iodobenzene (PIDA) or bis(trifluoroacetoxy)iodobenzene (PIFA) (Scheme 12.27). As a result, hetaryl and metallocenyl azoloazines **70** were obtained in yields of 40–93%. The ability of 1,2,4-triazolo[4,3-*a*]azines to activate heat shock factor 1 (HSF1) was explored to reveal HSF1 activity enhancement. It was shown that 1,2,4-triazines can be used to induce the expression of the heat shock protein Hsp70 and inhibit formation of mutant HTT aggregates, which may prove useful for protection from brain diseases [751].

It was found that pyrrolyl and indolylazine derivatives exhibit Hsp70-inducing properties. The synthesized compounds were tested on an in vitro model of Alzheimer's disease and a model of secondary damage after traumatic brain injury. In both cases, pyrrolyl- and indolylazines demonstrated a significant therapeutic effect, increasing the survival of neuronal cells. The half-lethal concentration of the selected compounds, estimated in human neuronal cell culture, suggest an extremely low level of their cytotoxicity [752–754].

In the search for new nervous system active compounds, previously unknown tacrine derivatives were synthesized as candidate therapeutics for Alzheimer's disease [755, 756]. In silico docking studies of the synthesized compounds as acetylcholinesterase inhibitors in relation to CYP1A2 and CYP3A4 protein complexes demonstrated promise these series of compounds hold in terms of searching for drug candidates.

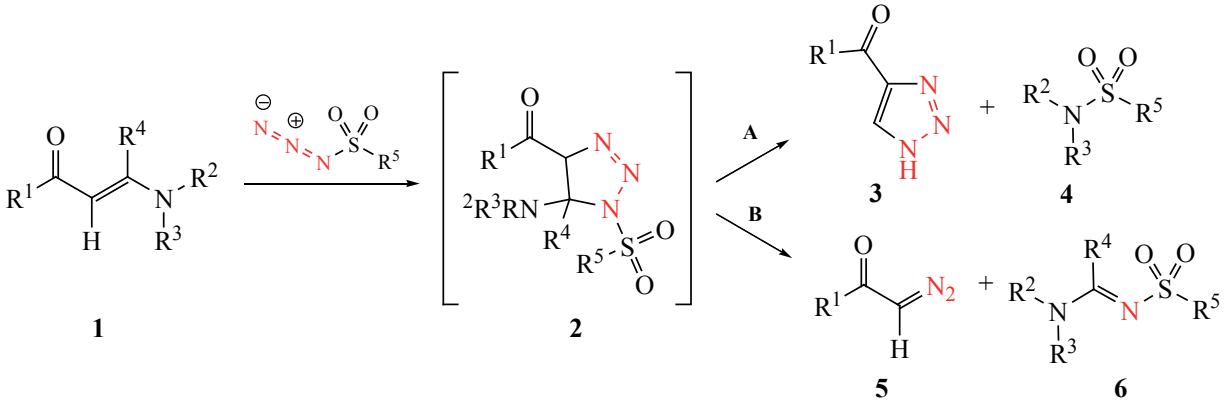
The condensation of mono- and dihydrazinylazines with (heteroaryl)carbaldehydes followed by oxidative cyclization of mono- and bis-azinylhydrazones in the presence of hypervalent iodine(III) gave mono- and bis-1,2,4-triazoloazines, which exhibited cytotoxic activity against MCF7, DLD-1 and A549 cancer cell lines, as well as selective toxicity to human dermal fibroblasts (DF-2) [757].

13. DEPARTMENT OF THE TECHNOLOGY OF ORGANIC SYNTHESIS OF URAL FEDERAL UNIVERSITY. FUNDAMENTAL AND APPLIED ASPECTS OF THE CHEMISTRY OF 1,2,3-TRIAZOLES

1,2,3-Triazoles have played an important role in heterocyclic chemistry since their discovery by Pechmann in 1888. Their popularity increased significantly after the development of efficient synthetic approaches to 1,4- and 1,5-disubstituted 1,2,3-triazoles by Meldal and Sharpless in 2002. Based on the CuAAC and RuAAC reactions, a powerful methodology was created for the synthesis of a variety of 1,2,3-triazole derivatives and, as a result, for the identification of substances with practically useful properties among them.

Despite the enormous achievements of the CuAAC and RuAAC reactions in the synthesis of 1,2,3-triazole derivatives, triazolines and triazoles with exocyclic double bonds remained inaccessible. A significant limitation of this strategy is the impossibility of direct synthesis of N^2-substituted derivatives of this heterocycle. This to a greater extent relates 2-aryl-1,2,3-triazoles, the interest in which is associated the fact that their photophysical and biological properties makes them promising for use in two important areas: as biologically active compounds [758] and in materials chemistry [759]. Therefore, Department of the Technology of Organic Synthesis of Ural Federal University is searching for and developing new methods of the synthesis of 1,2,3-triazoles by the reactions of azides with enamines, 2-cyanothioacetamides, and

Scheme 13.1.



amidines with electrophilic azides and the oxidation of hydrazones.

13.1. Reactions of enamines with azides: approach to 1,2,3-triazoles

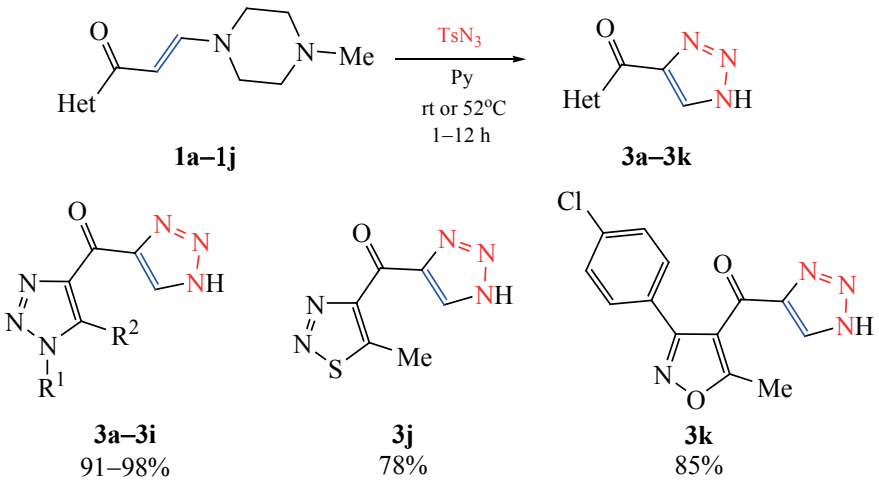
In our review published in 2018 [760] we reported evidence that enamines exhibit exceptionally high reactivity in 1,3-dipolar cycloaddition reactions with azides compared to other dipolarophiles. It was shown that the initial reaction products 1,2,3-triazolines are generally unstable and prone to interesting reactions and various ring transformations [760]. These data prompted us to continue our earlier research on enamine reactions with azides.

We found that the reaction of azolylenamines **1** with sulfonyl azides forms a hardly separable mixture of two pairs of compounds derived from triazolines **2**: 4-azoloyl-NH-1,2,3-triazoles **3** with sulfonamides **4** (route A) and azolyldiazo ketones **5** with *N*-sulfonylamidines **6** (route B) formed as a result of two competing reactions (Scheme 13.1) [761].

It was shown that the electron-donor methyl or methoxy group in the *para* position of arylsulfonyl azides facilitates the synthesis of NH-1,2,3-triazoles **3**. On the other hand, the highly electrophilic 4-nitrophenylsulfonyl azide facilitates the formation of a mixture of diazoketones 5 and sulfonylamidines **6**. We found that the direction of each reaction is controlled not only by the nature of the starting enaminones **1** and sulfonyl azides, but also depends on the solvent. Using pyridine as a solvent favors the formation of NH-triazoles **3**.

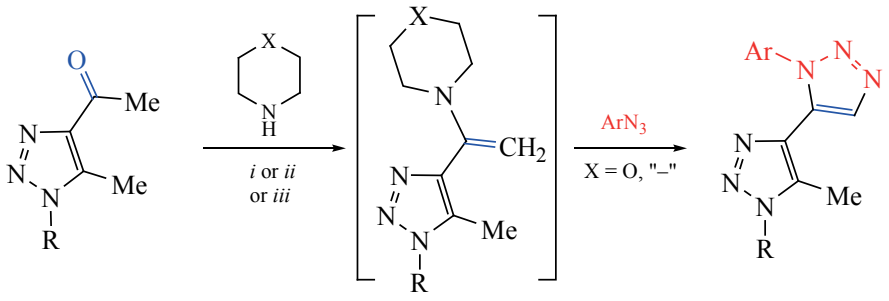
Our optimized procedure for the synthesis of NH-triazoles **3** involves the reaction of enaminones **1** with

Scheme 13.2.



R^1 = Ph, Me, Bu, 4-MeC_6H_4, 2-$O_2NC_6H_4$, 3-FC_6H_4; R^2 = H, Me

Scheme 13.3.



7 **8** **9**

40–70%

8 compounds

i, $TiCl_4$, 95–105°C, 7–36 h; *ii*, CaO, 120°C, 72 h

iii: *no additives*, 95–120°C,12–48 h

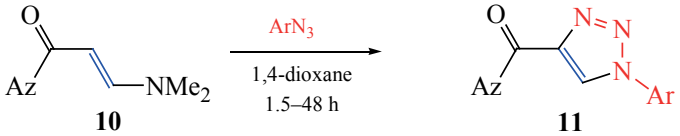
R = Ph, Me, *n*-Bu, 4-$BrC_6H_4CH_2$, Ar = Ph, 4-$O_2NC_6H_4$, 4-ClC_6H_4

tosyl azide in pyridine at 25−52°C, followed by washing out the water-soluble sulfonamides **4** with water (Scheme 13.2) [761].

Linearly coupled polyazoles containing heterocycles are attractive objects for medicinal chemistry [762], organic synthesis [763, 764], materials chemistry [765], and organic electronics [766]. In this regard, the synthesis of bis-1,2,3-triazoles coupled both by a single bond and via a carbonyl group is a topical task. The most popular synthesis of 1,2,3-triazoles by CuAAC reactions has limited application for the synthesis of azolyltriazoles due to the poor accessibility of azolylacetylenes. By contrast, the preparation of the target compounds by the reaction of azides with much more accessible enamines is a more promising synthetic route [760].

To obtain enamines **8**, we treated 4-acetyl-1,2,3-triazoles **7** with morpholine and pyrrolidine under various conditions (in the presence of CaO or *p*-TsOH, in the absence of a solvent or in toluene with its distillation) for a few hours [767]. However, in all these

Scheme 13.4.

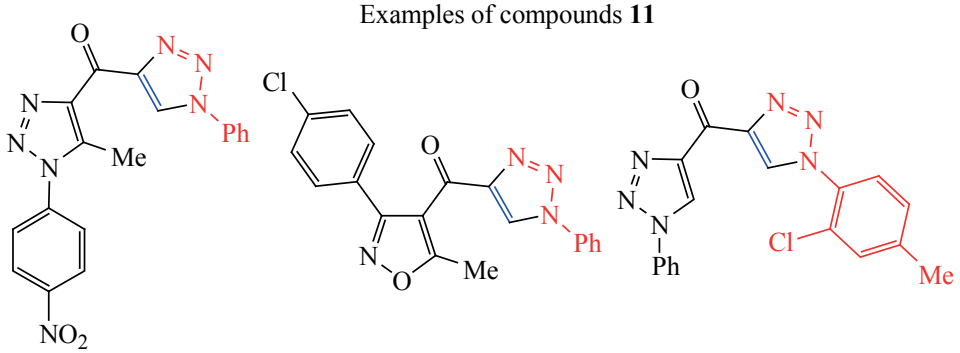


10 **11**

18–86%

9 compounds

Examples of compounds **11**

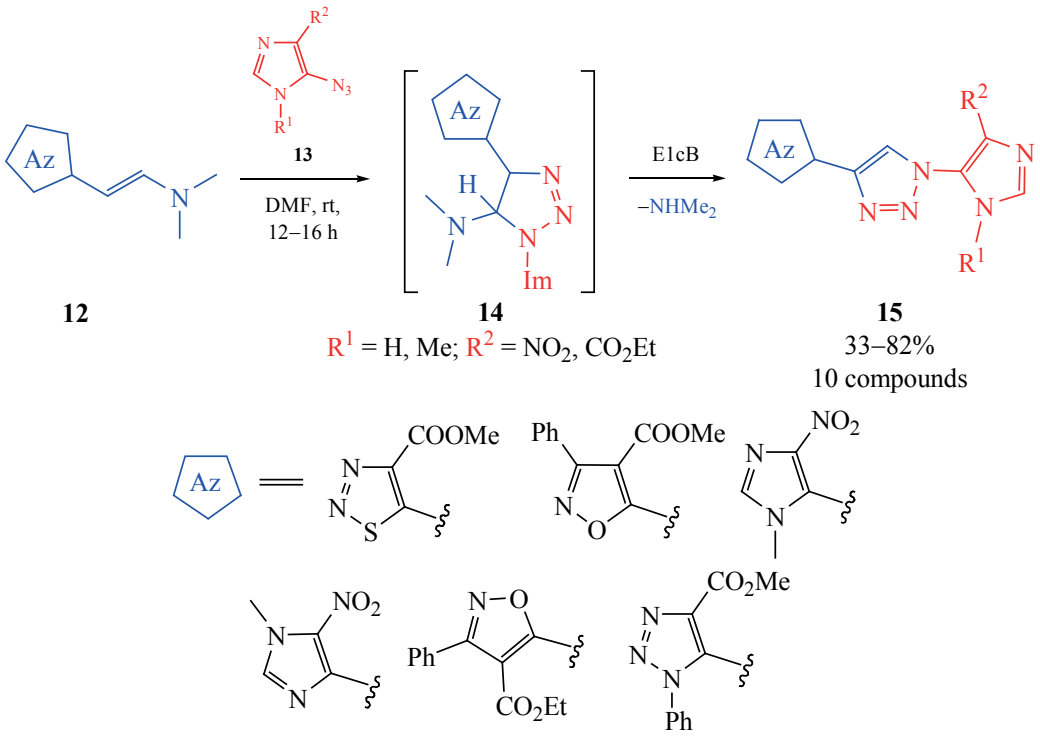


11a 86%

11b 63%

11c 79%

Scheme 13.5.



experiments, the starting ketones **7** remained unchanged. We found that refluxing ketones 7 and a large excess of aryl azides for many hours under various conditions (in morpholine or in pyrrolidine, without additives or in the presence of CaO or $TiCl_4$) results in the formation of bis-1,2,3-triazoles **9** (Scheme 13.3).

The use of enaminones for the synthesis of bisheterocyclic systems, where the azole fragment and the 1,2,3-triazole ring are linked via a carbonyl group, was demonstrated by the reaction of enaminones **10** with aryl azides (Scheme 13.4), providing 4-(1,2,3-triazole-4-carbonyl)azoles **11** in moderate to good yields [767].

2-Nitro- and 5-nitroimidazoles exhibit both high biological activity and luminescent properties and are used in the diagnosis and treatment of dangerous diseases [768]. Despite the progress in the development of new and simple methods for the synthesis of imidazoles, the synthesis of imidazoles linearly coupled with other azole rings via 1,2,3-triazoles is still a poorly developed issue. We proposed [769] an environmentally benign and efficient synthetic approach to triheterocyclic compounds, based on the 1,3-dipolar cycloaddition of β-azolylenamines **12** to 5-azidoimidazoles **13** followed by the aromatization of triazolines **14** to 1,2,3-triazoles **15** by the elimination of an amine molecule (Scheme 13.5).

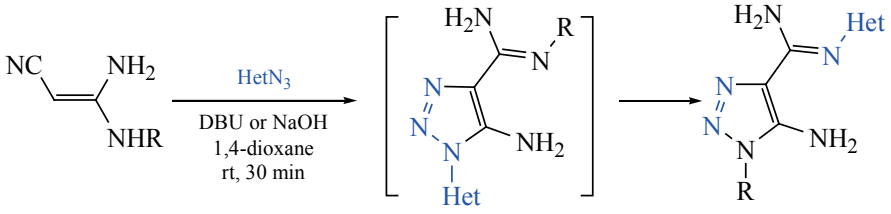
The experimental and theoretical evidence [767] suggested an E1cB mechanism of this transformation.

An efficient base-catalyzed synthesis of bisheterocycles coupled by an amidine linker and comprising, along with the newly formed 1,2,3-triazole ring, an additional heteroring: pyrimidinedione, 4-nitroimidazole, isoxazole, 1,2,4-triazole, 2-oxochromone, or thiazole [770]. The process involves the cycloaddition of 3,3-diaminoacrylonitriles **16** to heterocyclic azides [771] followed by the Cornforth-type rearrangement of intermediates **17** to final products **18** (Scheme 13.6). The developed method has a wide range of application and can be used to obtain various *N*-heteroaryl-1,2,3-triazole-4-carbimidamides **18** containing alkyl, allyl, propargyl, benzyl, cycloalkyl, or indolyl substituents on N^1.

13.2. Transformations of 1,2,3-triazole and 1,2,3-triazoline rings

Readily available 4,5-cycloalkeno-1-sulfonyl-1,2,3-triazoles **19** are stable toward hydrogen β-shift and can serve as a source of Rh(II) iminocarbenoids in the Rh(II)-catalyzed reaction with nitriles, which allowed

Scheme 13.6.



16 **17** **18**

46–98%
21 compounds

R = Bn, Ar, Alk, allyl, propargyl, heteroaryl

Het = pyrimidinyl, imidazolyl, isoxazolyl, 1,2,3-triazolyl, indolylethyl

the synthesis of 1-sulfonylcyclohexeno, cyclohepteno-, dihydropyrano-, 5-phenyltetrahydrobenzo-, and 4,5-dihydronaphtho[*d*]imidazoles **20** in good yields (Scheme 13.7) [772].

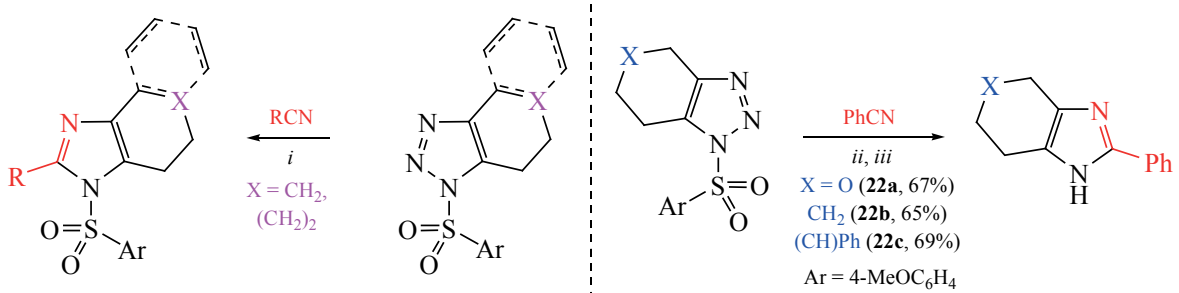
1-Sulfonyl-1,2,3-triazoles **21** can be readily converted to NH-imidazoles **22** by the Rh(II)-catalyzed reaction with benzonitrile and the subsequent hydrolysis of 1-sulfonylimidazole intermediates (Scheme 13.7) [772].

Ring transformation of heterocyclic compounds represents an interesting approach to different types of organic compounds [773]. These processes often have unusual mechanisms, expanding our knowledge of the reactivity of heterocycles. We found that 1,2,3-triazolines **24** formed from enamines **23** and heterocyclic azides lose nitrogen upon short reflux or at room temperature in methanol, which is accompanied by the opening of the cyclopentane ring to form *N*-heteroarylamidine valeric acid derivatives **25** (Scheme 13.8) [773].

Triazolines containing a 1,3,5-triazine ring in position *1* do not form valeric acid amidines under reflux in methanol. However, when dissolved in acetic acid, 1,2,3-triazoline **26** undergoes another type of transformation leading to *N*-(1,3,5-triazin-2-yl)diaminoalkene **27c**, the structure of which was confirmed by XRD analysis (Scheme 13.9) [774].

Thus, the presence of a strongly electron-acceptor heteroaromatic radical in position *1* of the triazoline ring is a key factor that controls the direction of the transformation of cyclopentano[*d*][1,2,3]triazolines **24** and **26** into valeric acid amidines **25** and diaminoalkenes **27**, respectively.

Scheme 13.7.



20 **19** **21a–21c** **22a–22c**

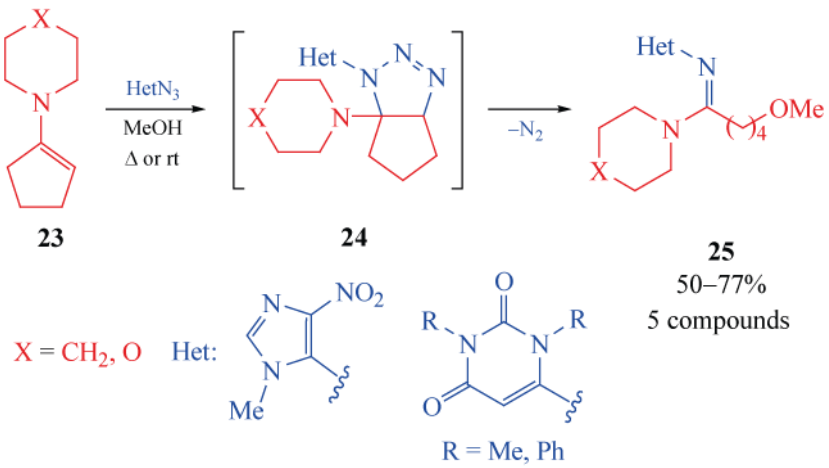
54–85%
15 compounds

R = Me, Ph, 3-FC_6H_4, 3-ClC_6H_4, 4-$O_2NC_6H_4$ etc.
Ar = 4-$MeOC_6H_4$, 4-$F_3CC_6H_4$, 4-MeC_6H_4 etc.

i, $Rh_2(Piv)_4$ or $Rh_2(OAc)_4$, $CHCl_3$, 100°C, 24 h

ii, $Rh_2(Piv)_4$, $CHCl_3$, 100°C, 6 h;
iii, H_2O, MeOH, 100°C, 10 h

Scheme 13.8.



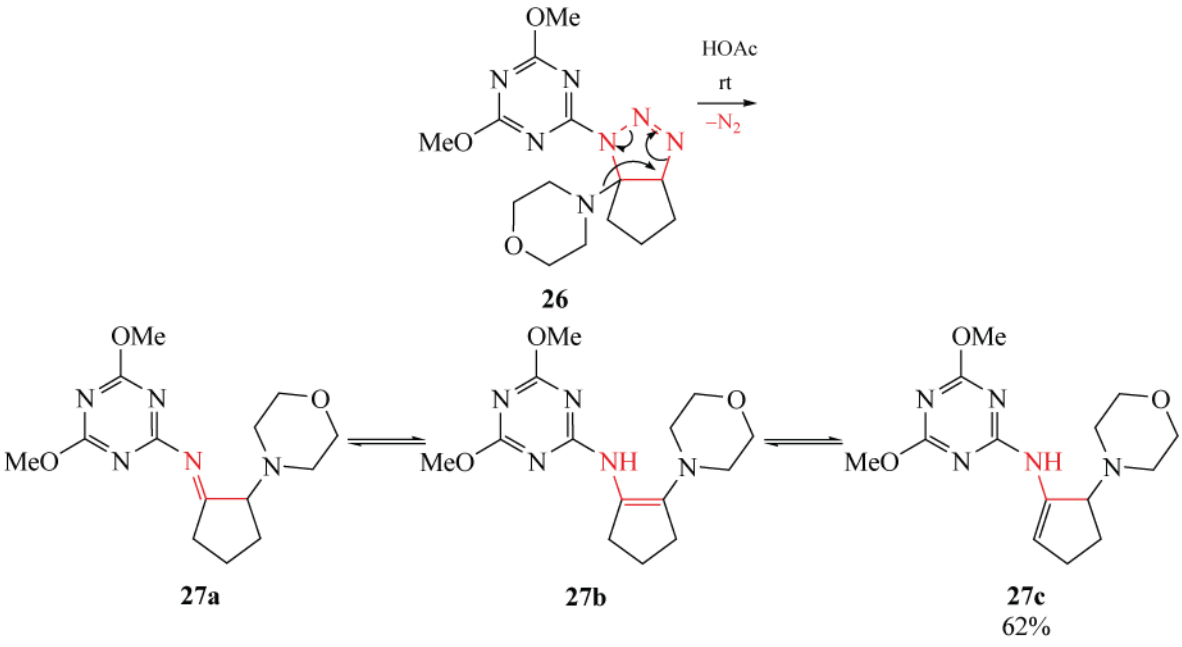
13.3. Synthesis and photophysical properties of mono- and bicyclic 2-aryl-1,2,3-triazoles

The synthesis of new 2-aryl-1,2,3-triazole derivatives was aimed at modeling functional groups, as well as introducing different combinations of functional groups and substituents into the heteroring and aromatic ring to study the theoretical aspects of photophysical properties and determine the possibilities of the realization of optical phenomena and tuning of the spatial structure and electronic properties.

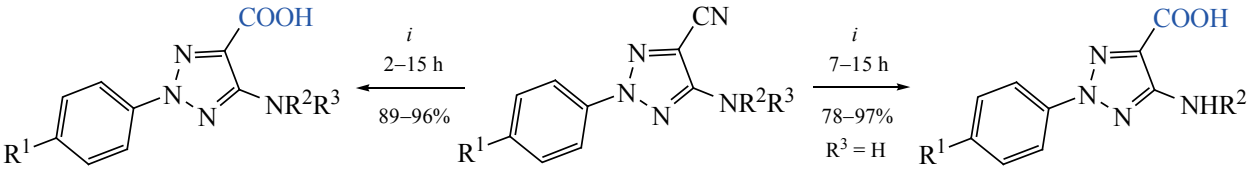
The interest in 2-aryl-1,2,3-triazole-4-carboxylic acids is due to the fact that the carboxyl group is widespread in natural and biomolecules and often present in the structure of synthetic biologically active compounds. Derivatives of triazolecarboxylic acids **29** and **30** were obtained by the alkaline hydrolysis of 1,2,3-triazole-4-carbonitriles **28** (Scheme 13.10) [775].

The study of the photophysical properties of acids **29** and **30** revealed a hypsochromic shift of the absorption (322–350 nm) and emission (416–451 nm) maxima and an increase in the quantum yields by 1.2–3.2 times (Φ = 20.6–96.0%) for compounds containing electron-donor substituents (R^1 = OMe). In a DMSO-water binary

Scheme 13.9.



Scheme 13.10.



29
11 compounds

28

30
7 compounds

NR^2R^3 = pyrrolidin-1-yl, piperidin-1-yl, azepan-1-yl, 4-Ph-piperazin-1-yl

R^1 = OMe,Cl, CF_3

R^2 = Me, Cy

i, $EtOH/H_2O$, excess NaOH

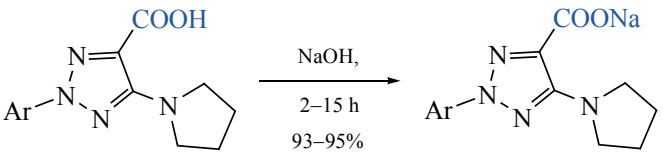
solvent, the absorption and emission maxima shift to the short-wavelength region by 10–22 and 1–14 nm, respectively. However, if the emission intensity of acids **29** does not change or increases, in the case of triazoles **30**, a significant decrease in the quantum yield (by 3.5–16 times) occurs. Thus, the structure of the amino group on the C^5 atom of the heteroring sharply separates the acids into two groups in terms of the sensitivity of fluorescence to the content of water in the binary solvent. The different aggregation behavior of acids **29** and **30** can be explained by the morphological differences in the formed nanoparticles due to different conformations and packing models, which are apparently mainly determined by the substituent in the amino group. The p*K*a values of acids **29** and **30** (7.65–8.08 and 3.05–3.45, respectively) were determined from their absorption and emission spectra in various buffer solutions. These values correspond to the pH ranges suitable for effective assessment of changes in the acidity of biological fluids.

To gain a deeper understanding of the nature of intra- and intermolecular contacts and evaluate their effect on the properties of 1,2,3-triazole-4-carboxylic acids, an extensive study of two representative examples, acids **29a** and **29b** and their sodium salts **31a** and **31b** was performed (Scheme 13.11). Experimental (spectral) and theoretical (DFT quantum-chemical calculations) methods were used in this study [776–778]. The optical properties of 1,2,3-triazole-4-carboxylic acids and their sodium salts were studied in different solvents (1,4-dioxane, DMSO, MeOH) and mixtures of these solvents with water. As follows from the resulting data, in polar and non-polar solvents, fluorescence is produced by strong neutral associates. Methanol (a protic solvent) weakens the association of acid molecules, and, a result, new fluorescent particles are formed. In binary mixtures with high water contents, acids **29** exhibit optical characteristics similar to those of their salts, and, therefore, an anionic character can be suggested. These results together pointed to a strong dependence of the photophysical properties of acids **29** on the medium, and allowed these acids to be considered as candidate sensors for analytes with labile protons. The spectral and calculated data provided evidence showing that, along with the COOH group, which is expectedly actively involved in intermolecular interactions, the nitrogen atoms of the 1,2,3-triazole ring, too, are active participants of such interactions.

Molecular docking revealed four sites (ASP185, LYS44, LEU49, and VAL51) capable of forming H-bonds between acid **29b** and amino acids of matrix metalloproteinase (MMP-2). The carboxyl group appears to play an important role by forming two H-bonds with the LEU49 and VAL51 residues [778].

The amide group is widespread in living systems and is a structural unit of compounds that play an important role in almost all biological processes. Carboxamides

Scheme 13.11.

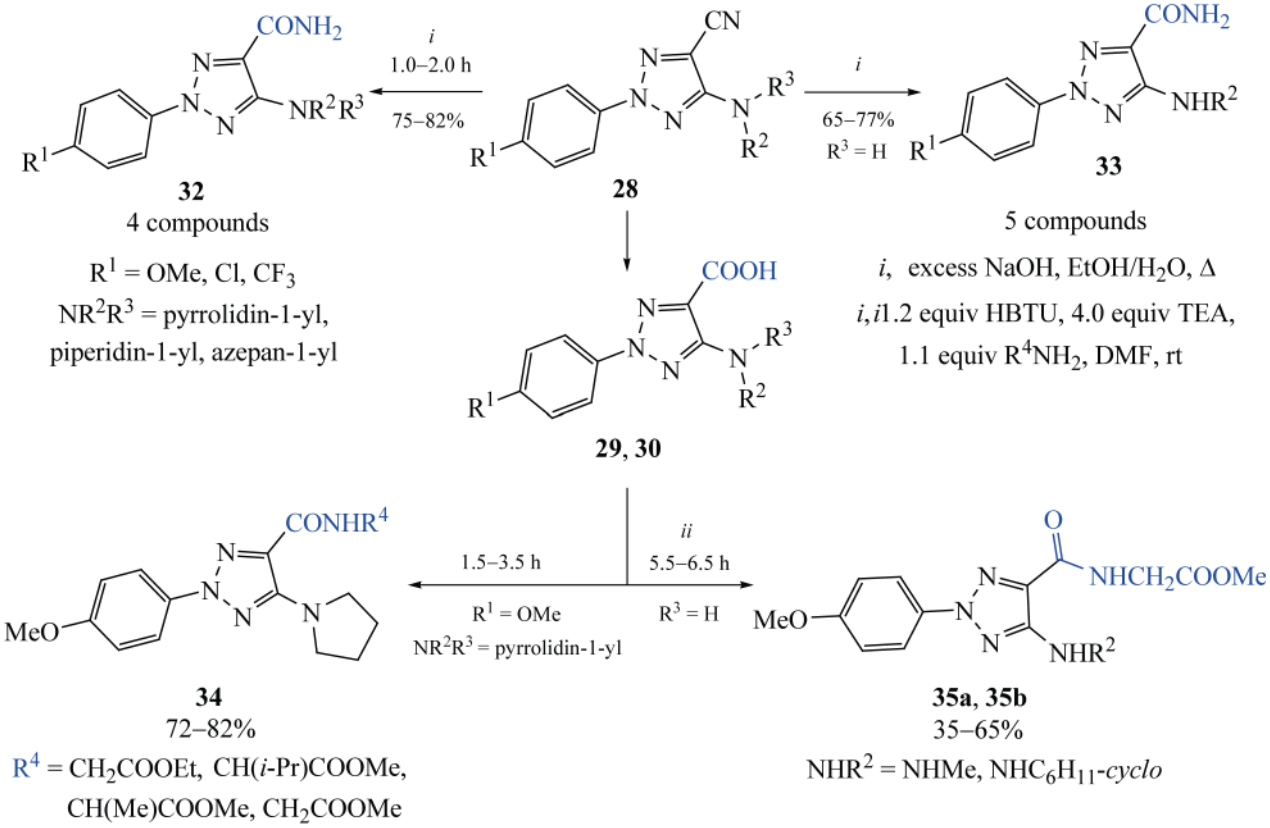


29a, **29b**

31a, **31b**

Ar = 4-$MeOC_6H_4$ (**a**), 4-ClC_6H_4 (**b**)

Scheme 13.12.



are neutral, stable, and have both acceptor and donor properties. Therefore, as a natural continuation of the search for new fluorescent sensors was the synthesis of new 2-aryl-1,2,3-triazole carboxamides **32–35** (Scheme 13.12) by the alkaline hydrolysis of 1,2,3-triazole-4-carbonitriles **28** or the acylation of amino acid esters [779].

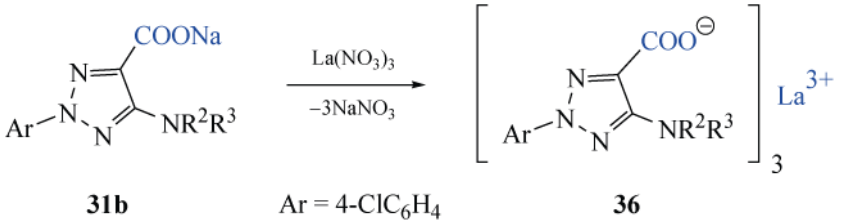
Comparison of the photophysical characteristics of 2-aryl-1,2,3-triazoles **32–35** and acids **29** and **30** showed that the properties of amides, especially those containing a secondary amino group (NHMe and NH C_6H_{11}-*cyclo*) at the C^5 atom of the triazole ring, differ from the properties of the corresponding acids. It should be noted that the fluorescence of 2-aryl-1,2,3-triazoles **32–35** enhances as DMSO and 1,4-dioxane are diluted with water, which is especially true of amides **33** and **35** (by 1.3–12.8 times). Using the dynamic light scattering method, it was found that compounds **32–35** form clearly detectable nanoparticles with a diameter of slightly more than 100 nm. Spectral studies revealed the ability of 2-aryl-1,2,3-triazoles **32–35** to selectively detect Hg^{2+} ions in a DMSO–water mixture (2/98). Thus, the synthesized fluorophores are new low-molecular-weight heterocyclic sensors with high selectivity, low detection limit, and ease of use. Filter paper strips

32a, R^1 = OMe, NR^2R^3 = pyrrolidin-1-yl

33a, R^1 = OMe, NHR^2 = NHMe

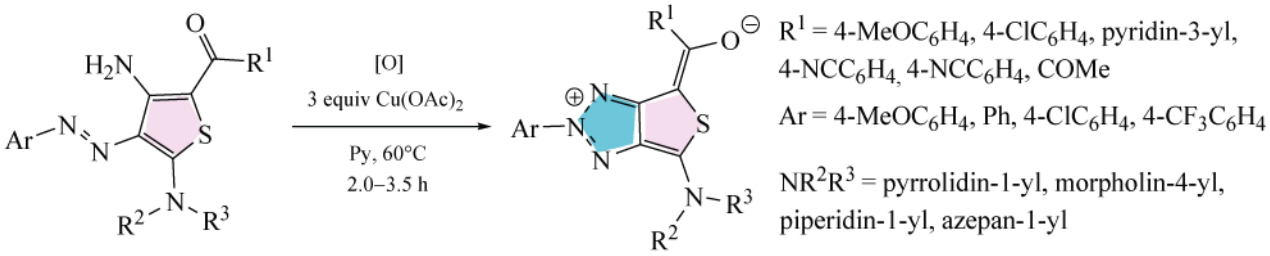
Fig. 13.1. Photographs of the test strips impregnated with a solution of 1,2,3-triazoles **32a** and **33a**, treated with aqueous solutions of metal ions, under irradiation with a 365-nm UV lamp.

Scheme 13.13.



31b Ar = 4-ClC_6H_4 **36**

Scheme 13.14.



37 **38** 30–51%

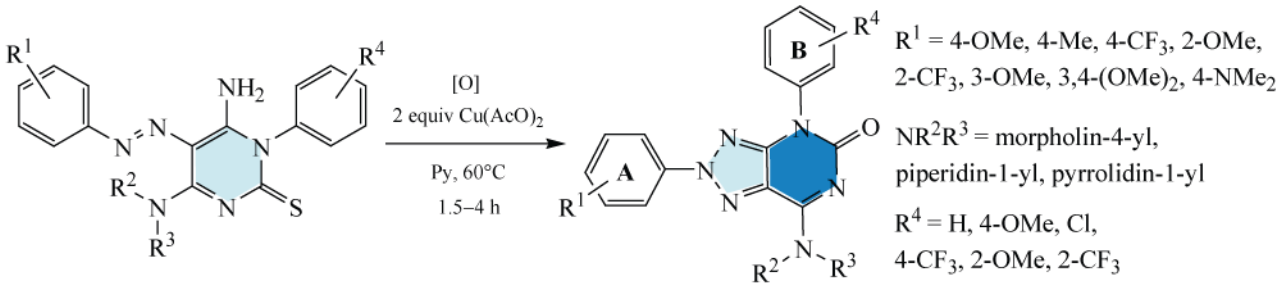
impregnated with a solution of 2-aryl-1,2,3-triazoles clearly demonstrated quenching of blue fluorescence in the presence of Hg^{2+} ions (Fig. 13.1).

We were the first to use the sodium salt of 1,2,3-triazole-4-carboxylic acid **31b** for preparing the La^{+} complex of the latter (Scheme 13.13) [780].

Quantum-chemical calculations (M06-2X/lanl2dz DFT) revealed a nearly symmetrical arrangement of ligands around the metal ion in complex **36**. The ability of complex **36**, acid **29b**, and sodium salt **31b** to trap free radicals, as well as the putative mechanisms of their antioxidant action were investigated on several model systems. A statistically significant effect as an OH• trap was found in all the studied compounds. However, in contrast to acid **29b** and sodium salt **31b**, complex **36** demonstrated a high activity at a lower concentration. The in vitro behavior of the complex provided evidence for interesting possibilities for its therapeutic application, both as an antioxidant and as a potential prooxidant.

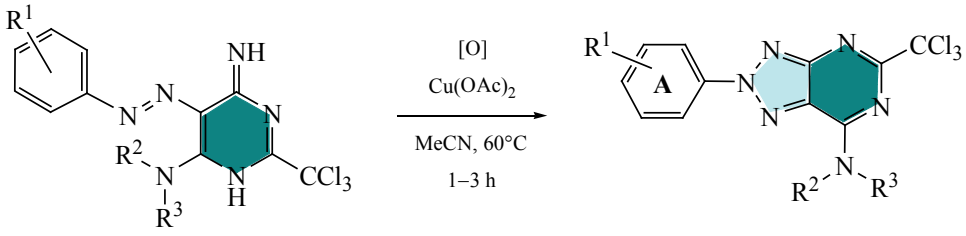
A series of mesoionic thieno[3,4-*d*]triazolium olates **38** were prepared by the oxidative cyclization of 3-amino-4-arylazothiophenes **37**, catalyzed by copper(II) salts (Scheme 13.14) [781]. The large set of possible resonance structures, long distance between the charge centers, and localization of the negative charge on the oxygen atom provide stabilization and facilitate formation of molecules **38**.

Scheme 13.15.



39 **40** 72–84% 12 compounds

Scheme 13.16.



41

42
78–83%
12 compounds

R^1 = 4-CF_3, 4-OMe, H, 4-CN, 4-NMe_2

NR^2R^3 = morpholin-4-yl, pyrrolidin-1-yl, piperidin-1-yl, NMe_2

A series of in vitro experiments with the use of confocal laser scanning microscopy showed that triazolium olates **38** successfully stain green monkey epithelial cells (*Vero*) and MIA PaCa-2 cells (human pancreatic cancer cell line) under laser irradiation at 405, 488, and 561 nm. Selective fluorophore accumulation in the endoplasmic reticulum (ER) should be mentioned (Scheme 13.15).

The emission maxima of compounds **40** are in the range 408–532 nm. The fluorescence characteristics strongly depend on the combination of substituents R^1 and R^4 and their positions in the aromatic rings **A** and **B**. Higher fluorescence quantum yields are observed for pyrimidin-5-ones **40** containing electron-donor substituents in the *para* position of ring **A** and electron-acceptor substituents in the *para* position of ring **B** (Φ = 23–39%).

It was shown by confocal laser scanning microscopy that pyrimidin-5-ones **40** easily penetrates into Hela cell membranes, fluoresce brightly upon excitation at λ_{ex} = 405 nm, and accumulates in lysosomes. The emission intensity after excitation is sufficient to obtain high-quality images.

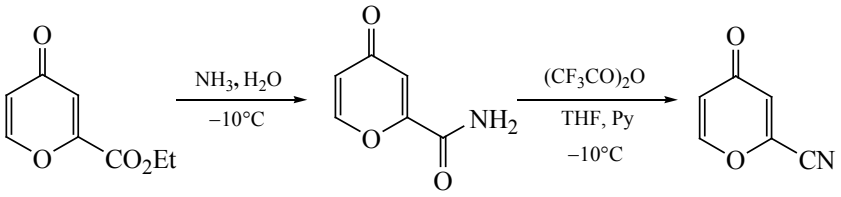
A series of 2-aryl-1,2,3-triazolo[4,5-*d*]pyrimidine derivatives **42** were obtained by oxidative cyclization of 5-arylazo-6-aminopyrimidines **41** (Scheme 13.16) [783].

The absorption spectra of triazolo[4,5-*d*]pyrimidines **42** contain long-wavelength absorption maxima at 337–397 and 331–402 nm in $CHCl_3$ and DMSO, respectively. The emission maxima are in the range of 407–495 nm in $CHCl_3$ and 414–545 nm in DMSO. The quantum yields of compounds **42** ranged from 1 to 11%. The Stokes shifts are quite large and increase significantly in DMSO (up to ~8008 cm^{-1}), indicating that triazolo[4,5-*d*]pyrimidines **42** are characterized by intramolecular charge transfer and positive solvatochromism. It was also found the fluorescence intensity of triazolo[4,5-*d*]pyrimidines **42** gradually increases.

Light-induced fluorescence enhancement was also observed on incubation with cell cultures. When excited by laser light at λ_{max} = 405 nm, the fluorescence intensity increased very rapidly and reached a maximum after 30 sec. An important result of the study is the detection of selective accumulation of triazolo[4,5-*d*]pyrimidines **42** in the cell membranes, as well as in the Golgi apparatus and ER.

The research conducted in 2018–2023 at the Department of the Technology of Organic Synthesis has provided evidence for the efficiency of new methods of synthesis and made it possible to discover interesting features of the chemical, biological, and optical physical properties of 1,2,3-triazoles. It was shown that 2-aryl-1,2,3-triazoles are new heterocyclic fluorophores with intense blue fluorescence. Their photophysical properties can be strongly modified by varying their substituents and functional groups, as well as by annulation to heterocycles. The synthesized mono- and bicyclic 2-aryl-1,2,3-triazoles easily penetrate living cells and selectively accumulate in the ER, Golgi apparatus, membranes, or lysosomes. All this allows us to conclude that 1,2,3-triazoles have good prospects for using in organic synthesis, ecology, biology, and medical research.

Scheme 14.1.



1 **2** **3**

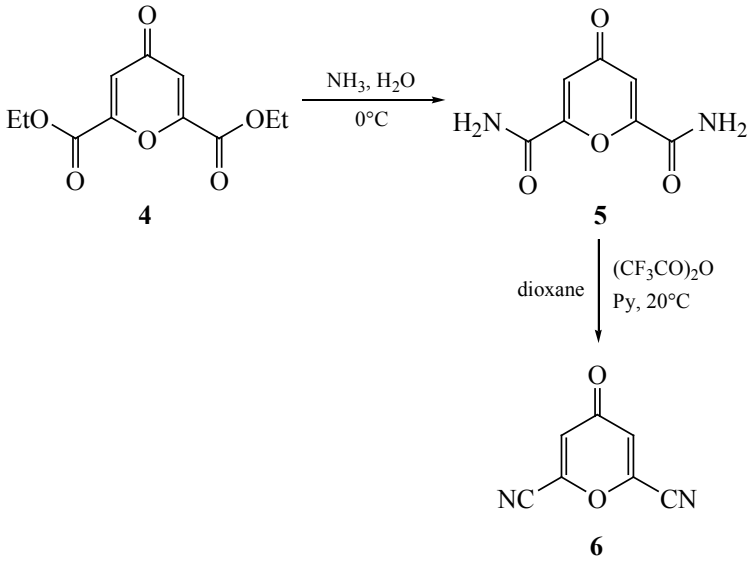
75% 48%

14. PROBLEMS OF REGIO- AND CHEMOSELECTIVITY IN THE CHEMISTRY OF COMANIC AND CHELIDONIC ACIDS IN RESEARCH AT THE INSTITUTE OF NATURAL SCIENCES AND MATHEMATICS OF URAL FEDERAL UNIVERSITY

Over the past years, 4-pyrone-2-carboxylic acids have become increasingly important due to their reactivity and use in medicinal chemistry [784, 785]. The progenitors of this class of oxygen-containing heterocycles are comanic (4-pyrone-2-carboxylic) and chelidonic (4-pyrone-2,6-dicarboxylic) acids, which are also the most accessible representatives of the 4-pyrone series [786]. Comanic acid is usually obtained from chelidonic acid, which in turn is synthesized by the condensation of acetone with diethyl oxalate [787]. Despite the fact that these molecules have been known for over 100 years, many of their properties, as well as methods of functionalization remain limitedly studied due to the high chemical activity of the pyrone ring. In this case, reactions can occur both by the ring itself and by the side substituent, which can not only activate the ring but also direct an attack to a certain position. The picture is complicated by the fact that the reaction can involve several electrophilic centers in the pyrone ring, but at the same time their close reactivity allows one to control the regioselectivity of the reaction by carefully selecting the reaction conditions.

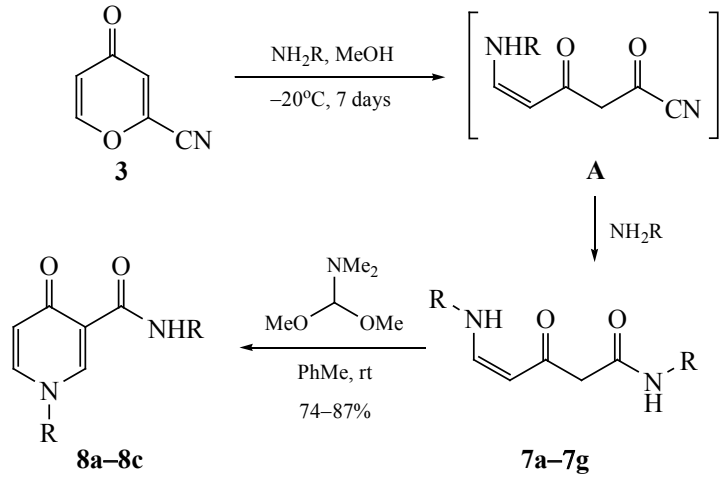
Derivatives of comanic and chelidonic acids are latent tetra- and pentacarbonyl compounds, and, therefore, they can serve as a convenient platform for constructing a wide variety of azaheterocyclic compounds. Nucleophilic reactions are usually an ANRORC process, which involves ring opening and formation of a polycarbonyl intermediate [788]. Cyclization of the latter can affect the entire process and usually depends on both the structure of the nucleophilic reagent and the reaction conditions [789].

Scheme 14.2.



4 **5** **6**

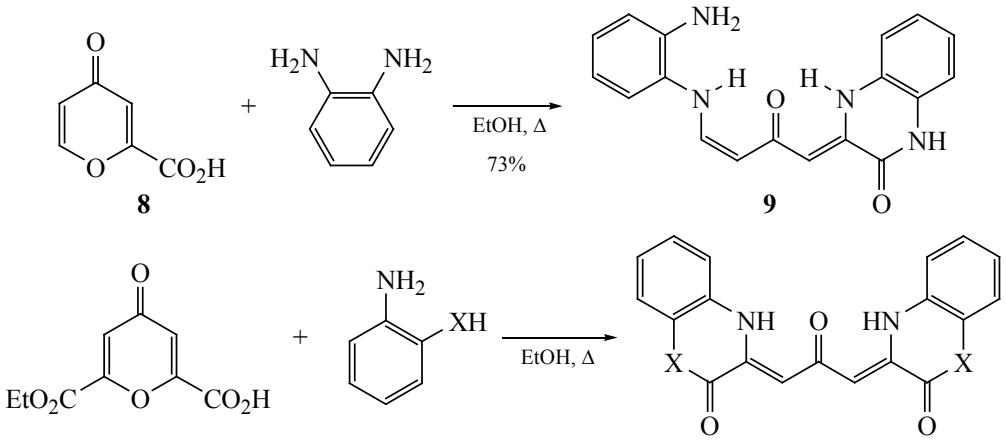
Scheme 14.3.



62–87%

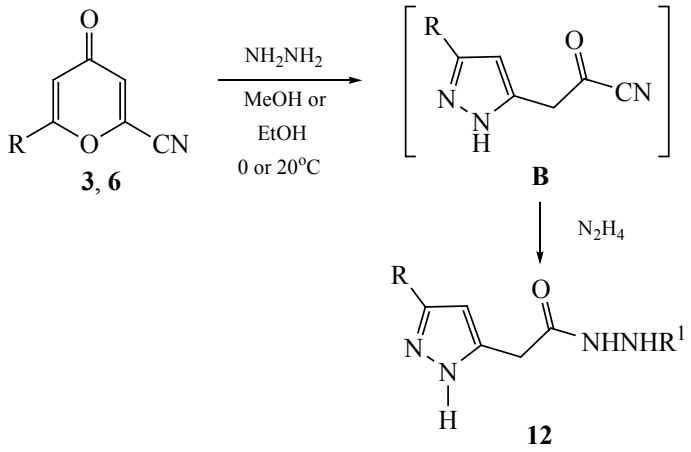
R = Bu, Bn, Oct, Ph, 2-MeC_6H_4, 4-$MeOC_6H_4$

Scheme 14.4.



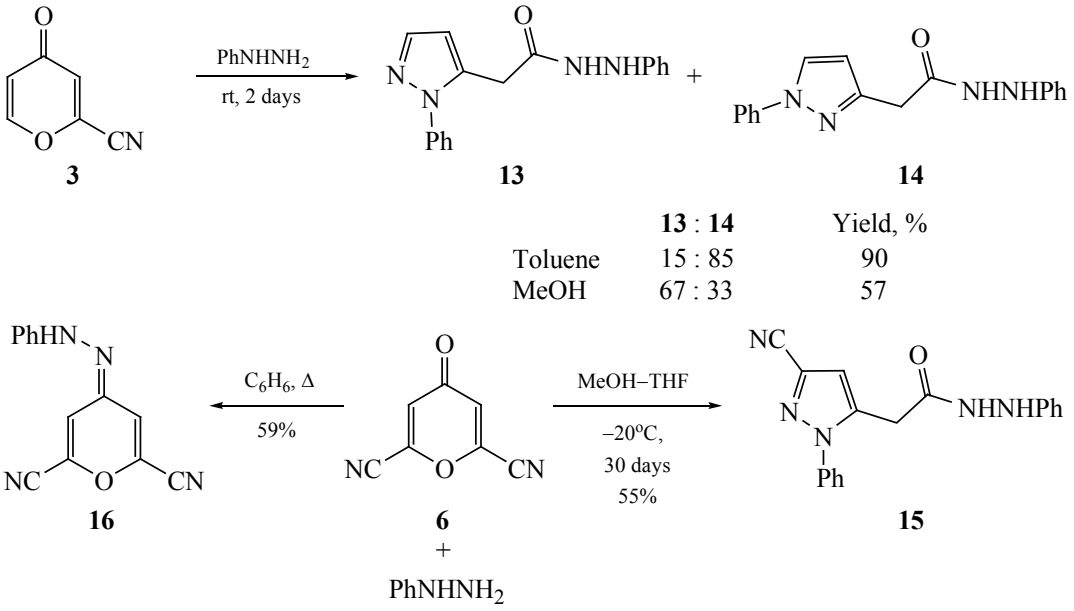
X = O (52%), NH

Scheme 14.5.



R = H (29%), CN (63%)

Scheme 14.6.



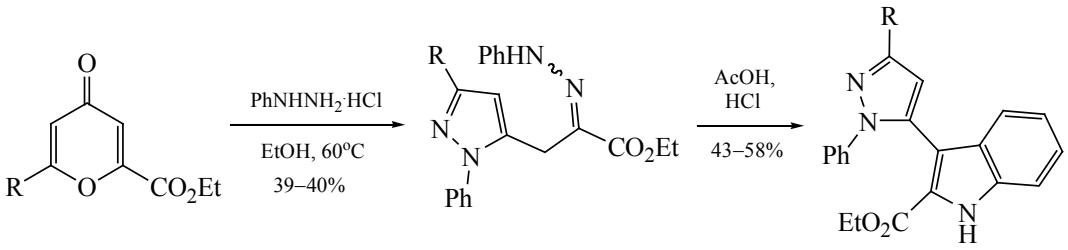
Comanic and chelidonic acids, as well as their esters, are well-studied compounds, but their amides and nitriles were unknown before our work. The difficulty in reacting the esters with ammonia consists in that that these reactions formed pyridones as more stable systems [788]. We found that treatment of ethyl comanate (**1**) with 20% aqueous ammonia allows selective synthesis of amide **2** in a yield of 75% (Scheme 14.1) [790, 791]. The subsequent stirring with trifluoroacetic anhydride and pyridine in THF under cooling gives results in the formation of 2-cyano-4-pyrone (**3**) in a yield of 48%.

Symmetrical 2,6-dicyano-4-pyrone (**6**) can be synthesized by the same procedure (Scheme 14.2) [787]. Treatment of diethyl chelidonate acid (**4**) with 20% aqueous ammonia at 0°C for 1 h gives chelidonic acid diamide (**5**) in 87% yield. The dehydration of the diamide to form dinitrile **6** (yield 71%) occurs under stirring with trifluoroacetic anhydride in dioxane at room temperature.

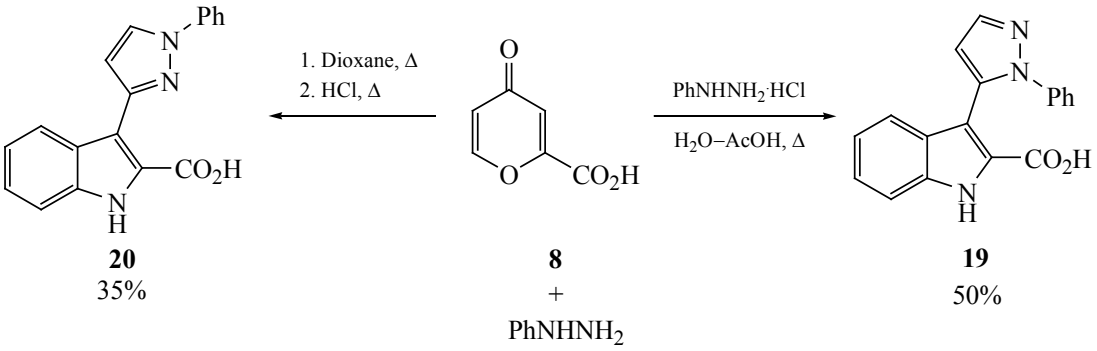
By contrast to 2,6-dicyano-4-pyrone (**6**), whose reactions with amines give complex product mixtures, 2-cyano-4-pyrone (**3**) reacts with primary amines in anhydrous methanol at –20°C for 1 week (Scheme 14.3) [790]. The reaction involves the initial attack on C^6 to form acyl cyanide **A** and the subsequent reaction of the latter with one more amine molecule, leading to carbamoylated enaminones **7a–7g**. Enaminones **7**, as polynucleophilic substrates, are capable of undergoing cyclization in the presence of DMA–DMF in dry toluene for 24 h at room temperature to give 4-pyridone-3-carboxamides **8a–8c** in yields of 74–87%.

4-Pyrone-2-carboxylic acids contain a pyruvic acid moiety and are therefore capable of entering in the Hinsberg reaction. We found that comanic acid (**8**)

Scheme 14.7.



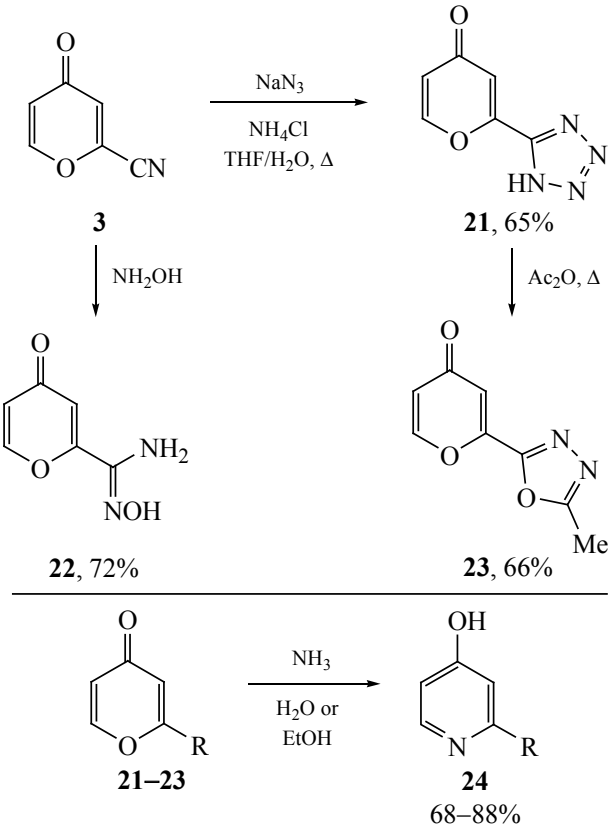
Scheme 14.8.



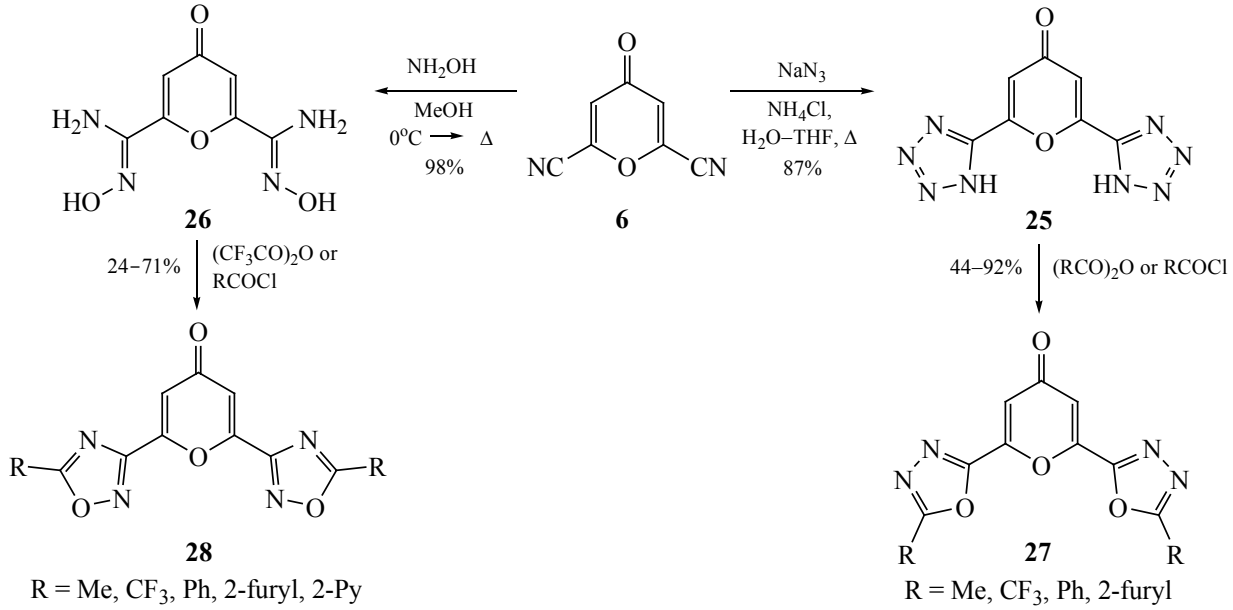
and monoethyl chelidonate (**10**) can react selectively with *o*-phenylenediamine to form functionalized quinoxalinones (Scheme 14.4) [792]. The reaction of acid **8** in ethanol under reflux proceeds via an attack on C^2 and C^6 to form aminovinyl ketone **9**, but the latter does not undergo cyclization upon heating. The reactions of ester **10** with *o*-aminophenol or *o*-phenylenediamine give rise to symmetrical structures **11** containing a benzoxazinone or a quinoxalinone moiety. Products **11** were not isolated pure and contained an admixture of 3-[1*H*-1,5-benzodiazepin-2(3*H*)-ylidenemethyl]quinoxalin-2(1*H*)-one (12%).

The reactions of comanic and chelidonic acid derivatives allowed the synthesis of substituted pyrazoles (Scheme 14.5) [790]. The reaction of 2-cyano-4-pyrone (**3**) with hydrazine proceeds via the substitution of the cyano group with the intermediate formation of 2-pyrazolyl acetyl cyanide **B** and provides pyrazolylacetic acid hydrazides **12** in yields of 29–63%. As with amines, the transformation of 2-cyano-

Scheme 14.9.



Scheme 14.10.



4-pyrones (**3**) involves the initial attack on C^6 atom to form a monosubstituted pyrazole.

The reactions of 2-cyano-4-pyrones **3** and **6** with phenylhydrazine were less selective (Scheme 14.6) [790], but their direction was highly dependent on the conditions, and this allowed regioselective syntheses. 2-Cyano-4-pyrone (**3**) reacts with phenylhydrazine to form a mixture of regioisomeric pyrazolylacetic acid hydrazides **13** and **14**. The reaction in toluene results in the preferential formation of product **14** through an attack on C^4 and C^2, while the attack on C^2 and C^6, leading to product **13**, is favored by methanol. In the case of 2,6-dicyano-4-pyrone (**6**), prolonged storage at –20°C yielded 3-cyano-*N*-phenylpyrazole **15** [787], and refluxing in benzene induces an attack on the C^4 atom, leading to phenylhydrazone **16**, which is a stable compound and does not undergo further transformations by reacting with phenylhydrazine.

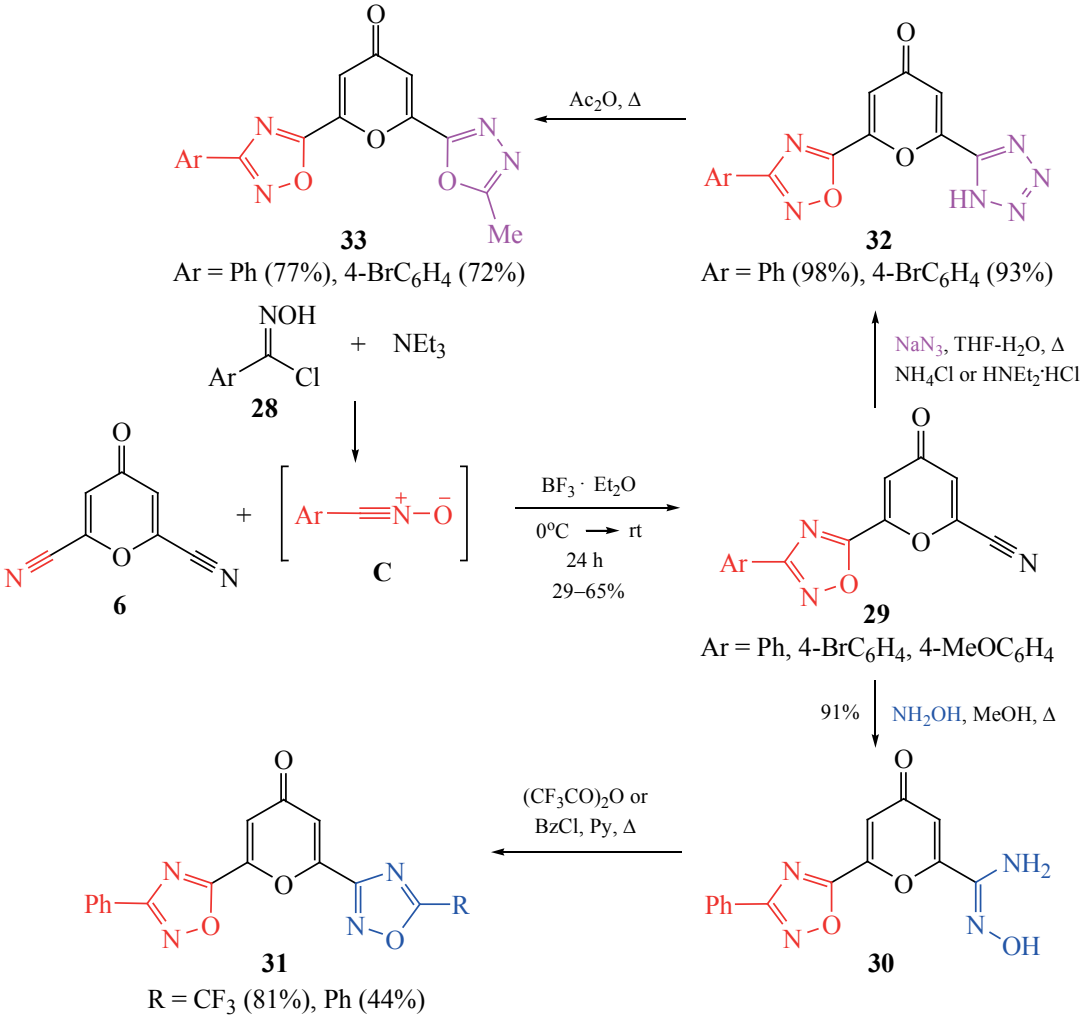
These transformations provide clear evidence for the regularity that aprotic solvents promote the reaction at position C^4, while protic solvents, at positions C^2 and C^6. Therewith, the most active substituent is always found in the side chain of the product, which may be associated with the formation of the most stable intermediates and the ease of ring opening.

The reaction of 4-pyrone-2-carboxylic acids with phenylhydrazine attracted out attention as a convenient strategy for constructing 3-pyrazolylindoles through the Knorr reaction to obtain pyrazoles and through the Fischer indolization (Scheme 14.7) [793]. The reactions of ethyl esters **1** and **4** with phenylhydrazine hydrochloride in ethanol proceeds selectively at the C^2 and C^6 atoms to form phenylpyrazoles **17**, which contains a pyruvic phenylhydrazone residue in position 5. It is important to note that we failed to react esters **1** and **4** in toluene because of their low reactivity. The subsequent refluxing of phenylhydrazones **17** in acetic acid in the presence of HCl gave pyrazolylindoles **18** in yields of 43–58%.

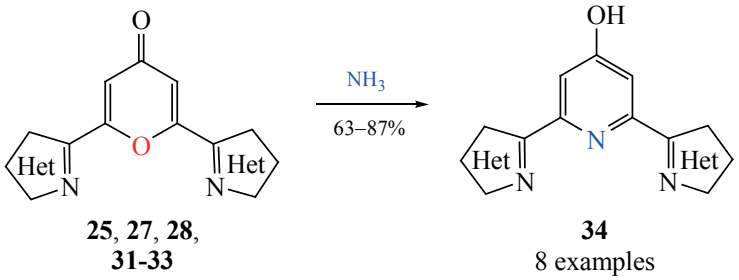
The introduction of a carboxyl group into the pyrone structure leads to an increase in reactivity. Of particular interest was the reaction of comanic acid (**8**) with phenylhydrazine, which allowed the regioselective one-pot synthesis of 3-pyrazolylindoles from 4-pyrones (Scheme 14.8) [792]. Heating acid **8** with phenylhydrazine hydrochloride in aqueous acetic acid yields 3-(*N*-phenylpyrazol-5-yl)indole-3-carboxylic acid **19**. The reaction with the basic phenylhydrazine in dioxane followed by treatment with HCl leads to 3-(*N*-phenylpyrazol-3-yl)indole-2-carboxylic acid **20**.

Of particular interest were the selective transformations of 2-cyano-4-pyrones at the side cyano group, since they can result in the formation of difficult-to-access hetaryl-substituted 4-pyrones and pyridines. 2-Cyano-4-pyrones are hidden acyl cyanides, which produces additional activation of

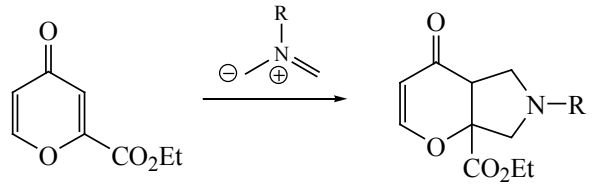
Scheme 14.11.



Scheme 14.12.



Scheme 14.13.



the side substituent. It was found that the reaction of pyrone **3** with sodium azide and hydroxylamine occurs exclusively at the cyano group to form 2-tetrazolyl-4-pyrone **21** and amidoxime **22**, respectively (Scheme 14.9) [791]. The selectivity of these transformations is presumably explained by a specific coordination of the nucleophile with the nitrile nitrogen atom. 2-Tetrazolyl-4-pyrone (**21**) can be acylated with acetic anhydride to form 2-(1,3,4-oxadiazolyl)-4-pyrone **23**. The reactions of 4-pyrones **21**–**23** with ammonia afford 4-hydroxypyridines **24** in yields of 68–88%.

2,6-Dicyano-4-pyrone (**6**), in turn, provides access can be provided to symmetrical and asymmetrical heterocyclic assemblies (Scheme 14.10) [787]. Both cyano groups can be functionalized by the previously discovered selective reactions with hydroxylamine and sodium azide to obtain bistetrazolyl derivative **25** and bisamidoxime **26** in high yields. The subsequent acylation with acid chlorides or anhydrides gives heterocyclic triads: 2,6-bis(1,3,4-oxodiazol-2-yl)-4-pyrones **27** and 2,6-bis(1,2,4-oxodiazol-2-yl)-4-pyrones **28**.

The reactions of 2,6-dicyano-4-pyrone (**6**) with nitrile oxides, generated in situ by reacting imidoyl chlorides with triethylamine, in the absence of boron fluoride etherate led to heterocyclization at only one cyano group (Scheme 14.11) [787], forming 6-(1,2,4-oxadiazol-5-yl)-2-cyano-4-pyrones **29** in yields of 29–65%. The intact cyano group was involved in selective reactions with hydroxylamine or sodium azide by the developed scheme followed by acylation to obtain heterocyclic assemblies **31** and **33**. It is interesting to note that when reacting 6-(1,2,4-oxadiazol-5-yl)-2-cyano-4-pyrone **29** containing a *para*-bromophenyl substituent with sodium azide, only replacing ammonium chloride by diethylamine hydrochloride made it possible to synthesize the corresponding 2-tetrazolyl-4-pyrone **32**.

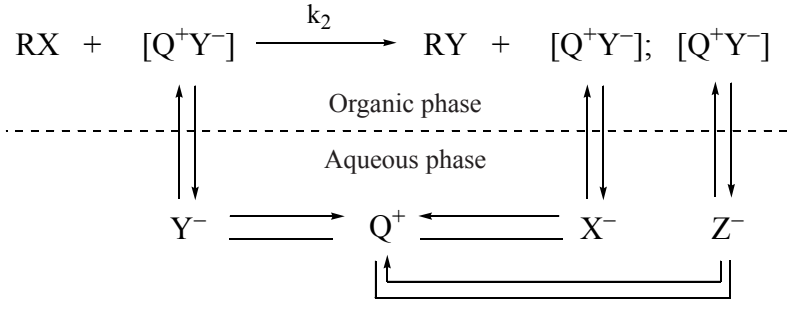
2,6-Bis(hetaryl)-4-pyrones can be converted to 2,6-bishetaryl-4-hydroxy-pyridines by the selective reaction of 4-pyrones with ammonia (Scheme 14.12) [787]. The most active was 2,6-bis(tetrazolyl)-4-pyrone (**25**), which reacted in aqueous ammonia at room temperature for 2 days. The other pyridines **34** were synthesized by heating the corresponding pyrones under more severe conditions in 15% ammonia in ethanol for 8 h at 100°C in an autoclave.

The reactions of 4-pyrones with 1,3-dipoles still remain poorly studied [794]. Of the series of comanic and chelidonic acid derivatives, only comanic acid esters entered into cycloaddition reactions with azomethine ylides to form pyrano[2,3-*c*]pyrrolidines **35** in yields of 55–63% (Scheme 14.13) [795]. Azomethine ylides were generated by refluxing in benzene with sarcosine and paraformaldehyde, as well as by stirring with *N*-benzyl-1-methoxy-*N*-[(trimethylsilyl)methyl]methanamine in CH_2Cl_2 in the absence of CF_3CO_2H. The attack occurred predominantly at the double bond activated by the ester substituent.

Thus, it was shown that derivatives of comanic and chelidonic acids are convenient substrates for the regio- and chemoselective synthesis of azaheterocycles, as well as functionalized 4-pyrones. The main transformations are usually accomplished by reactions with N-nucleophiles, but more and more attention has begun to be paid to cycloaddition reactions. The most active proved to be nitriles derived from 4-pyrone-2-carboxylic acids: depending on the nature of the nucleophile, they react with substitution of the cyano group or selectively by the cyano group, thereby determining a wide range of resulting structures.

The review was written with the financial support of the Russian Science Foundation (project no. 22-73-10236).

Scheme 15.1.



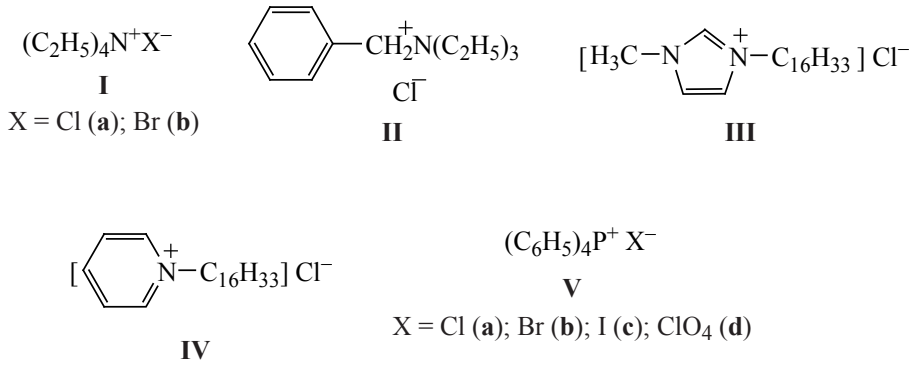


Fig. 15.1. Phase-transfer catalysts **I–V**.

15. MAIN TRENDS OF DEVELOPMENT AND RESULTS OF RESEARCH IN THE FIELD OF ORGANIC CHEMISTRY AT DONETSK UNIVERSITY

For a long time (1976–2009), the department was headed by L.M. Litvinenko's student, **Dr. Chem. Sci., Prof. N.M. Oleynik**, the author of a series of well-known monographs on homogeneous organocatalysis.

Research on the relationships between the structure and reactivity of organic compounds was also conducted at the Department of Biochemistry of Donetsk University within the framework of the scientific school headed by **Dr. Chem. Sci., Prof. N.M., Academician of the National Academy of Sciences of Ukraine A.F. Popov**, another student of Acad. L.M. Litvinenko.

The topics of research currently being carried out in the field of organic chemistry (already at the united Department of Biochemistry and Organic Chemistry) are still closely related to the kinetics and catalysis of organic reactions. There are two main lines of research. The first is the the kinetics and mechanism of catalytic and enzymatic phase-transfer catalysts (the research was initiated by **Cand. Chem. Sci., Assoc. Prof. V.V. Kosmynin** and is continued today by **Cand. Chem. Sci., Assoc. Prof., Head of Department O.V. Baranova and Researcher V.S. Doroshkevich**). The object for study is acyl transfer reactions (hydrolysis, aminolysis) in two-phase liquid/liquid systems under phase-transfer conditions with a branched catalytic cycle. These reactions can be described by Scheme 15.1.

In the above scheme, Q^+ is the phase-transfer catalyst; RX, substrate; Y^-, nucleophilic reagent; Z^-, catalyst counterion; and M^+, inorganic salt or base cation.

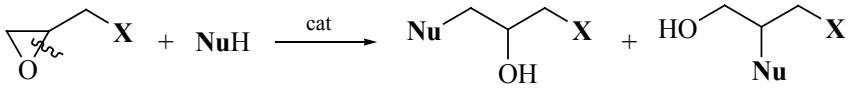
The radical difference between the phase-transfer catalytic process presented in Scheme 15.1 and the Starks model is that the formation of the reaction product is associated with the development of new extraction flows. The kinetic patterns of such reactions will be determined not only by the initial distribution of the catalyst, but also by the depth of substrate conversion.

Using the example of model aminolysis and hydrolysis reactions of activated amino acid esters, the reaction mechanism, specifically the process topology, rate-limiting stage, and efficiency of onium salts as a phase-transfer catalyst, was studied. In [796], the kinetics of the aminolysis of 4-nitrophenyl *N*-benzyloxycarbonylglycinate in the two-phase 1-butanol/glycine buffer (pH 10.4) system was studied at a phase volume ratio of 1 : 1 in the absence of phase-transfer catalysts **I–V** (Fig. 15.1).

It was established that the highest activity is characteristic of imidazolium, benzimidazolium, and tetraphenylphosphonium salts. The dependence of the apparent rate constants of the studied aminolysis reaction in a two-phase system on the nature of the anion decreases in the order $Cl^- > Br^- > I^- > ClO_4^-$, in parallel with decreasing degree of transfer of the active form of the phase-transfer catalyst Q^+Y^- into the organic phase.

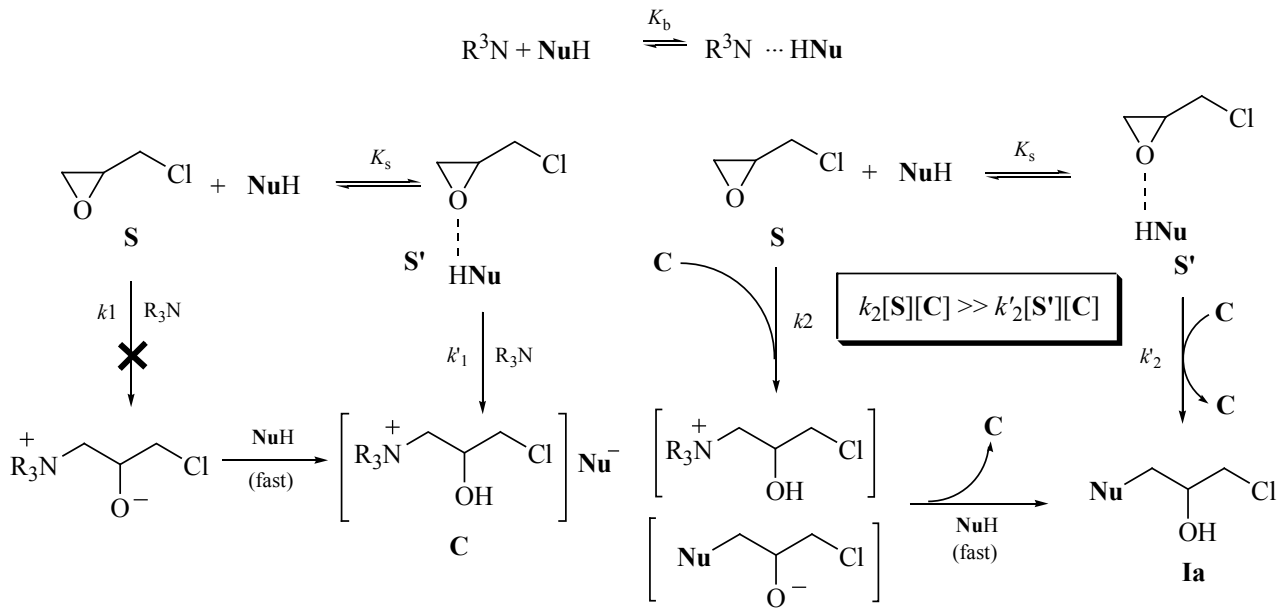
Over the past years, the department has been developing an approach to quantitative characterization of the activity of phase-transfer catalysts in acyl-transfer reactions in two-phase liquid/liquid systems. The experimental data on the catalytic activity of the studied salts in the aminolysis reaction of 4-nitrophenyl *N*-benzyloxycarbonylglycinate in the two-phase chloroform/glycine buffer (pH 10) system were compared with the calculated standard enthalpies of formation of the ion pairs of the onium salt cations with the

Scheme 15.2.



Scheme 15.3.

Scheme 15.4.

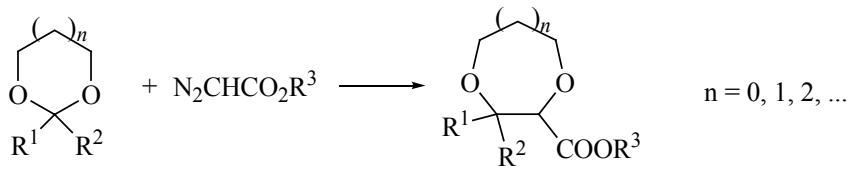


nucleophile (glycinate anion), which are the active forms of the phase-transfer catalysts, because it is their interaction with the substrate in the organic phase that leads to the formation of the target product. It was found that the catalytic efficiency of onium salts in phase-transfer aminolysis processes in a two-phase liquid/liquid system is determined by an energy factor: the lower the standard enthalpy of formation of the active form of the catalyst, the higher the rate of the process under study. This value can be used as a quantitative measure of the efficiency of onium salts as phase-transfer catalysts in the extraction mechanism of phase-transfer catalysis.

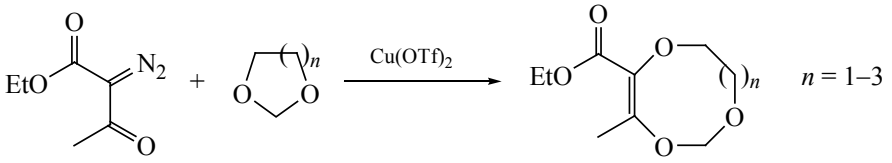
The second line of research is focused on the mechanism of nucleophilic epoxide ring opening (the research was initiated by **Dr. Chem. Sci., Prof. E.N. Shved** and is continued today by **Cand. Chem. Sci., Assoc. Prof. S.G. Bakhtin** and **Senior Lecturer M.A. Sinelnikova**). It should be noted that nucleophilic substitution at the carbon atom of the oxirane ring under the action of protic nucleophiles (NuH) is one of the most important organic reactions widely used in industry and fine organic synthesis (Scheme 15.2).

Tertiary amines (R_3N) and their salts are among the most effective catalysts for this reaction. For example, the half-life (Φ 1/2) of the noncatalytic reaction (80°C)

Scheme 16.1.



Scheme 16.2.



in Scheme 15.2 (X = Cl and Nu = AcO) and under Et_3N catalysis conditions (60°C; C_{cat} = 0.005 M) is ≈ 11 days and 2.5 h, respectively. In addition, a combination of the experimental data [798] and the results of quantum-chemical calculations [799] provided evidence to show that, in the absence of tertiary amines or tetraalkylammonium salts, the ring opening reaction proceeds with high regioselectivity.

Undoubtedly, the role of R_3N is to generate reactive Nu^- species, but there is no consensus regarding the detailed mechanism of this generation. Most often, the mechanism of tertiary amine catalysis is discussed in terms of general base catalysis (Scheme 15.3).

Less popular is the alternative idea, where the amine acts not as a base but as a nucleophile and forms **Nu**$^-$ via the initial quaternization of the amine, involving both oxirane and **Nu**H.

To answer the controversial question about the role of amines (base or nucleophile), systems, where the basic and nucleophilic properties of R_3N change in opposite directions, were developed and studied [800–802]; furthermore, ^1H and ^{13}C NMR [803, 804] and UV spectroscopy [804–797] were used to gain detailed insight in nucleophilic–electrophilic and acid–base interactions in the oxirane – proton donor – amine reaction systems (Scheme 15.4).

Recently, the staff members of the department published a work [798] concerning the kinetic laws of stereoselective catalysis of nucleophilic substitution with the participation of chiral amines.

16. WORKS IN ORGANIC CHEMISTRY AT THE DEPARTMENT OF GENERAL, ANALYTICAL AND APPLIED CHEMISTRY OF UFA STATE OIL TECHNICAL UNIVERSITY

In the early 70s, D.L. Rakhmankulov, a PhD graduate of the Gubkin Moscow Institute of Oil and Gas Industry [now Gubkin Russian State University of Oil and Gas (National Research University)] and a student of the Academician of the Academy of Sciences of Armenian SSR V.I. Isagulants, established at the Department of General Chemistry of Ufa Oil Institute [now Ufa State Oil Technical University (USOTU)] a scientific school on the chemistry and technology of cyclic acetals and related compounds.

Currently, the head of the Department of General Chemistry is Corresponding Member of the Academy of Sciences of the Republic of Bashkortostan S.S. Zlotskii. The main results obtained in 2018–2023 have been discussed and systematized in a number of reviews [809–811]. During this period, the scientific research of the department is focused on the following areas.

16.1. Catalytic reactions of cyclic acetals with diazo compounds

The catalytic reaction of cyclic acetals with diazocarbonyl compounds is one of the convenient and selective methods of their functionalization. It was found that 2-mono- and 2,2-disubstituted 1,3-dioxolanes have the highest activity in the ring expansion reaction [812–814]. In particular, the corresponding 2,3-disubstituted 1,4-dioxane with a diequatorial arrangement of substituents is formed from 2-phenyl-1,3-dioxolane in a yield of 80% (Scheme 16.1).

α-Diazo-β-ketoesters react with cyclic acetals (Scheme 16.2) to form polyoxygenated 8-, 9-, and 10-membered heterocycles in yields of up to 90%.

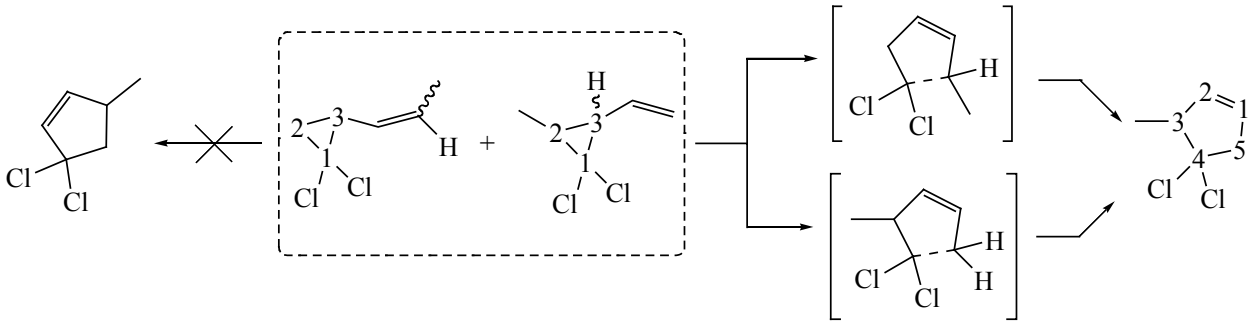
It was found that the presence and size of substituents in the ring strongly affect the regioselectivity of the reaction.

16.2. Heterogeneous catalytic transformations of cyclic acetals and gem-dichlorocyclopropanes

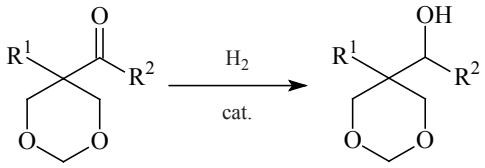
The thermocatalytic isomerization of vinyl-*gem*-dichlorocyclopropanes in the presence of SAPO-34 zeolites at 200–250°C for 1–3 h, leading to 4,4-dichloro-3-methylcyclopentene in a yield of more than 90%) was studied (Scheme 16.3).

It was established that the cyclopentene structure is formed by three-membered olefin ring opening at the

Scheme 16.3.



Scheme 16.4.



R^1, R^2 = alkyl, aryl

C^1–C^3 bond and gives 4,4-dichlorocyclopentenes as the main products.

A method for the synthesis of secondary 1,3-dioxacycloalkanoic alcohols (Scheme 16.4) by the reduction of the keto group in 5-acyl-1,3-dioxanes with metal hydrides or the hydrogenation on Pd-containing catalysts was developed [815]. The best results were obtained with Pd/C (conversion 85–90%, selectivity 92–98%). The conversion of ketones depends on the substituents at the carbonyl group and in the 5 position of the 1,3-dioxane ring.

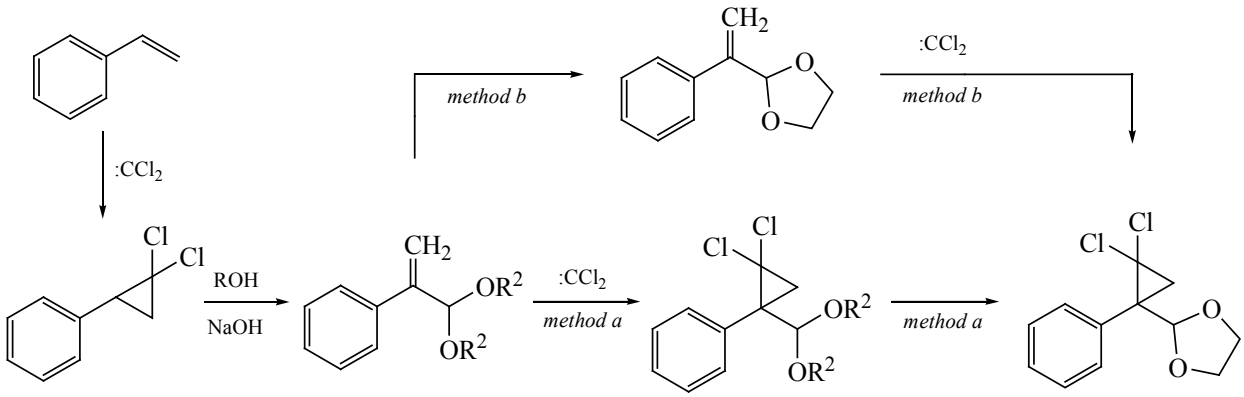
The alcoholysis of phenyl-*gem*-dichlorocyclopropane in the presence of anion exchangers provided phenylacrolein acetals (Scheme 16.5).

The highest yields of these products were obtained with ethanol and butanol (80–87%) and decreased with increasing molecular weight of the alcohol, as well as in going to iso and secondary alcohols. The resulting linear acetals were used to obtain the corresponding *gem*-dichlorocyclopropanes in yields of 72–80% [816, 817].

The heterogeneous catalytic condensation of ammonia and aniline with 2,2-dimethyl-4-oxymethyl-1,3-dioxolane (solketal) in the temperature range of 350–500°C gave the corresponding pyridine derivatives (Scheme 16.6).

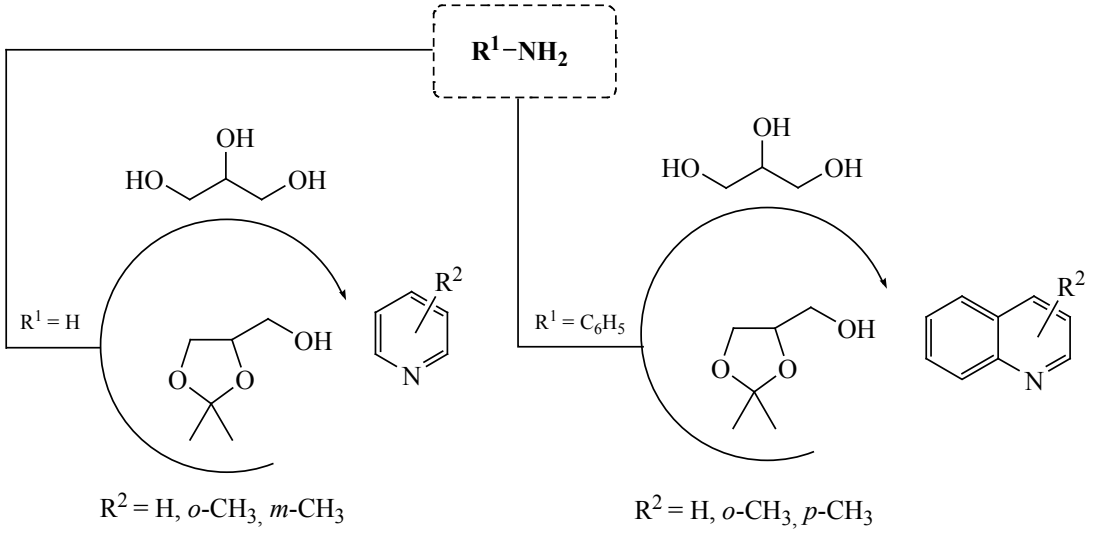
The results of above-mentioned work show that solketal can successfully replace glycerol. The best yields of the target *N*-heterocycles (up to 75%) were

Scheme 16.5.

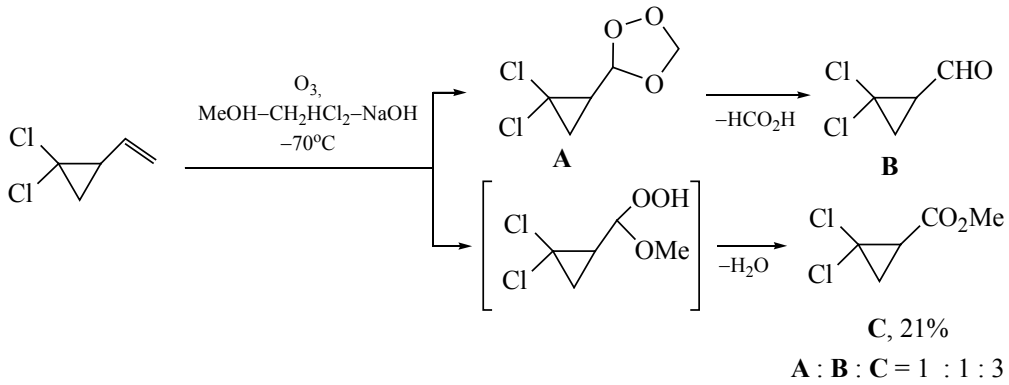


R = Et, Bu

Scheme 16.6.



Scheme 16.7.



obtained with a solketal:glycerol mixture with a weight ratio of 2 : 1. These studies [818] were carried out jointly with the research group of **Dr. Chem. Sci., Senior Researcher N.G. Grigorieva** (Laboratory of Catalyst Preparation, Institute of Petroleum Science, Ufa Federal Research Center, Russian Academy of Sciences).

16.3. Oxidation and low-temperature ozonolysis

The low-temperature ozonolysis of vinyl-*gem*-dichlorocyclopropanes forms aldehydes or esters, depending on the conditions of treatment of intermediate peroxide compounds, (Scheme 16.7).

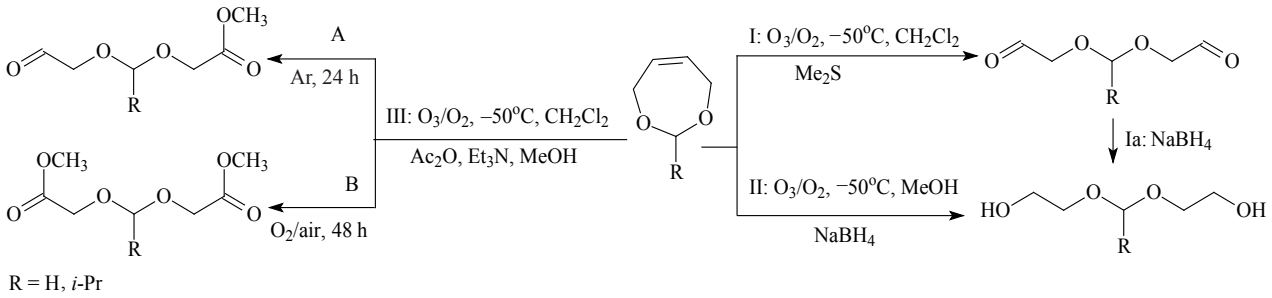
The low-temperature ozonolysis of 4,7-dihydro-1,3-dioxepine and 2-isopropyl-4,7-dihydro-1,3-dioxepine (Scheme 16.8) yielded dialdehydes, diols, or diesters, depending on the method of treatment of intermediate products. In all cases, the acetal fragment was not affected, and the 1,3-arrangement of the oxygen atoms was retained in the target products.

The ozonolysis of 1,3-dioxacycloalkanes with an exocyclic double bond (2,2-dialkyl-4-methylene-1,3-dioxolane), too, involved no acetal ring opening and gave 4-keto derivatives (yields 80–85%) as the main products (Scheme 16.9). Similar compounds were previously obtained in low yields by the condensation of oxyacetic acid with the corresponding ketones.

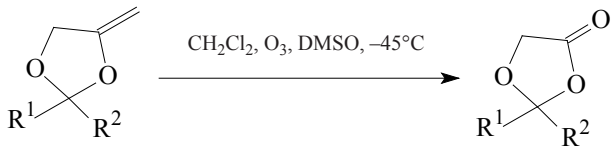
These studies [819] were performed in collaboration with the laboratory headed by **Dr. Chem. Soc., Prof. G.Yu. Ishmuratov** (Laboratory of Insect Bioregulators, Ufa Institute of Chemistry, Ufa Research Center, Russian Academy of Sciences).

Comprehensive studies over the period 2018–2023 were aimed at identifying potential applications of the synthesized compounds. Lead compounds that proved to be effective inhibitors of acid corrosion, additives to oils and polymers, herbicides, and drugs were found [820–822].

Scheme 16.8.



Scheme 16.9.



$R^1 + R^1 = (C_2H_5)_5$, $R^1 = CH_3$, $R^2 = i\text{-}C_4H_9$

Some scientific results were obtained as part of the implementation of the state assignment from the Ministry of Education and Science of the Russian Federation in the field of scientific activity (no. FEUR-2022–0007 "Petrochemical reagents, oils, and materials for thermal power engineering").

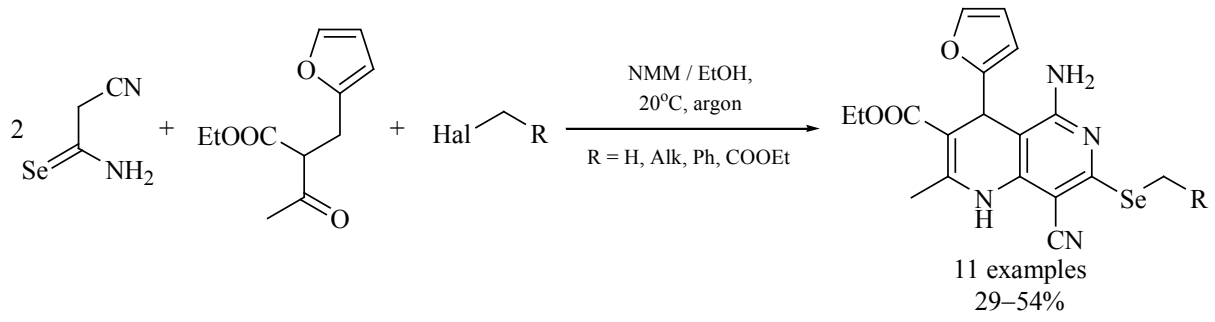
17. DEPARTMENT OF CHEMISTRY AND BIOCHEMISTRY OF LUGANSK STATE PEDAGOGICAL UNIVERSITY

The Department of Chemistry has been functioning in the structure of the university practically since its foundation. Until 1938, it was the only center for training chemistry teachers for the entire Donbass. The first teachers at the department were **I.A. Voitenko** (head of the department from 1923 to 1957) and his spouse **O.P. Alekseeva** (head of the department from 1957 to 1959), who was a student of **Acad. A.E. Favorskii** and worked at his laboratory for some time. Graduates of the department are the prominent Russian chemists **Doct. Chem. Sci., Prof. A.M. Shestopalov, Doct. Chem. Sci., Prof. L.A. Rodinovskaya, Doct. Chem. Sci., Prof. S.G. Krivokolisko, and Doct. Chem. Sci., Assoc. Prof. V.V. Dotsenko**.

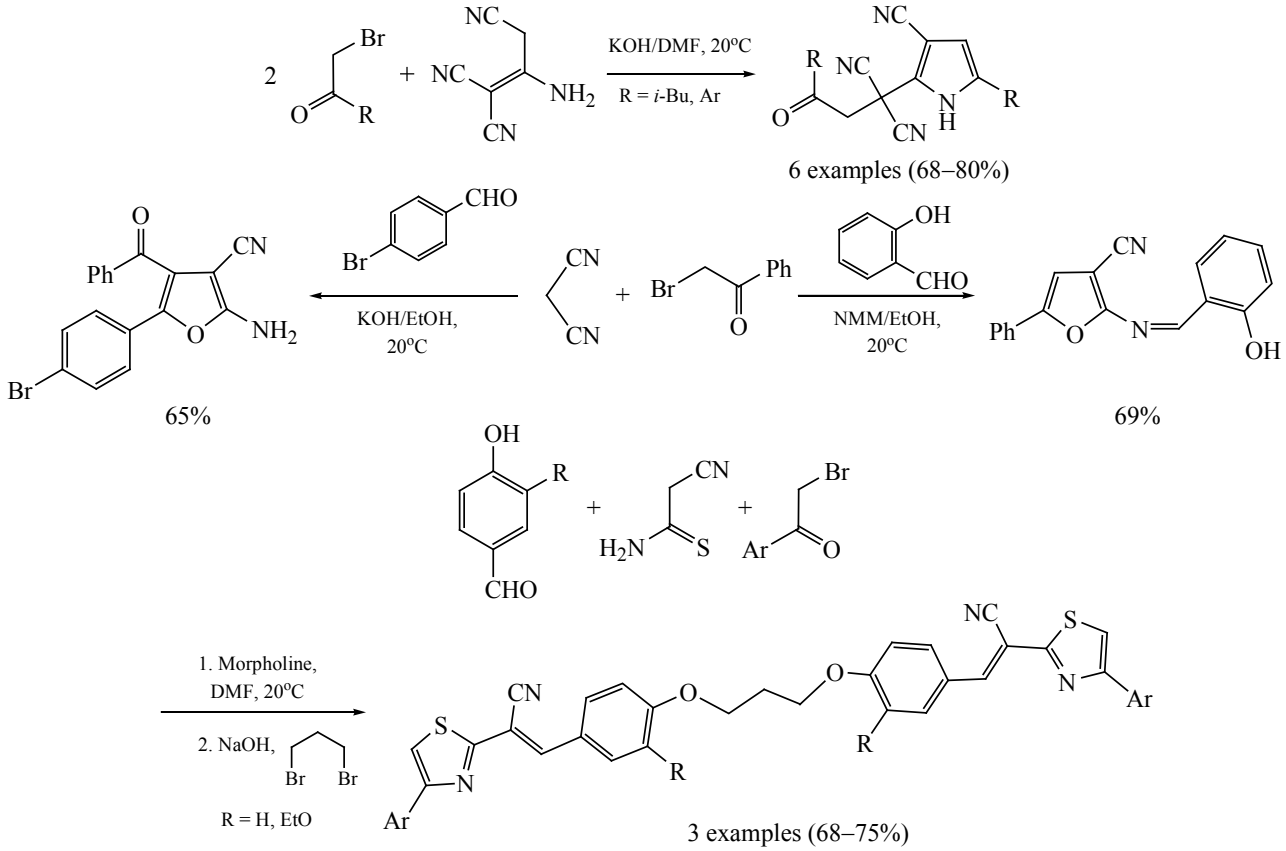
The modern history of the department can be traced back to 1974, when it was headed by **Doct. Chem. Sci., Prof. Yu.A. Sharanin**. The organizational talent allowed him to develop active research on the synthesis of heterocyclic compounds [823–825]. From 1994 to the present, the Department of Chemistry and Biochemistry has been successfully headed by his student, **Doct. Chem. Sci., Prof. V.D. Dyachenko**. His main scientific interests are focused on the methods of synthesis of selenium-containing heterocycles [826–830]. As a result, a previously unknown multicomponent condensation leading to substituted 7-alkylseleno-1,4-dihydro-1,6-naphthyridines was discovered (Scheme 17.1) [831].

Since 2000, the scientific directions actively developed by the department include the synthesis of

Scheme 17.1.



Scheme 17.2.

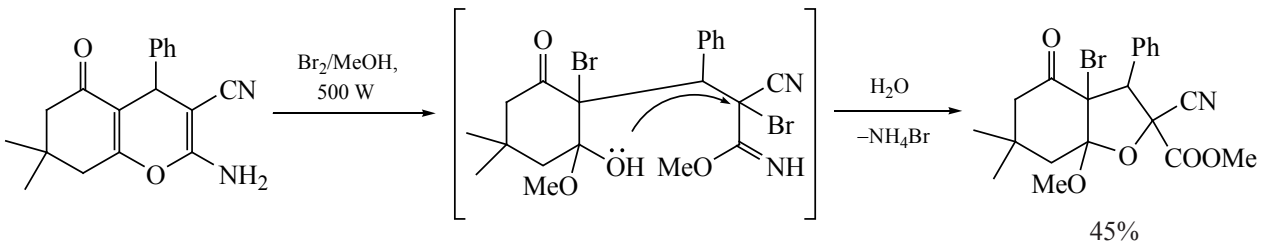


carbo- and heterocycles based on activated olefins [832–834] and the synthesis of biologically active compounds based on functionalized CH-acids [835–838]. New synthetic approaches to important 5-membered natural heterocycles, such as pyrrole, furan [839, 840], and thiazole have been discovered (Scheme 17.2) [841–843].

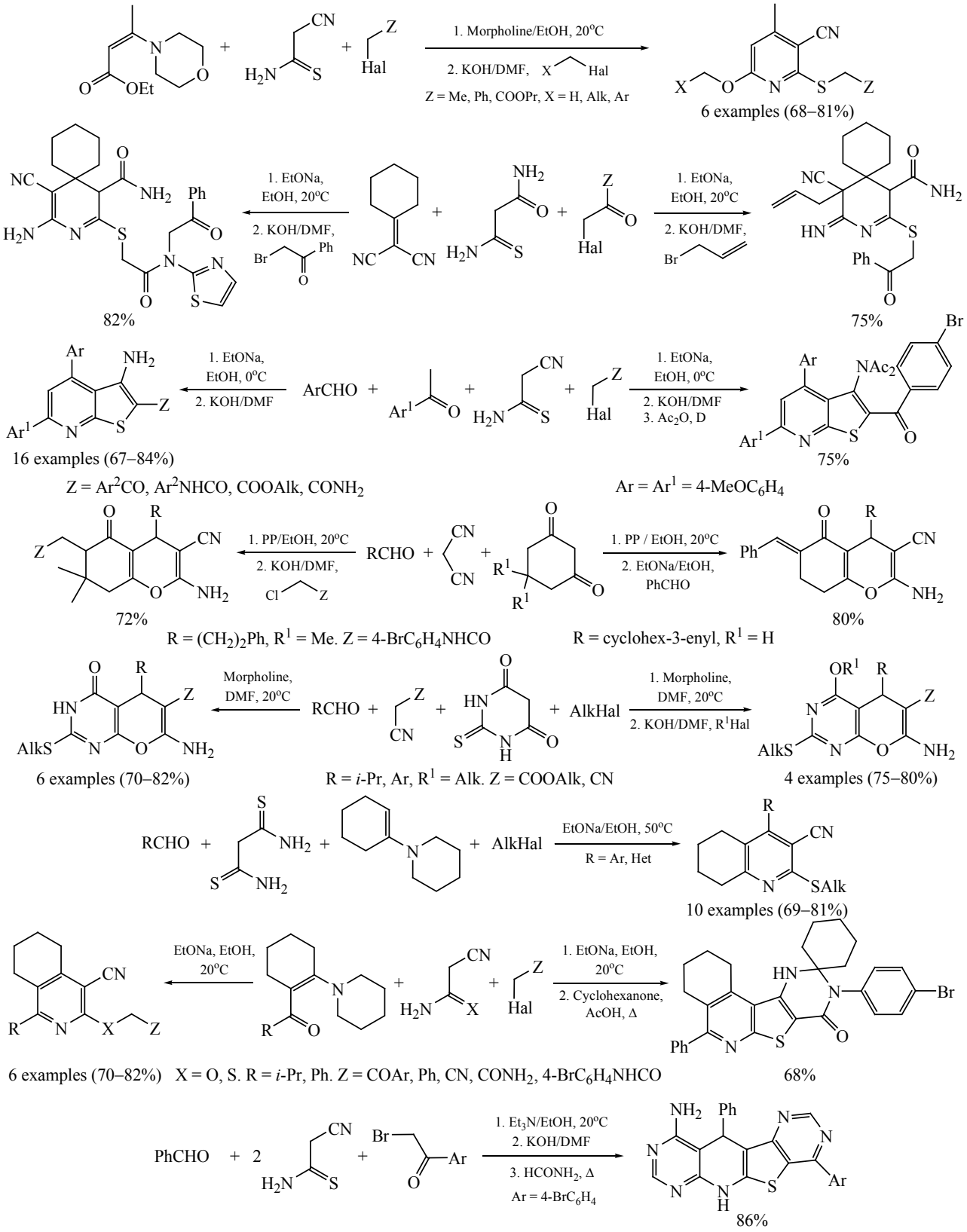
Special attention is also paid to such basic problems of synthetic organic chemistry as recyclization [844, 845] and rearrangement [846–849]. A literature review devoted to [3,3]-sigmatropic rearrangements has been published [850], and a previously unknown recyclization of the pyran ring into a furan ring has been discovered (Scheme 17.3) [851].

Currently, the main focus of research is on the rapidly developing multicomponent reactions (MCRs) initiated by Knoevenagel, Michael, and Stork reactions, as well as nucleophilic vinyl substitution and alkylation reactions. New derivatives of nicotinic acid [852–863], substituted spiropyridines [864, 865] and thieno[2,3-*b*]pyridines [866–870], functionalized benzo[*b*]pyrans [871, 872], and pyrano[2,3-*d*]pyrimidines [873], partially hydrogenated quinolines [874–877] and isoquinolines [878, 879], as well as fused pyrimidines (Scheme 17.4) [880, 881] have been synthesized by MCRs. A convenient starting reagent for MCRs aimed at the construction of heterocycles with a programmable

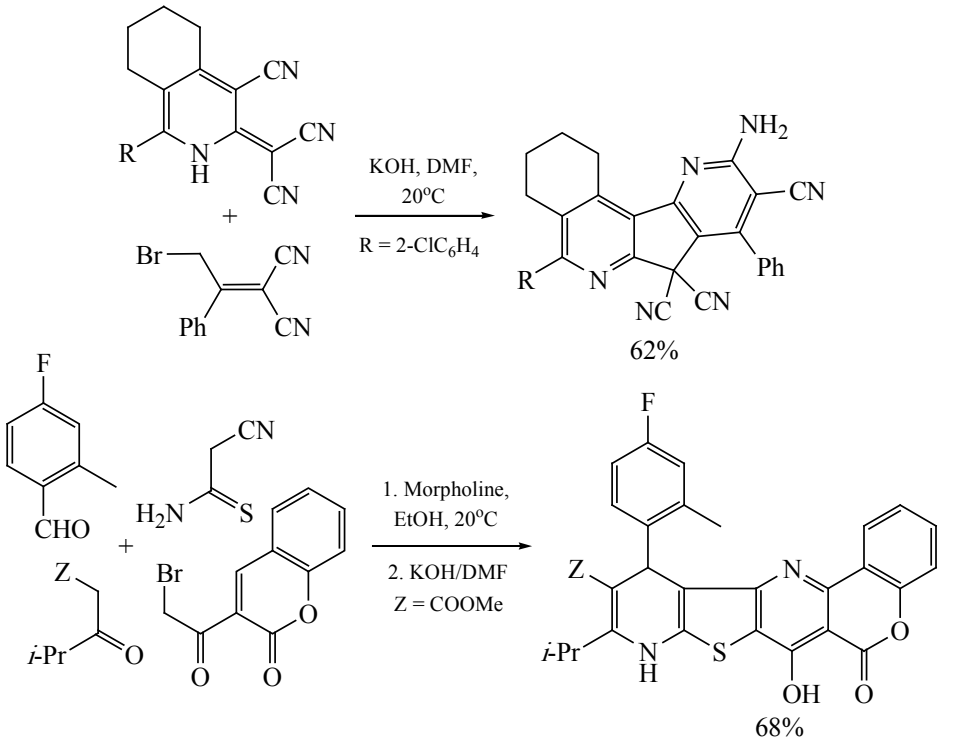
Scheme 17.3.



Scheme 17.4.


1. Morpholine/EtOH, 20°C
2. KOH/DMF, X Hal
Z = Me, Ph, COOPr, X = H, Alk, Ar
6 examples (68–81%)
1. EtONa, EtOH, 20°C
2. KOH/DMF,
82%
75%
1. EtONa, EtOH, 0°C
2. KOH/DMF
ArCHO +
16 examples (67–84%)
1. EtONa, EtOH, 0°C
2. KOH/DMF
3. Ac2O, D
75%
Z = Ar2CO, Ar2NHCO, COOAlk, CONH2
Ar = Ar1 = 4-MeOC6H4
1. PP/EtOH, 20°C
2. KOH/DMF,
RCHO +
72%
1. PP / EtOH, 20°C
2. EtONa/EtOH, PhCHO
80%
R = (CH2)2Ph, R1 = Me. Z = 4-BrC6H4NHCO
R = cyclohex-3-enyl, R1 = H
Morpholine, DMF, 20°C
RCHO +
6 examples (70–82%)
1. Morpholine, DMF, 20°C
2. KOH/DMF, R1Hal
AlkHal
R = i-Pr, Ar, R1 = Alk. Z = COOAlk, CN
4 examples (75–80%)
RCHO +
AlkHal
EtONa/EtOH, 50°C
R = Ar, Het
10 examples (69–81%)
EtONa, EtOH, 20°C
1. EtONa, EtOH, 20°C
2. Cyclohexanone, AcOH, Δ
6 examples (70–82%) X = O, S. R = i-Pr, Ph. Z = COAr, Ph, CN, CONH2, 4-BrC6H4NHCO
68%
PhCHO + 2
1. Et3N/EtOH, 20°C
2. KOH/DMF
3. HCONH2, Δ
Ar = 4-BrC6H4
86%

Scheme 17.5.



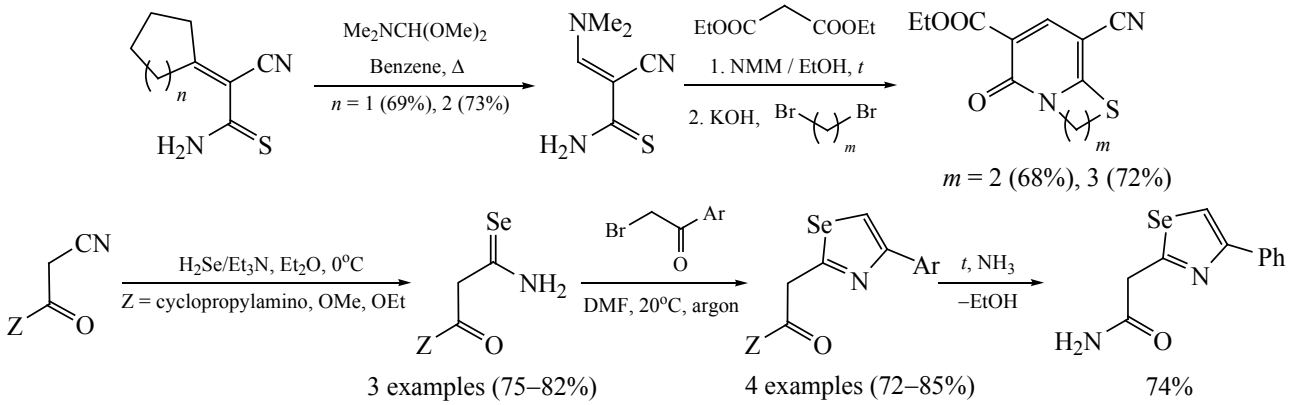
set of heteroatoms is cyanothioacetamide, whose numerous and diverse reactions is the subject of our monograph [882] and literature review [883].

In the course of our studies, we synthesized new heterocyclic systems, specifically a functionalized pyrido[2',3':3,4]cyclopenta[1,2-*c*]isoquinolines [884, 885] and chromeno[3",4":5',6']pyrido[2',3':4,5]thieno-[3,2-*e*]pyridine [886], as a result of cascade (domino) one-pot processes (Scheme 17.5).

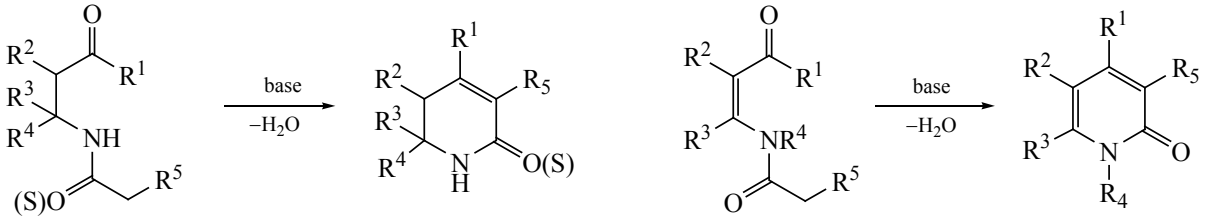
New reagents for organic synthesis were obtained: 3-dimethylamino-2-cyanoprop-2-enethioamide [887, 888], and selenium-containing CH-acids [889, 890]. Their synthetic potential was demonstrated by preparation of substituted thiazolo[3,2-*a*]pyridine, pyrido-[2,1-*b*][1,3]thiazine, and selenazoles (Scheme 17.6).

Today, the staff of Department of Chemistry and Biochemistry of Luhansk State Pedagogical University includes 3 doctors and 5 candidates of sciences Over the past 10 years, the department's staff has synthesized more than 800 new heterocyclic compounds. At the moment, we are not resting on our laurels and plan to continue searching for new options for the synthesis of practically important and biologically promising N, O, S, and Se-containing heterocycles.

Scheme 17.6.



Scheme 18.1.



1 **3** **2** **4**

R^1, R^2, R^3, R^4 = H, Alk, Ar; R^5 = H, Alk, Ar, FG R^1, R^2 = H, Alk, Ar; R^3 = Alk,Ar; R^4 = H, Alk; R^5 = Ar, FG

The staff of the Department of Chemistry and Biochemistry of Luhansk State Pedagogical University expresses special gratitude to Doct. Chem. Sci., Prof. V.G. Nenajdenko for comprehensive assistance and support and to Doct. Chem. Sci., Prof. V.N. Khrustalev and P.V. Dorovatovskii for X-ray structural analysis of our synthesized compounds, as well as hopes further cooperation.

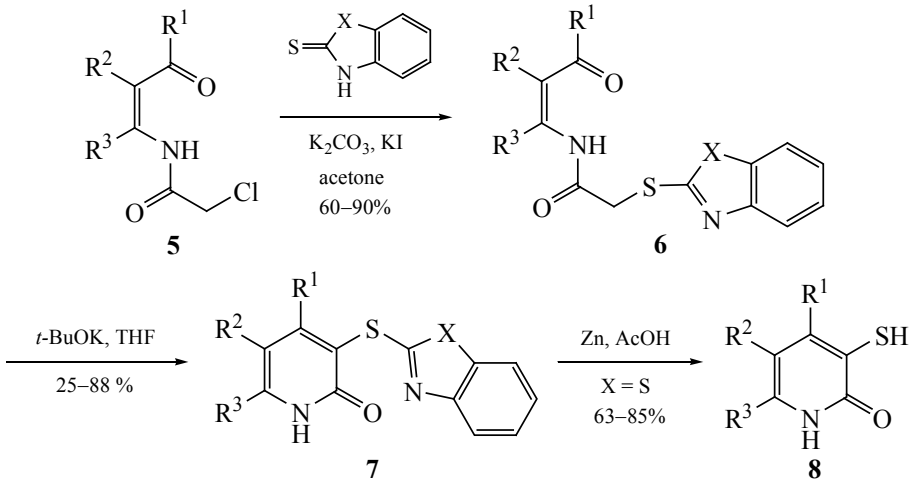
The review was prepared with the financial support of the state assignment for Lugansk State Pedagogical University VGEA-2024-0004 (reg. no. 1023082100012-4-1.4.1).

18. DEPARTMENT OF ORGANIC AND ANALYTICAL CHEMISTRY OF DOSTOEVSKII OMSK STATE UNIVERSITY AND DEPARTMENT OF CHEMISTRY AND CHEMICAL TECHNOLOGY OF OMSK STATE TECHNICAL UNIVERSITY

Over the past years, the department has been conducting active research on the synthesis, properties, and potentially practically useful compounds. These studies were supported by a number of grants from the Russian Foundation for Basic Research and the Russian Science Foundation. The review published in 2020 [891] provided the first systematization and analysis of the literature data on the cyclization of *N*-(2-acylaryl)-amides (Camps reaction), leading to quinolones, as well as the cyclization of their close analogs *N*-(3-oxoalkyl)- and *N*-(3-oxoalkenyl)amides and thioamides **1** and **2** into 5,6-dihydropyridin-2(1*H*)-ones, -thiones **3**, and pyridin-2(1*H*)-ones **4**, which was first implemented and studied at the Department of Organic Chemistry of Omsk State University (Scheme 18.1) [892–899].

The reaction of *N*-(3-oxoalkenyl)chloroacetamides **5** with 1,3-benzothiazole-2(3*H*)-thione, 1,3-benzoxazole-2(3*H*)-thione, and 1-methyl-1,3-dihydro-2*H*-benzimidazole-2-thione afforded the corresponding 2-(heteroarylsulfanyl)-*N*-(3-oxoalkenyl)acetamides **6**. Under the action of a base, these compounds were converted into pyridin-2(1*H*)-ones **7** containing a divalent sulfur

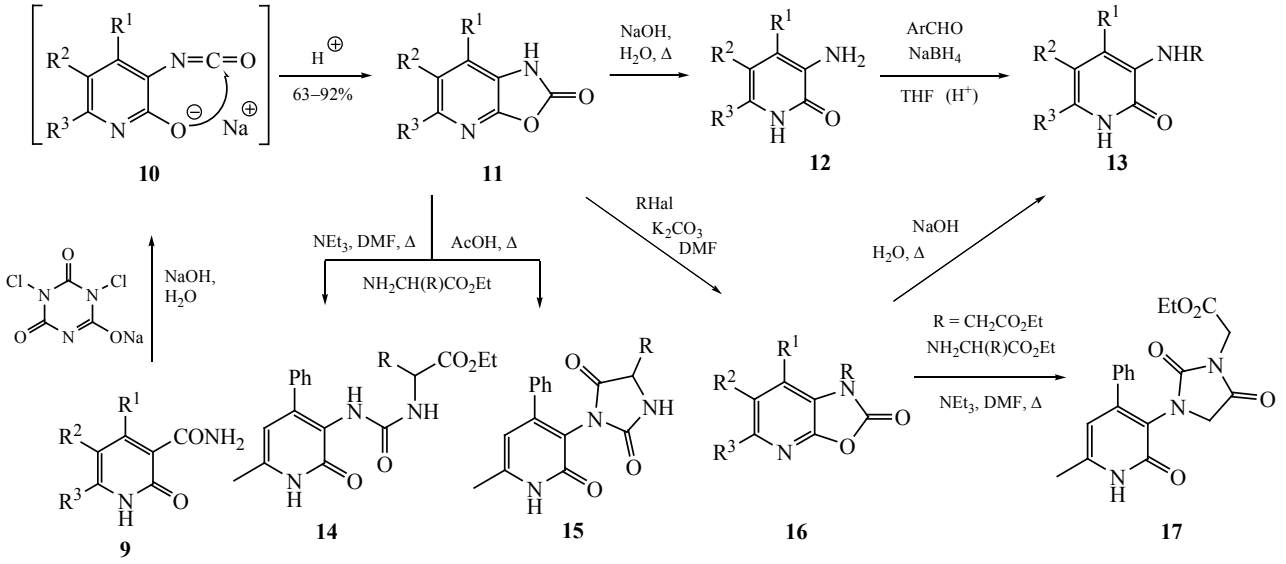
Scheme 18.2.



5 **6** **7** **8**

R^1 = Me, Ph; R^2 = H, R^2 + R^3 = $(CH_2)_4$; R^3 = Me; X = O, S, NMe

Scheme 18.3.



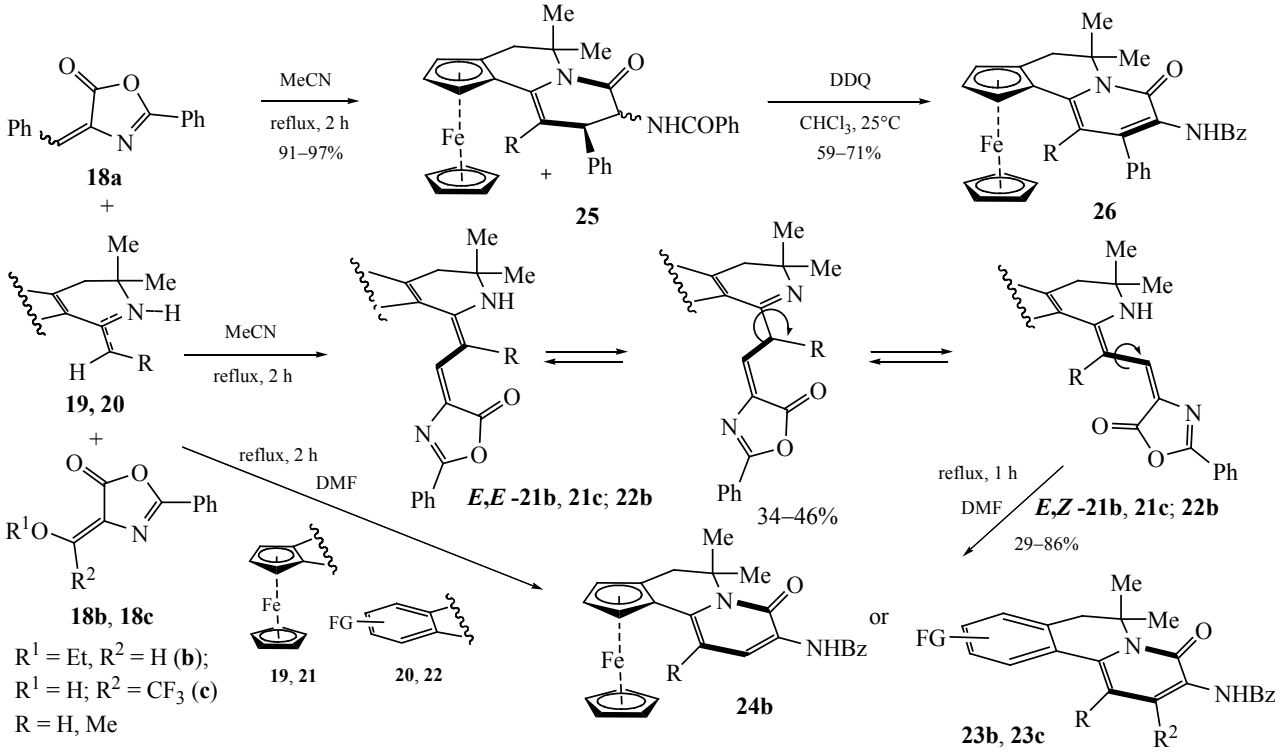
atom linked to a heterocycle at position C^3. Bromination, nitration, and alkylation of 3-(1,3-benzothiazol-2-ylsulfanyl)pyridin-2(1*H*)-ones **7** (X = S) were studied. The action of zinc in acetic acid on compounds **7** (X = S) yielded 3-sulfanylpyridin-2(1*H*)-ones **8** [900] (Scheme 18.2).

Previously, a method for the preparation of 4-aryl-3-aminopyridin-2(1*H*)-ones **12** based on *N*-(3-oxoalkenyl)-chloroacetamides was developed [898, 899]. Since these compounds were of interest as effective luminophores and antioxidants, alternative methods for their synthesis were studied. On attempted Hofmann reaction, 2-oxo-1,2-dihydropyridine-3-carboxamides **9** converted to oxazolo[5,4-*b*]pyridin-2(1*H*)-ones **11** through intramolecular cyclization of intermediate isocyanate **10**. Compounds **11** underwent hydrolysis upon heating with aqueous alkali to form 3-aminopyridin-2(1*H*)-ones **12** [901]. The alkylation of oxazolo[5,4-*b*]pyridin-2(1*H*)-ones **11** at the nitrogen atom followed by hydrolysis of cyclic carbamate **16** gave *N*-alkyl-3-aminopyridin-2(1*H*)-ones **13**. Compounds **13** were also synthesized by reductive amination from 3-aminopyridin-2(1*H*)-ones **12**, aromatic aldehydes, and $NaBH_4$ in THF with the addition of formic acid [902] or by $NaBH_4$ reduction of the corresponding Schiff bases in isopropanol (Scheme 18.3) [903]. The reactions of oxazolo[5,4-*b*]pyridin-2(1*H*)-ones **11** and **16** with amines involved five-membered ring opening to form ureas [902], while the reactions with amino acid esters resulted the formation of hydantoins **15** and **17** and ureidoacetate esters **14** (Scheme 18.3) [904].

Recently, a synthesis of fused 3-aminopyridin-2-one derivatives **23b**, **23c**, **24**, and **26** by the cycloaddition of azlactones 18b and 18c to 3,4-dihydroferroceno[*c*]-pyridines **19** and 1-alkyl-3,4-dihydroisoquinolines **20** under heating in DMF has been developed. The intermediate products of the addition of azlactone **18b** to the methyl substituents at the dihydropyridine rings in compounds **19** and **20** were isolated and characterized. The reaction of azlactone **18a** with 3,4-dihydroferroceno[*c*]pyridines **19** gave compounds **25**, which were oxidized with DDQ to 3-amino-2-phenyl-6,7-dihydroferroceno[*a*]quinolizin-4-ones **26** (Scheme 18.4) [905, 906].

At the same time, 3,4-dihydropyridin-2(1*H*)-ones **29a–29d**, **29j**, and **30a–30i**, obtained by the reaction of azlactones **18a–18j** with acetoacetic ester enamines **27** or 3-methyl-1-phenyl-1*H*-pyrazol-5-amines **28**, could not be oxidized to pyridin-2-ones **33** [907, 908]. However, the heating of these compounds with $POCl_3$ resulted in the isolation of oxazolo[5,4-*b*]pyridines **31a–31d**, and **31j** and **32a–32i**, formed by five-membered ring closure and oxidation of the dehydropyridine ring with atmospheric oxygen. Treatment of compounds **31a–31d**, and **31j** with aqueous alkali led to their hydrolysis to form amides **33a–33d**, and **33j** [16]. 5-Amino-1,7-dihydro-6*H*-pyrazolo[3,4-*b*]pyridin-6-ones **34a–34i** were prepared by the reaction of oxazolo[5,4-*b*]pyridones **32a–32i** with alkali and hydrazine hydrate in DMSO

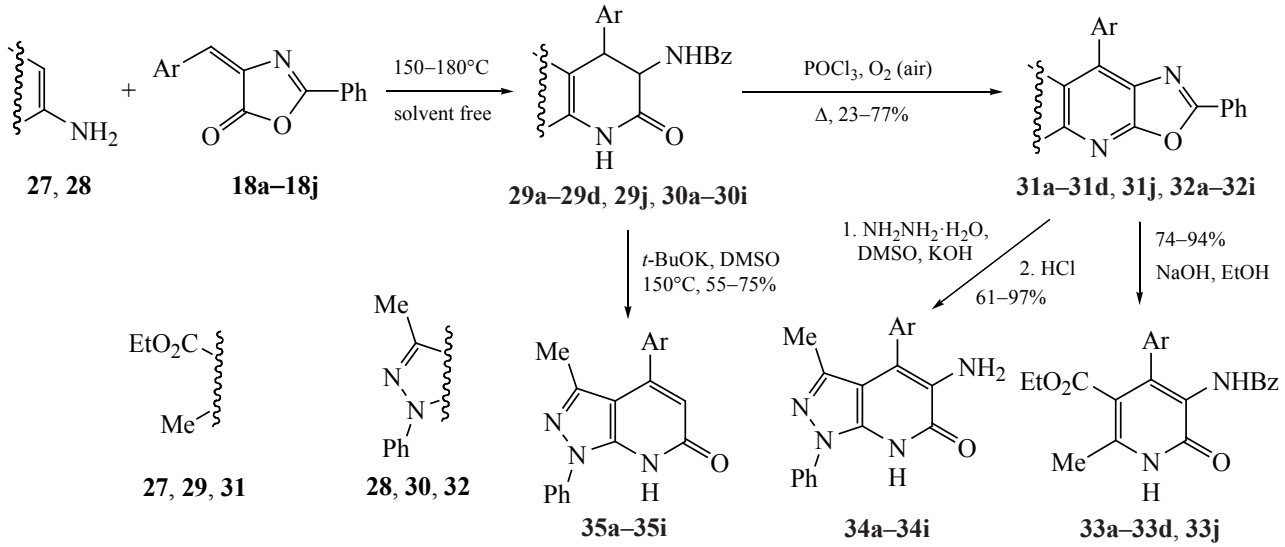
Scheme 18.4.



[907]. It was found that compounds **30a–30i** in a superbasic medium (DMSO, *t*-BuOK) eliminated benzamide and transformed into 1,7-dihydro-6*H*-pyrazolo[3,4-*b*]pyridin-6-ones **35a–35i** in good yields (Scheme 18.5) [909].

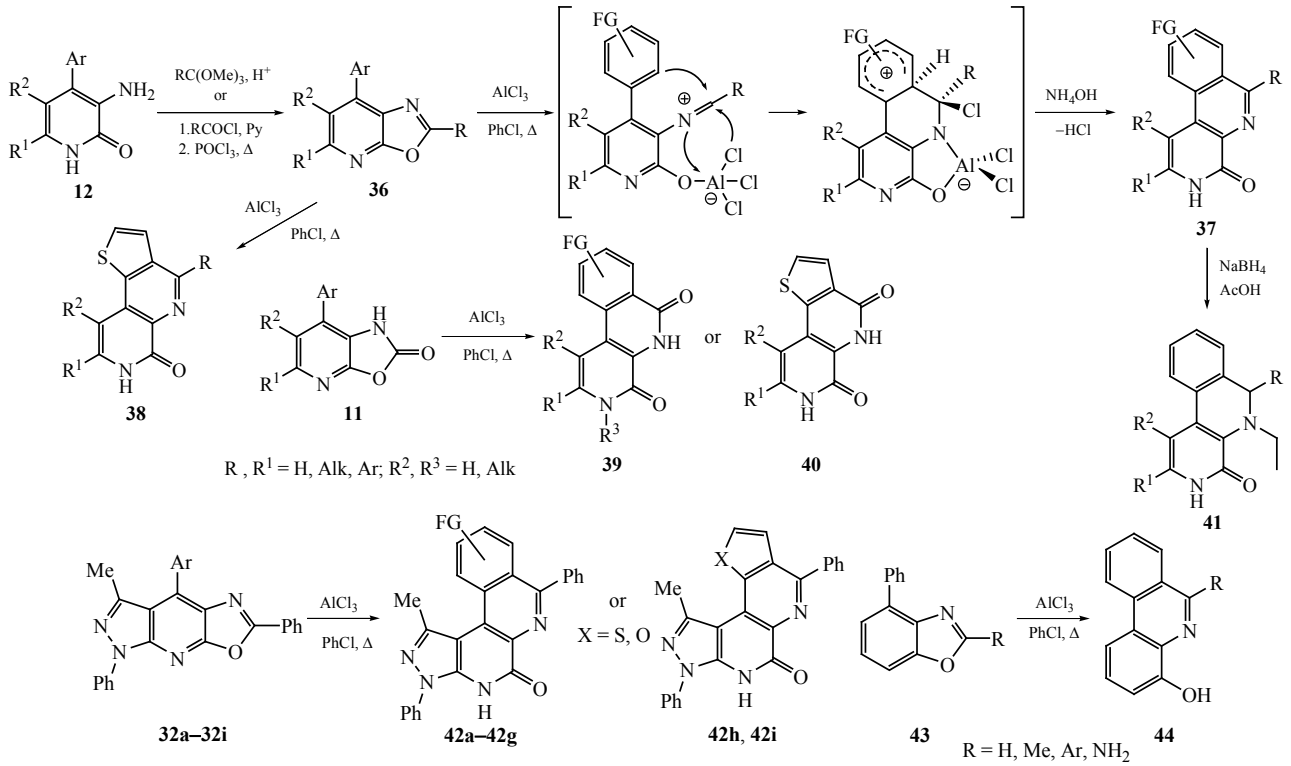
It was found that upon heating with aluminum chloride at 90°C in chlorobenzene, 7-aryl-substituted oxazolo[5,4-*b*]pyridines **36** underwent a previously unknown rearrangement to into benzo[*c*][1,7]naphthyridinones **37** [910]. Compounds **11**, **16** [910] and **32**

Scheme 18.5.



Ar = Ph (**a**), Ar = 4-$MeOC_6H_4$ (**b**), Ar = 4-FC_6H_4 (**c**), Ar = 4-ClC_6H_4 (**d**), Ar = 4-MeC_6H_4 (**e**), **4**, Ar = 3,4-$(MeO)_2C_6H_3$ (**f**), Ar = 3,4,5-$(MeO)_3C_6H_2$ (**g**), Ar = 2-thienyl (**h**), Ar = 2-furyl (**i**), 4-$NO_2C_6H_4$ (**j**)

Scheme 18.6.



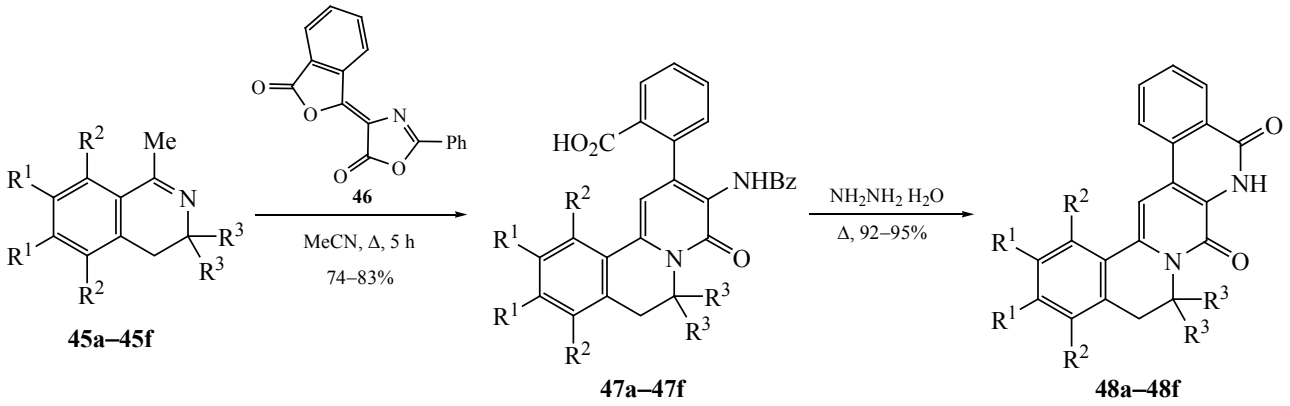
[908], as well as oxazolo[5,4-*b*]pyridines **36** with p-donor thiophene or furan rings enter into a similar reaction to form products **38–40** and **42** (Scheme 18.6).

It should be noted that we first obtained naphthyridines 37 and 38 by the Pictet–Spengler reaction from aromatic aldehydes and 3-aminopyridin-2(1*H*)-ones **12** [911, 912]. This reaction occurred under harsh conditions (heating in phosphoric acid at 130°C), which significantly limited its versatility. The synthetic approach to naphthyridinones, based on rearrangement, made it possible to obtain not only thiophene and furan derivatives **38**, **40**, and **42**, but also to introduce an alkyl substituent and a hydrogen atom into the pyridine ring (Scheme 18.6). It was recently shown that 4-phenylbenzo[*d*]oxazoles **43** undergo a similar rearrangement to form phenanthridin-4-ols **44** [913], which provides evidence for the general nature of this rearrangement (Scheme 18.6). In [910, 913], a mechanism of this reaction was proposed, and its elementary acts were studied using quantum-chemical calculations. The new heterocyclic systems benzo[*c*]-[1,7]naphthyridine-4(3*H*)-ones were studied in oxidation, alkylation, and electrophilic substitution reactions, as well as other transformations [914]. Treatment of compounds **37** with $NaBH_4$ in AcOH gave rise to an unusual reductive amination, which involved acetic acid and formed 5-ethyl-5,6-dihydrobenzo[*c*]-[1,7]naphthyridine-4(3*H*)-ones **41** (Scheme 18.6) [915]. Among 3-aminopyridin-2(1*H*)-ones **12–14**, **34** and dihydronaphthyridines **41**, effective luminophores [901, 902, 908, 915] and potent antioxidants [901, 908] were found. This finding made it possible to develop on their basis dyes for immunoenzyme analysis [902] and histochemical staining of skin cryosections [915].

The reaction of 3,4-dihydroisoquinolines **45a–45f** with azlactone **46** was used to obtain, for the first time, carboxylic acids **47a–47f** [916], whose treatment with hydrazine hydrate gave previously unknown [1,7]naphthyridine-5,7-diones **48a–48f** (Scheme 18.7).

Recently, a method of synthesis of 1-tosyl-3*H*-naphtho[1,2,3-*de*]quinoline-2,7-diones **51a–51c** by the reaction of chloroacetamides **49a–49c** with sodium *para*-toluenesulfinate has been propposed. It has been found that the nucleophilic substitution of the tosyl group in compounds **51a–51c** by nitrogen and oxygen nucleophiles occurs under mild conditions, in some cases at room or low temperatures (Scheme 18.8) [917].

Scheme 18.7.



R^1 = OMe, R^2 = H, R^3 = Me (**a**), $R^1 + R^1 = O(CH_2)_2O$, R^2 = H, R^3 = Me (**b**), R^1 = Me, R^2 = H, R^3 = Me (**c**), $R^1 + R^1 = (CH_2)_4$, R^2 = H, R^3 = Me (**d**), R^1 = H, R^2 = OMe, R^3 = Me (**e**), R^1 = OMe, $R^2 = R^3$ = H (**f**)

A new synthesis of the alkaloids harmane, harmine, and their analogs **56** by thermolysis of substituted 3-azido-4-arylpyridines **55** obtained from 3-amino-4-arylpyridines **53** [918, 919] via diazonium salts **54** was developed. It should be noted that in the presence of methoxy substituents in the aromatic ring adjacent to the diazonium cation, intramolecular diazotization with the formation of pyrido[3,4-*c*]- or pyrido[3,2-*c*]cinnolines **61** and **62** occurs [919, 920]. The Cadogan reaction was also used to close the pyrrole ring of compounds **58** and **59**. 3-Nitropyridines **57** containing an aromatic substituent on C^2 were converted into γ-carbolines in yields of 30–71% under heating in *p*-cymene with PPh_3 in the presence of 5 mol % $MoO_2Cl_2(DMF)_2$ or with 1,2-bis(diphenylphosphino)ethane in solvent-free conditions [921, 922]. This method was used to obtain δ-carbolines **59** annulated with unsaturated carbocycles, including 2,3,4,10-tetrahydro-1*H*-indolo[3,2-*b*]quinolines **59** (*n* = 1) [922]. The dehydrogenation of the latter compounds under heating in diphenyl oxide in the presence of Pd/C gave the alkaloid quindoline and its analogs 60 [921]. β-Carbolines **56** can also be obtained by the Cadogan reaction from nitropyridines **57** (R^3 = Ar) and $P(OEt)_3$, but, however, in this case the reaction proceeds in low yields (Scheme 18.9) [919].

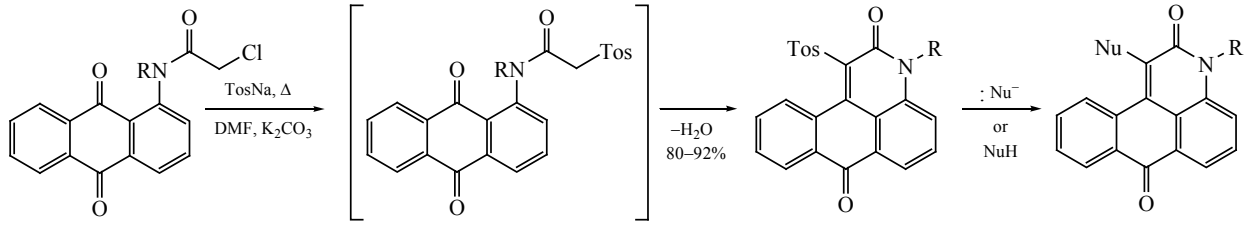
Thermolysis of azido derivatives was also used for the synthesis of thienoindoles. To this end, 2-azido-1-arylethan-1-ones **63a–63h** were subjected to Vilsmeier reaction ($DMF/POCl_3$), and the resulting mixtures of (*Z*)- and (*E*)-2-azido-3-aryl-3-chloroacrylaldehydes **64a–64h** were heated with mercaptoacetate in the presence of Cs_2CO_3 in methanol (Scheme 18.10).

When 1.5 equiv of $HSCH_2CO_2Me$ were used, the reaction did not proceed to completion; with 2 equiv of of $HSCH_2CO_2Me$, a mixture of products **65** and **66** formed, while with 3 equiv, complete reduction of the azido group took place to give amines **65a–65h** (yields 61–78%). Compounds **65a–65h** were converted via the corresponding diazonium salts to azidothiophenes **66a–66h** in yields of 49–78%. The thermolysis of azides **66a–66h** in a MW reactor led to compounds **67a–67h** (Scheme 18.10) [923]. Recently, another synthetic approach to fused thiophene derivatives **71a–71j** has been proposed, based on iodine-promoted photocyclization of 4,5-diaryl-substituted thiophenes **70a–70j** (Scheme 18.11) [924]. Thiophene derivatives **71** have been used to synthesize structures of interest as materials for organic electronics [925].

For the same purpose, as well as to study the relationship between of electronic and photophysical properties on the structure of conjugated five-membered heterocyclic systems, compounds **72** [926], **73** [927,928], and **74** [929] were synthesized (Fig. 18.1).

The Pd-catalyzed cyclization of 4-[(arylamino)methyl]thiophene-2- or 4-(aryloxymethyl)thiophene-2-carbaldehydes containing an iodine atom in the thiophene **75** and benzene rings **76** rings was used to prepare 4,5-dihydrothieno[3,2-*c*]quinoline-2- and 4*H*-thieno-[3,2-*c*]chromene-2-carbaldehydes **77** and **78**. Comparison of these two approaches showed that the best yields of thienoquinolines **78** are achieved by the cyclization of compounds containing a halogen atom in the benzene ring **76** [930], whereas thienochromenes **77**

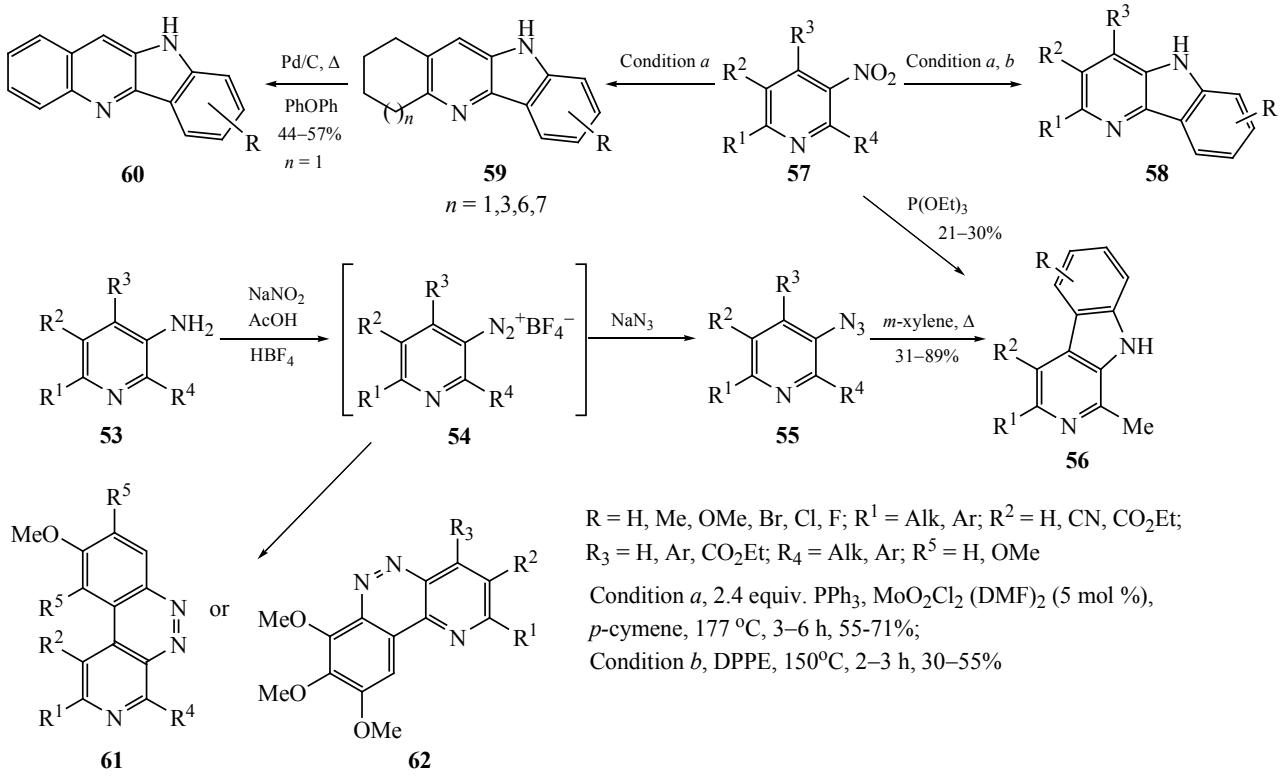
Scheme 18.8.



49a–49c **50a–50c** **51a–51c** **52a–52c**

R = H (**a**), Me (**b**), *n*-C_6H_{13} (**c**) $Nu^- = N_3^-, HO^-, RO^-$; NuH = R_2NH

Scheme 18.9.



60 **59** *n* = 1,3,6,7 **57** **58**

53 **54** **55** **56**

61 **62**

R = H, Me, OMe, Br, Cl, F; R^1 = Alk, Ar; R^2 = H, CN, CO_2Et; R_3 = H, Ar, CO_2Et; R_4 = Alk, Ar; R^5 = H, OMe

Condition *a*, 2.4 equiv. PPh_3, $MoO_2Cl_2(DMF)_2$ (5 mol %), *p*-cymene, 177 °C, 3–6 h, 55-71%;
Condition *b*, DPPE, 150°C, 2–3 h, 30–55%

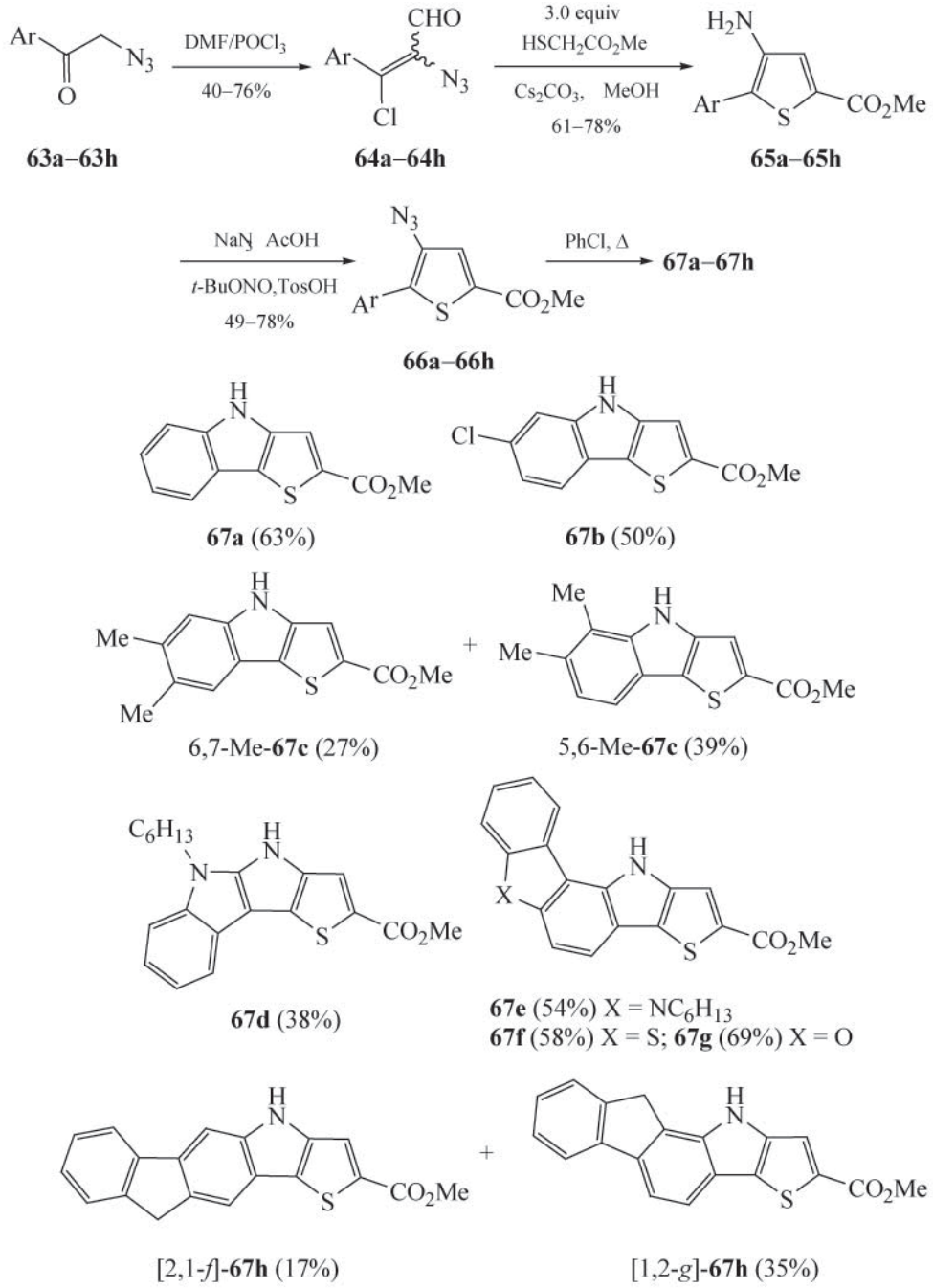
are best prepared from compounds **75**, which contain a halogenated thiophene ring [931, 932] (Scheme 18.12). It should be noted that compounds **92** can be cyclized under heterogeneous catalysis with 10% Pd/C, but the yields of products **78** in this case are slightly lower. Good results were obtained in the photochemical closure of the pyran ring [933]. With both compounds **75** and **76** (X = O), good yields of thienochromenes **77** were obtained, but the reaction with compounds **75** containing an iodine atom in the thiophene ring was twice as fast.

Compounds **77** and **78** are efficient luminophores luminescing in the blue in the blue and yellow-green spectral regions with high quantum yields (up to 0.87) and large Stokes shifts (up to 159 nm). This allowed these compounds to be proposed for use as luminescent inks for hidden marking of paper [930, 933].

The photochemical oxidative cyclization of dithioamides **79a–79e**, **80a–80e**, and **83a**, **83c–83f** in the presence of chloranil, leading to the formation of benzo[1,2-*d*:4,3-*d*']bis(thiazolam) **84a**, **84c–84f** [934] and previously unknown dithiazolobenzo[1,2-*c*][1,2,5]-thiadiazolams **81a–81e** and dithiazolo[1,2-*d*][1,2,3]-triazolams **82a–82e** was studied for the first time (Scheme 18.13) [935].

The HOMO and LUMO energies the synthesized compounds were calculated by the DFT method and measured by cyclic voltammetry. The photophysical and

Scheme 18.10.



Ar = Ph (**a**); Cl-4-C_6H_4 (**b**); 3,4-$(Me)_2$ (**c**); 1-hexyl-1*H*-indolyl-3 (**d**); 9-hexyl-9*H*-carbazol-3-yl (**e**); dibenzo[*b*,*d*]thiophen-2-yl (**f**); dibenzo[*b*,*d*]furan-2-yl (**g**)

(spectro)electrochemical properties of the fused systems and the polymer films electrochemically deposited on ITO electrodes were studied. The optical contrast and response time of the synthesized oligomers were determined. The resulting data allow us to consider these compounds as promising candidates for electrochromic devices [935].

The review was prepared with the financial support of the Russian Science Foundation (project no. 22-13-00356).

Scheme 18.11.

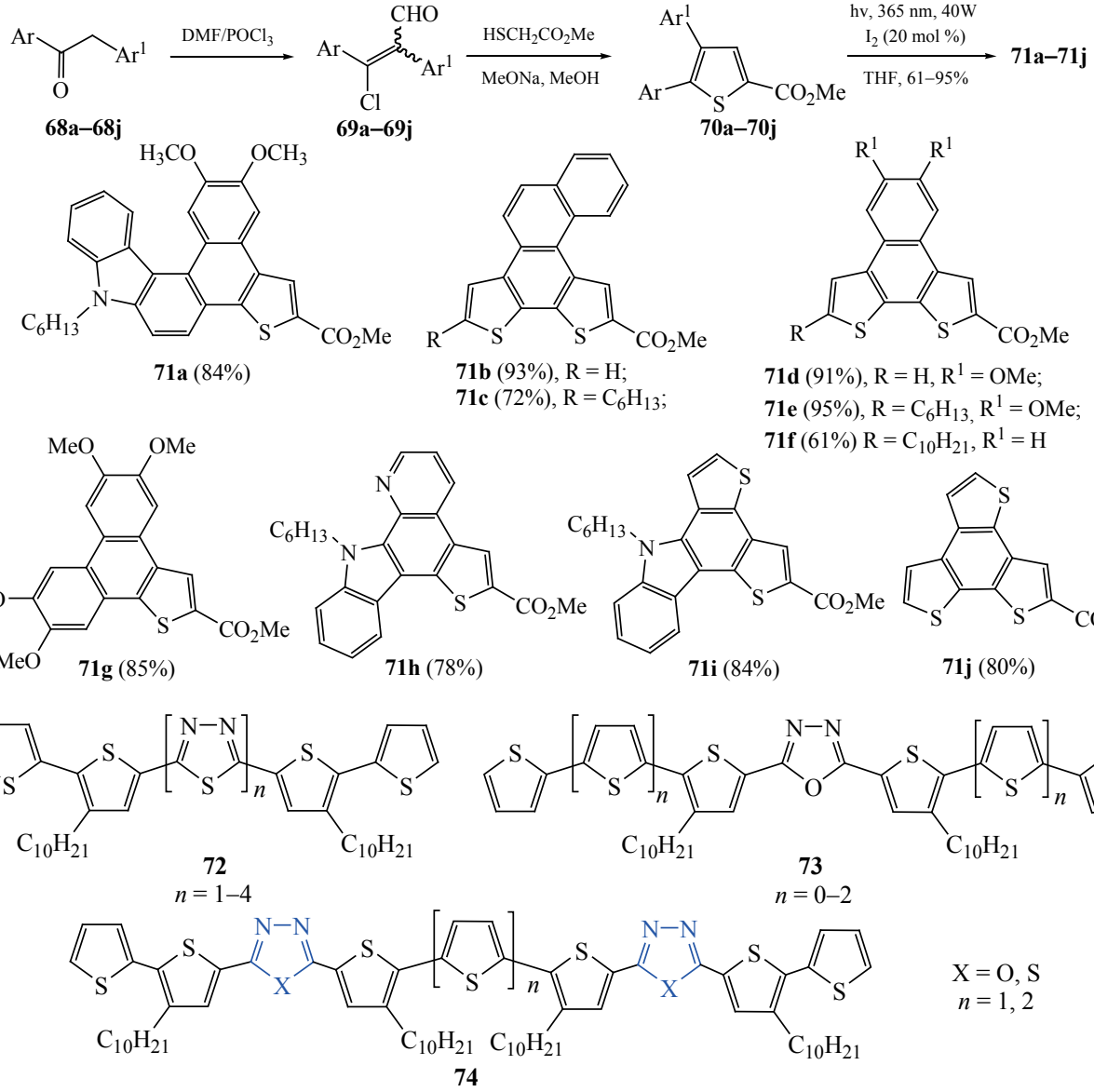
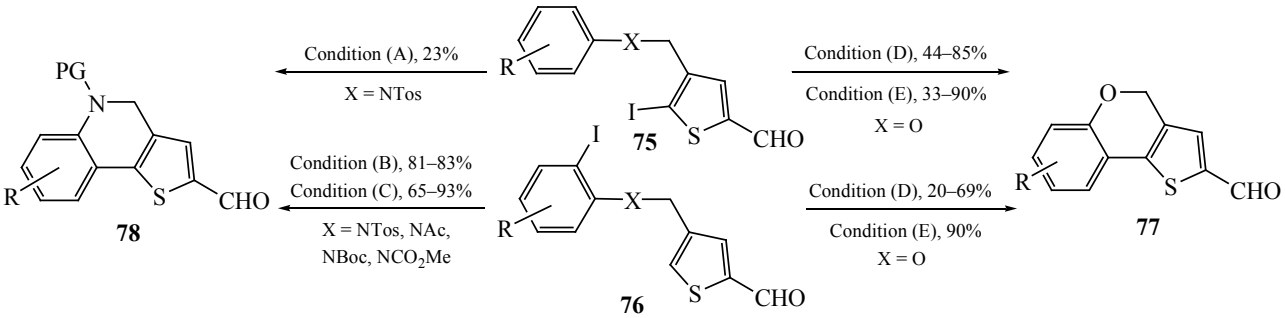


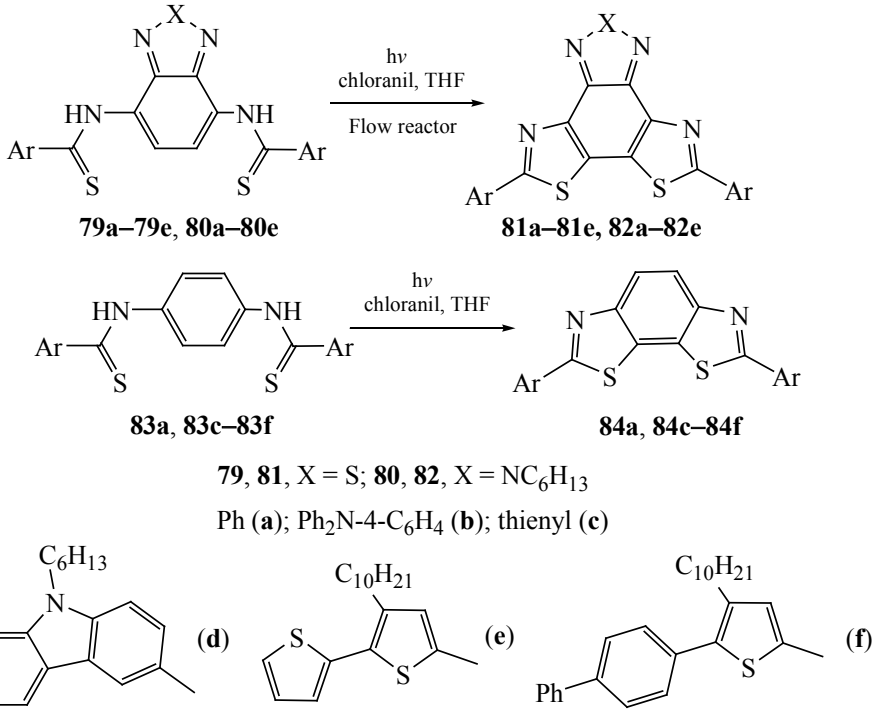
Fig. 18.1. Ensembles of conjugated five-membered heterocycles **72**–**74**.

Scheme 18.12.



Condition: (A) $P(PPh_3)_4$, DMF, Δ; (B) Pd/C, DMA, Δ; (C) $P(PPh_3)_4$, DMA, Δ; (D) $Pd(OAc)_2$, PPh_3, K_2CO_3, $C_{16}H_{33}NMe_3Br$, DMF, Δ; (E) hv, 254 nm, MeCN; R = H, Cl, F, OMe, CO_2Me, NO_2, NMe_2

Scheme 18.13.



19. DEPARTMENT OF ORGANIC CHEMISTRY OF KUBAN STATE UNIVERSITY—THE SOUTHERNMOST BRANCH OF THE SCHOOL OF ACADEMICIAN A.E. FAVORSKII

The Department of Organic Chemistry was created at the Faculty of Natural Sciences of the Krasnodar State Pedagogical Institute named after the 15th Anniversary of the Komsomol. The first head of the department was **Mikhail Petrovich Pyatnitskii** (1905–1981), a talented agricultural chemist, Doctor of Biological Sciences (1961), Professor, author of more than 150 scientific papers, and holder of the Order of Lenin (1965). Scientific interests of Prof. Pyatnitskii were focused on methods for extracting vitamin C from citrus fruits, as well as of citric acid from tobacco leaves and makhorka. The antiscorbutic and wound-healing cedar tinctures obtained by M.P. Pyatnitskii saved lives for many Soviet soldiers during the Great Patriotic War.

The modern stage of research in organic chemistry at Kuban State University began in 1971, when the charismatic young scientist **Nikolai Vasil'evich Komarov** (1928–2008) was invited to head the department at the newly reorganized university. He was one of the first to defend his doctoral dissertation at the Irkutsk Institute of Organic Chemistry under the supervision of Corr. Member Acad. Sci. USSR M.F. Shostakovskii, a student of Acad. A.E. Favorskii. Komarov was among the pioneers of the chemistry of unsaturated compounds of silicon, germanium, tin, and

lead, whose research made a significant contribution to the development of organometallic methods for forming Si(Ge,Sn,Pb)–C_{sp} bonds. Subsequently, new preparative methods for the synthesis of this type of compounds using heterofunctional silicon, germanium, and tin derivatives were discovered, which did not require the preliminary enhancement of the nucleophilicity of 1-alkynes by metalation with traditional reagents, such as organolithium compounds or Grignard reagents.

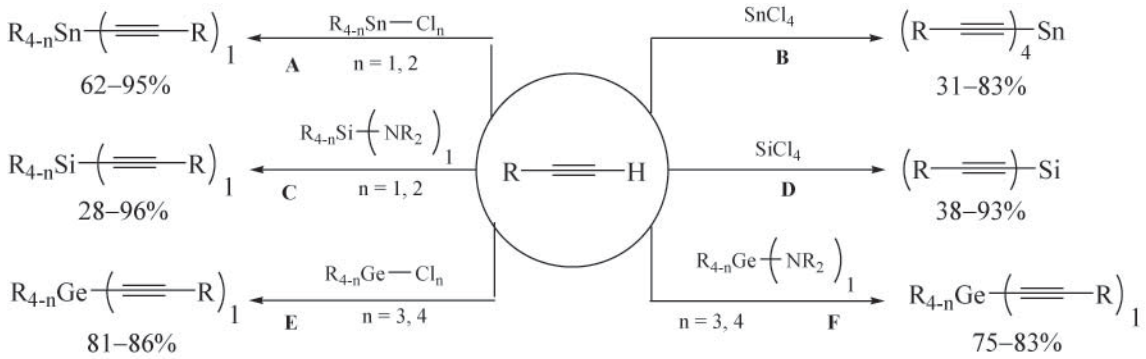
Many of the graduates of the Department of Organic Chemistry continued their careers in science and higher education. Thus, among the best graduates of the department, noteworthy are **Vladimir Gevorgyan**, a 1978 graduate, Professor at the University of Texas at Dallas, US, a world-famous organic chemist, author of more than 200 scientific publications on transition metal catalysis, and **Alexei Alekseevich Andreev** (1953–2010), a 1976 graduate, an experienced teacher and brilliant practical chemist, who devoted many years to working at the Department of Organic Chemistry of Kuban State University.

Since the Department of Organic Chemistry became a separate structural unit in 1971, its staff at various times included Cand. Chem. Sci. E.V. Serebrennikova, Cand. Chem. Sci. L.I. Ol'khovskaya, Cand. Chem. Sci. K.S. Pushkareva, Cand. Chem. Sci. T.N. Dybova, Cand. Chem. Sci. E.M. Pokrovskaya, a graduate of the Herzen State Pedagogical University and a representative of the school of Prof. V.V. Perekalin, Cand. Chem. Sci. O.I. Yurchenko, Cand. Chem. Sci. I.S. Akchurina, Cand. Chem. Sci. V.A. Kovardakov, Doct. Chem. Sci. V.D. Buikliskii, Cand. Chem. Sci. L.I. Komarova, Cand. Chem. Sci. L.P. Vakhrushev, and Cand. Chem. Sci. V.S. Senichev. In 2007, the department was renamed the Department of Organic Chemistry and Technologies. In 2012, the department was headed by Doct. Chem. Sci. **Vladimir Denisovich Strelkov** (1952–2024), an agrochemist, who worked for a long time at the Russian Research Institute of Biological Plant Protection. After his arrival at the department, Doct. Chem. Sci. Strelkov initiated research on the synthesis and properties of new plant growth regulators and phytoimmunomodulators or antidotes to the herbicide 2,4-D.

Since 2017, the Department of Organic Chemistry and Technology of KubSU has been headed by **Victor Victorovich Dotsenko** (b. 1976), Doctor of Chemical Sciences (2015), a representative of the Lugansk school of heterocyclic chemistry. At this time, a new direction related to the heterocyclization reactions of methylene-active nitriles and thioamides appears and begins to actively develop at the department. As of January 2024, the department employs 2 Doctors of Sciences (V.D. Strelkov, V.V. Dotsenko), 5 Associate Professors/Candidates of Chemical Sciences N.A. Ryzhkova, D.Yu. Lukina, A.S. Levashov, A.V. Bespalov, and D.S. Buryi, and 4 support staff members involved in the educational process (L.V. Konovalov, V.A. Kotsegubova, L.V. Buchinskaya, and V.A. Somova). Every year the department graduates 10–14 bachelors and 8–10 masters in chemistry; a postgraduate program in the field of organic chemistry is functioning.

In general, two research groups have been formed and are successfully working at the Department of Organic Chemistry. One of the groups is conducting research that originates from the works of Acad. Favorskii and his students and is related to the synthesis and properties of silicon, germanium, and tin acetylides (Cand. Chem. Sci. A.S. Levashov, Cand. Chem. Sci. V.V. Konshin, and Cand. Chem. Sci. D.S. Buryi). The second group is focused on the chemistry of derivatives of methylene-active nitriles and thioamides (O/S/N-heterocyclic compounds of the thieno[2,3-*b*]-pyridine, 1,3,5-thiadiazine, and 1,2,4-thiadiazole series, etc.), including in silico studies on their reactivity

Scheme 19.1.



A, Et_2NH; **B**, $ZnCl_2$ (110 mol %), Et_2NH (225 mol %), PhMe, 20°C, 20 min; **C**, $ZnCl_2$ (165 mol %), 1,4-dioxane, 100°C; **D**, Zn(OTf)2 (125 mol %), *i*-Pr_2NEt (125 mol %), DCE, 45°C, 3–12 h; **E**, $ZnCl_2$ (107 mol %), Et_3N (108 mol %), 1,4-dioxane, 100°C, 4 h; **F**, $ZnCl_2$ (100 mol %), 1,4- dioxane, 100°C, 4 h.

and biological activity, and well as studies on the agrochemical potential of these compounds as herbicide antidotes and plant growth regulators (Doct. Chem. Sci. V.D. Strelkov, Doct. Chem. Sci. V.V. Dotsenko, Cand. Chem. Sci. D.Yu. Lukina, and Cand. Chem. Sci. A.V. Bespalov).

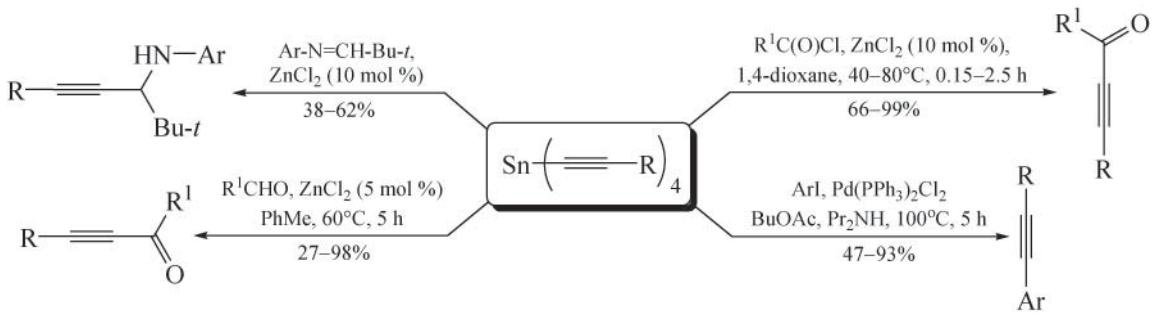
The discovery by A.A. Andreev and N.V. Komarov that alkynyltriorganylstannanes and dialkynyldiorganylstannanes can be easily obtained by the reaction of the corresponding organotin halides with 1-alkynes in a diethylamine medium [936] proved to be historically significant for the development of the first scientific direction. A similar reaction of tin tetrachloride and phenyltrichlorostannane, but occurring in the presence of zinc halides, opened the way to previously relatively difficult-to-access tin trialkynylides and tetraalkynylides (Scheme 19.1) [937].

The use of aminosilanes for the silylation of 1-alkynes also becomes possible when the reaction is carried out in 1,4-dioxane in the presence of excess zinc chloride [938]. Later this reaction was extended to diaminosilanes [939]. Tetraaminosilanes were found to be unreactive under these conditions; in this case, tetraalkynylsilanes formed only in trace amounts and could be synthesized by the reaction of silicon tetrachloride with 1-alkynes in the presence of excess zinc triflate and Hunig's base [940].

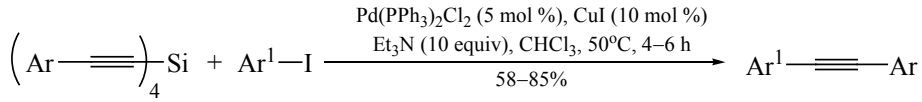
1-Alkynes were quite smoothly germylated with the assistance of zinc halides under the action of germanium tetrachloride, phenyltrichlorogermane, and their dialkylamino derivatives [941].

Tin tetraalkynylides can be considered as atom-economical alkynylating reagents, free of such disadvantages of tin triorganoalkynylides as toxicity, unpleasant odor, and a large molecular weight of the "ballast" fragment, which adversely affects the efficiency of the reaction. We found that tin tetraalkynylides can be very effectively used in the Stille reaction [942], in the synthesis of acetylenic ketones from acyl chlorides [943], and with aliphatic acid chlorides the yield of the acylation reaction becomes practically quantitative (Scheme 19.2). The reaction with aldehydes proceeds interestingly and, contrary to expectations, the main reaction products are acetylenic ketones [944, 945] rather than propargyl

Scheme 19.2.



Scheme 19.3.



alcohols, that is, in this case, the intermediate tin alkoxides undergo Oppenauer oxidation.

Alkynylation of imines occurs in moderate yields and best performed in solvent-free conditions [946].

Tetraalkynylsilanes can also be used as alkynylating reagents in the cross-coupling reaction, but, unlike the corresponding tin acetylenes, a copper cocatalyst is required in the sila-Sonogashira reaction (Scheme 19.3) [947].

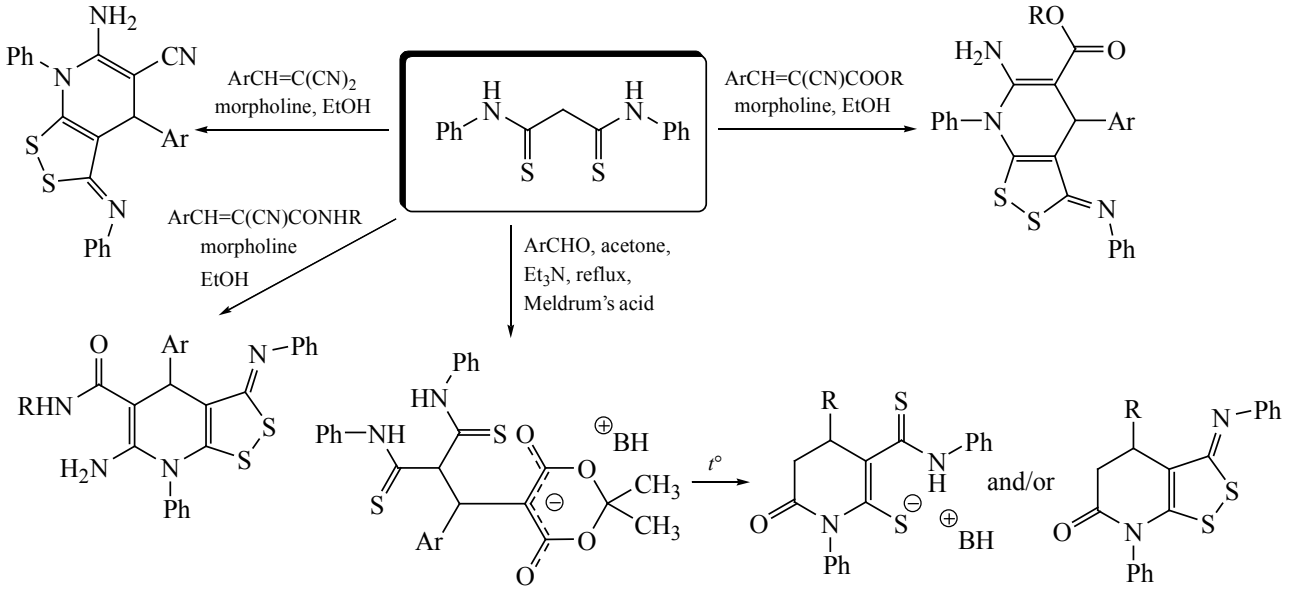
Tetraalkynylgermanes under similar conditions were less reactive, and the yield of the corresponding tolanes as low as 34% [947].

Over the previous 10 years, the department's staff has published more than 100 articles on the heterocyclization reactions of methylene-active nitriles and thioamides and the properties of the synthesized compounds and received more than 30 patents for inventions. Some results are summarized in the reviews [948–954].

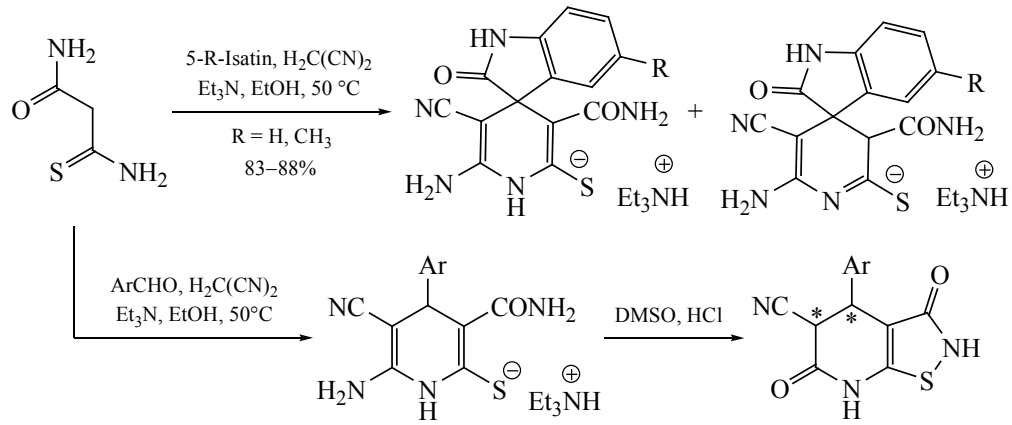
Recent studies at the department have been aimed at assessing the potential of dithiomalondianilide in the synthesis of heterocyclic compounds (Scheme 19.4). Dithiomalondianilide has long been known as a bidentate complexing agent, but, at the same time, it is a rather strong CH acid (pK_a 10.25–10.28) [955], which creates wide possibilities for its use in organic synthesis. Thus, dithiomalondianilide reacts with acrylonitrile derivatives in the presence of morpholine to form functional derivatives of [1,2]dithiolo[3,4-*b*]pyridine [956–959]. Dithiomalondianilide reacts with aromatic aldehydes and Meldrum's acid, yielding stable Michael adducts [960, 961]. Upon prolonged heating, the Michael adducts undergo cyclization with simultaneous partial oxidation, resulting in the formation of substituted pyridine-2-thiolates and tetrahydro[1,2]dithiolopyridines [962]. Some of the resulting compounds showed a pronounced antidote effect against the herbicide 2,4-D in tests on sunflower seedlings.

The use of monothiomalonamide as a methylene-active reagent allows one-pot synthesis of various nicotinamide derivatives. Thus, by the reaction of isatins, malononitrile, and monothiomalonamide in the presence of Et_3N gave spiro-fused nicotinamide derivatives as a mixture of prototropic tautomers [962]. A similar reaction with aromatic aldehydes forms the corresponding thiolates, whose oxidation with the DMSO–HCl system provides hexahydroisothiazolo[5,4-*b*]pyridines (Scheme 19.5) [963].

Scheme 19.4.



Scheme 19.5.



In general, oxidation reactions of thioamides of different structures can be used to obtain a variety of S,N-heterocycles. Thus, under the action of a wide range of oxidizing agents, 3-aryl-2-cyanothioacrylamides are converted into functional derivatives of 1,2,4-thiadiazole (Scheme 19.6) [964–967]. Some of the obtained compounds demonstrate high agrochemical potential as antidotes of 2,4-D.

The traditional line of research of the department is the synthesis and properties of thieno[2,3-*b*]pyridine derivatives. Thus, an unusual oxidative dimerization of 3-aminothieno[2,3-*b*]pyridine-2-carboxamides under the action of sodium hypochlorite has been recently discovered (Scheme 19.7) [968, 969].

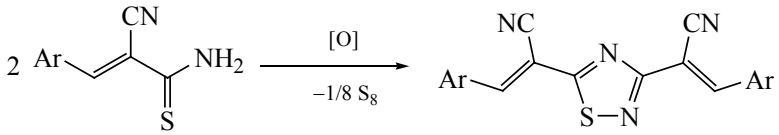
Acetylenic ketones react with cyanothioacetamide in the presence of morpholine to form 2-thioxo-1,2-dihydronicotinonitriles, which easily convert to the corresponding 3-aminothieno[2,3-*b*]pyridines under Thorpe–Ziegler reaction conditions (Scheme 19.8) [970].

3-Aminothieno[2,3-*b*]pyridines are a convenient molecular platform for further functionalization and construction of polycyclic assemblies. Thus, treatment of 3-aminothieno[2,3-*b*]pyridine-2-carboxamides with phosphorus sulfide in hot pyridine leads to 1,3,2λ^5-diazaphosphinine derivatives [971], the reaction with phthalic anhydride results in the formation of thieno[2',3':5,6]pyrimido[2,1-*a*]isoindoles [972], and acid-catalyzed condensation with ninhydrin gives spirocyclic derivatives of pyrido[3',2':4,5]thieno[3,2-*d*]-pyrimidine (Scheme 19.9) [973].

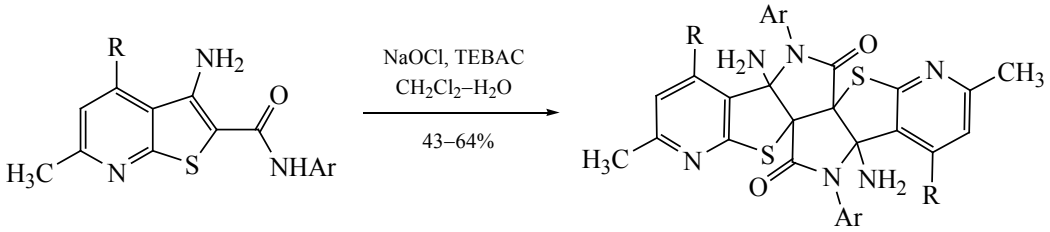
3-Aminothieno[2,3-*b*]pyridines readily react with 3,5-dimethyl-1-(cyanoacetyl)pyrazole to form the corresponding cyanoacetamides, which undergo Camps cyclization under the action of strong bases, as exemplified by the synthesis of dipyridothiophene derivatives (Scheme 19.10) [974].

Another direction of research in heterocyclic chemistry, actively developed at the department, is

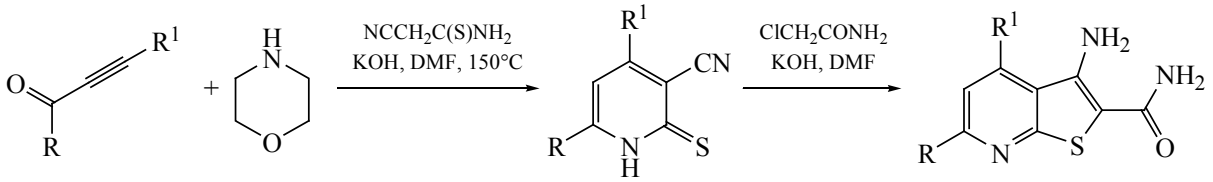
Scheme 19.6.



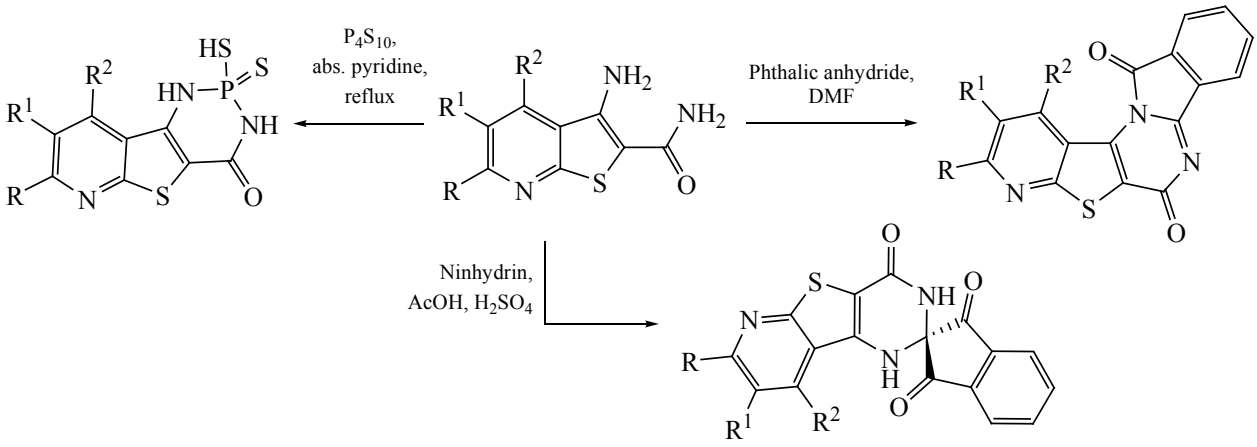
Scheme 19.7.



Scheme 19.8.



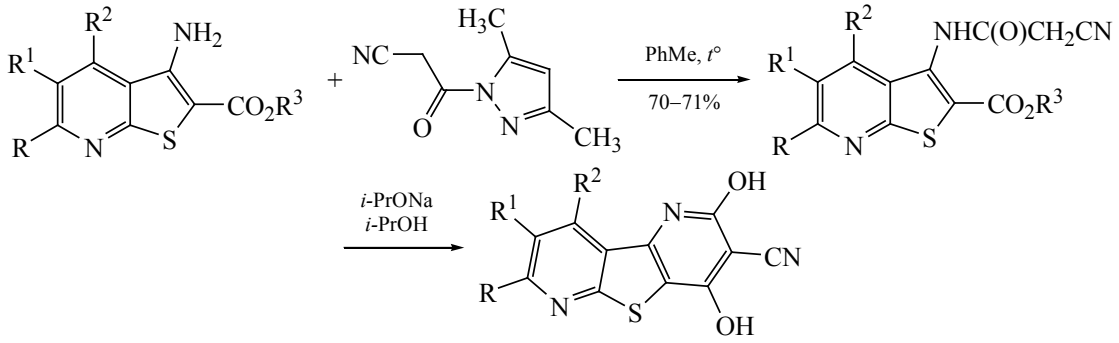
Scheme 19.9.



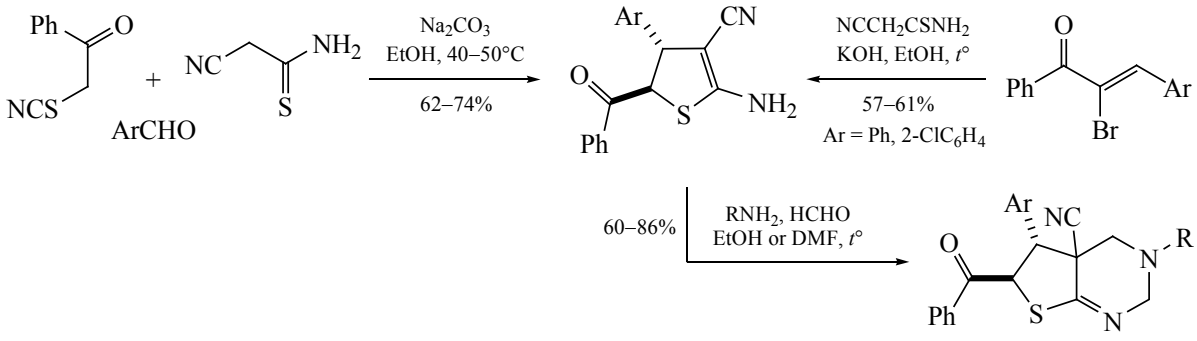
aminomethylation of C,S,N-nucleophilic heterocyclic substrates. Thus, a radically new method for constructing the bicyclic system of thieno[2,3-*d*]pyrimidine through the reaction of 2-amino-4,5-dihydrothiophene-3-carbonitriles with HCHO and primary amines was developed (Scheme 19.11) [975].

The behavior of 1,6-diaminopyridines in the Mannich reaction was studied to show that the

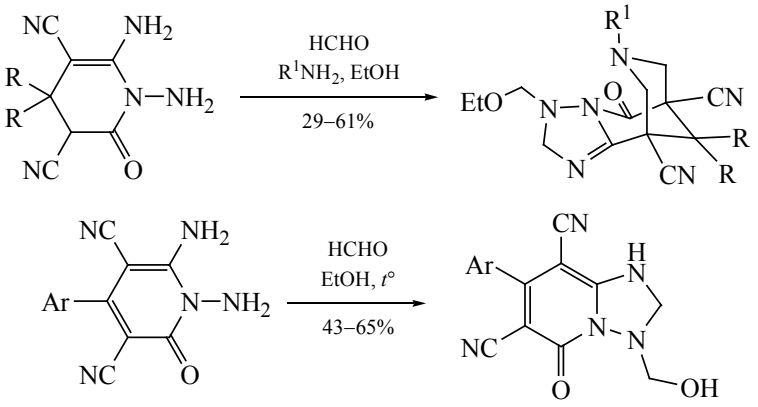
Scheme 19.10.



Scheme 19.11.



Scheme 19.12.



structure of the products is determined by the possibility of aminomethylation of the pyridine ring: this variant is realized in the case of partially saturated aminopyridines, and leads to bispydine derivatives fused with the 1,2,4-triazole ring [976]. If aminomethylation in the ring is impossible, only amino groups are involved in reaction, and the products are substituted triazolopyridines (Scheme 19.12) [977].

20. DEPARTMENT OF ORGANIC CHEMISTRY OF SAMARA STATE TECHNICAL UNIVERSITY: SYNTHESIS OF FUNCTIONALIZED FRAMEWORK DERIVATIVES. NON-AROMATIC HETEROCYCLES FORMED BY METAL-CATALYZED MICHAEL AND REDUCTIVE HECK REACTIONS

The Department of Organic Chemistry of Samara State Technical University works along several interpenetrating and complementary line of research, of which dominant and uniting most of the topics is the chemistry of framework compounds. An important place in the research work is also occupied by enantioselective metal complex catalysis. Both directions include the search for and study of the biological activity of the synthesized compounds and the creation of materials with a complex of valuable properties based on them.

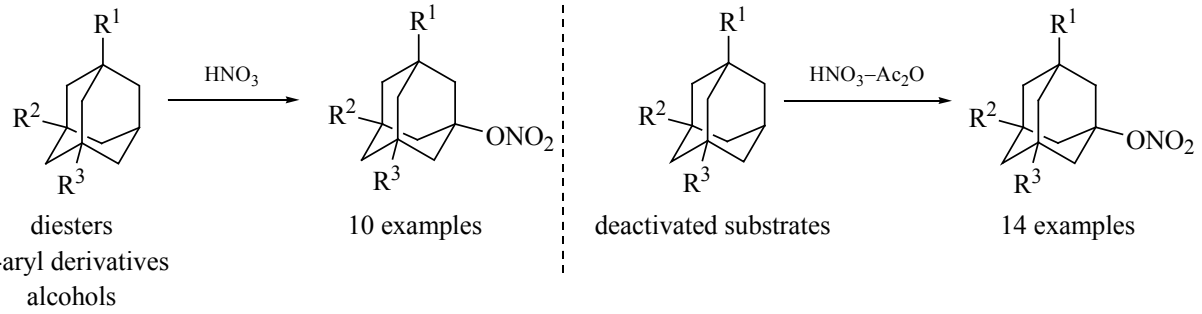
In the chemistry of framework compounds, adamantane plays a leading role due to its unique structure and physicochemical properties. The work biography of adamantane and its derivatives includes tens of thousands of compounds, and studies of their chemical properties are still relevant. The direction of research historically established at the department and involving the functionalization of framework compounds in a nitric acid medium continues to develop actively and has been replenished over the past 5 years by a vast amount of knowledge on the methods of synthesis of the synthesized compounds, as well as their nitroxylation chemoselectivity [978], reactivity, and chemical properties.

A number of new framework nitroxy derivatives have been synthesized using fuming nitric acid [979–983] and the HNO_3–Ac_2O system (Scheme 20.1) [984]. Substituted adamantane derivatives, including those containing acceptor groups, as well as the almost inert 1,3,5,7-tetramethyladamantane, were used as initial substrates. Nitroxylation in the HNO_3–Ac_2O system proceeds more selectively compared to HNO_3. Due to the high electrophilicity and lower acidity of this reaction medium, the stability of nitrates increases, the probability of alcohol formation strongly decreases, and in some cases nitrolysis and oxidation of functional groups in the initial substrates are suppressed.

Diamantane nitroxylation features the extreme hydrolytic instability of 1-nitroxydiamantane, as well as the relatively high contents of 1-nitrodiamantane and disubstituted products in the reaction mixture. Using the HNO_3-AcOH system, a one-pot synthesis of diamantane derivatives was carried out (Scheme 20.2) [982]. The intermediate 1-diamantanol nitrate gives substitution products, when external nucleophiles are added to the reaction mixture.

A similar approach was developed to obtain acylamines [985] and amino alcohols [986] derived from framework hydrocarbons (Schemes 20.3, 20.4). Both methods are quite versatile. At one of the stages of the synthesis of amino alcohols, too, conc. H_2SO_4 is added at one of the stages, which increases the concentration of the nitronium cation, increases the oxidation potential

Scheme 20.1.



of the H_2SO_4–HNO_3 system, and leads to oxidation at the adjacent bridgehead position.

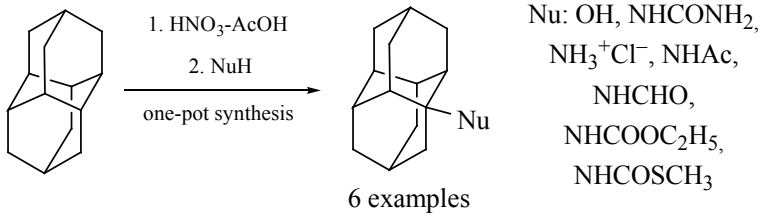
The use of the H_2SO_4–HNO_3 system has proven effective to prepare polyfunctional derivatives of the adamantane series containing substituents simultaneously in the bridgehead and bridging positions of the framework (Scheme 20.5) [987]. The reactions proceed through the stage of formation of a tertiary carbocation stabilized by the nucleophile that is the most abundant in the reaction mixture.

A significant contribution to the development of theoretical concepts was made by the study of the oxidation kinetics of deactivated framework substrates in the H_2SO_4–HNO_3system [988]. The reaction is described by a pseudo-first-order kinetic equation. The primary kinetic isotope effect was measured (2.9 ± 0.3). This study made it possible to answer the questions: *what is the preferred sequence of introducing carboxy- and carboxymethyl groups into the adamantine core and to what extent can the framework be deactivated by introducing electron-acceptor substituents without interfering with its successful functionalization?*

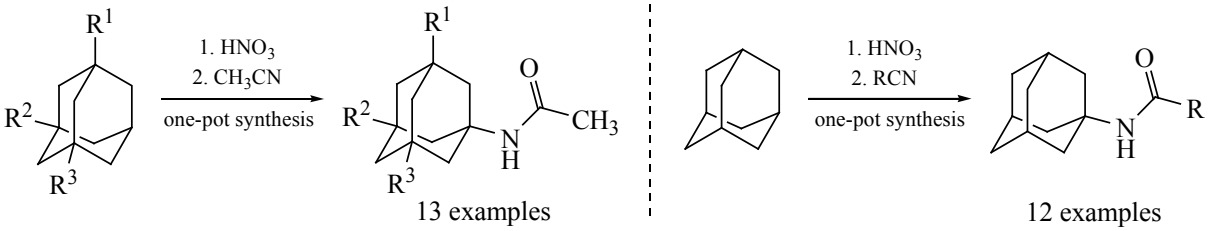
Traditionally, hydroxy derivatives and carboxylic acids of the adamantane series are used as the main molecular platform for preparing functionally substituted derivatives as the most accessible substrates. Much less attention is paid to halogenated adamantanes, most often they play the role of alkylating agents. The department's staff studied the transformations of halogenated adamantanes in a fuming nitric acid medium. The initial stage of the large-scale research was the kinetic study of nitrolysis of haloadamantanes [989].

Reactions of 1,3-dihaloadamantanes with fuming nitric acid proceed through a series of intermediate transformations: the nitrolysis of the starting dihalogen derivatives to form the corresponding nitroxy derivatives, followed by structural transformations of the framework in the latter, including Grob fragmentation and transannular cyclizations. These transformations give hardly accessible 2-oxaadamantane derivatives (Scheme 20.6) [990, 991].

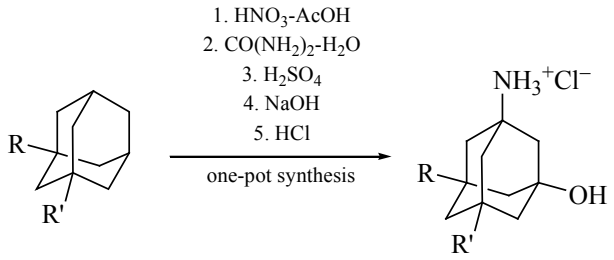
Scheme 20.2.



Scheme 20.3.

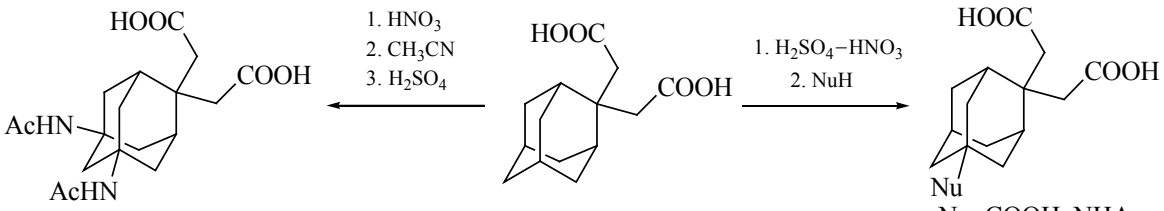


Scheme 20.4.



14 examples

Scheme 20.5.



Nu: COOH, NHAc

The reactions of 3-bromomethyl-5,7-dimethyl-2-oxaadamantan-1-ol in a 96% acid medium both in the presence and in the absence of nucleophiles were studied (Scheme 20.7). The reactions involve a series of structural transformations of the 2-oxaadamantane framework and open up ways to the synthesis of difficult-to-access of 1,2,3-trisubstituted adamantanes [992].

Nucleophilic substitution in conc. H_2SO_4 was used to obtain a wide range of functional derivatives with a framework structure from the synthesized nitroxide derivatives (Scheme 20.8) [993–995].

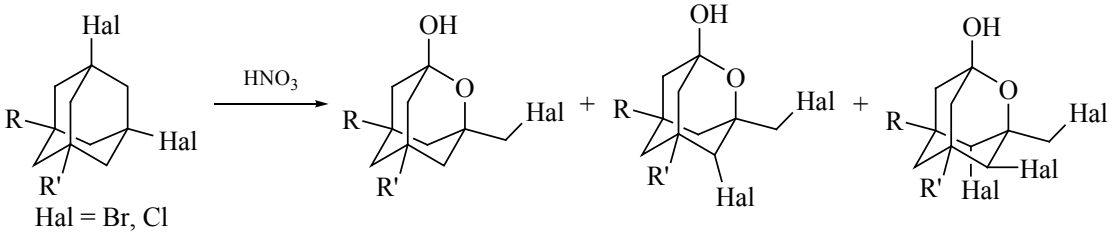
Approaches to framework substrates that have a high preparative potential as molecular platforms for the synthesis of potentially biologically active compounds and materials with a range of valuable properties were proposed (Scheme 20.9) [996, 997].

Another research topic of the department is the synthesis and chemical properties of heterocycles containing framework fragments. Based on the transformations of sterically hindered unsaturated adamantane derivatives, the possibility to prepare N,O,S-containing heterocyclic systems of different sizes was demonstrated (Scheme 20.10) [998, 999].

The direction of opening of three-membered rings in 1-adamantyl-substituted oxiranes and aziridines depends largely on the nature of the starting heterocycle. Under the action of nucleophiles, the oxirane ring opening gives rise to difunctional derivatives containing an adamantane fragment (Scheme 20.11) [1000]. Under the action of trifluoroacetic anhydride, 2-(adamantan-1-yl)aziridines undergo rearrangement to form a dihydro-1,3-oxazine ring fused with the homoadamantane framework. In some cases, amino alcohols acylated at the nitrogen atom are formed both individually and in a mixture with dihydro-1,3-oxazine. The direction of aziridine ring opening is determined by the structure of the starting substrate (Scheme 20.11) [1001].

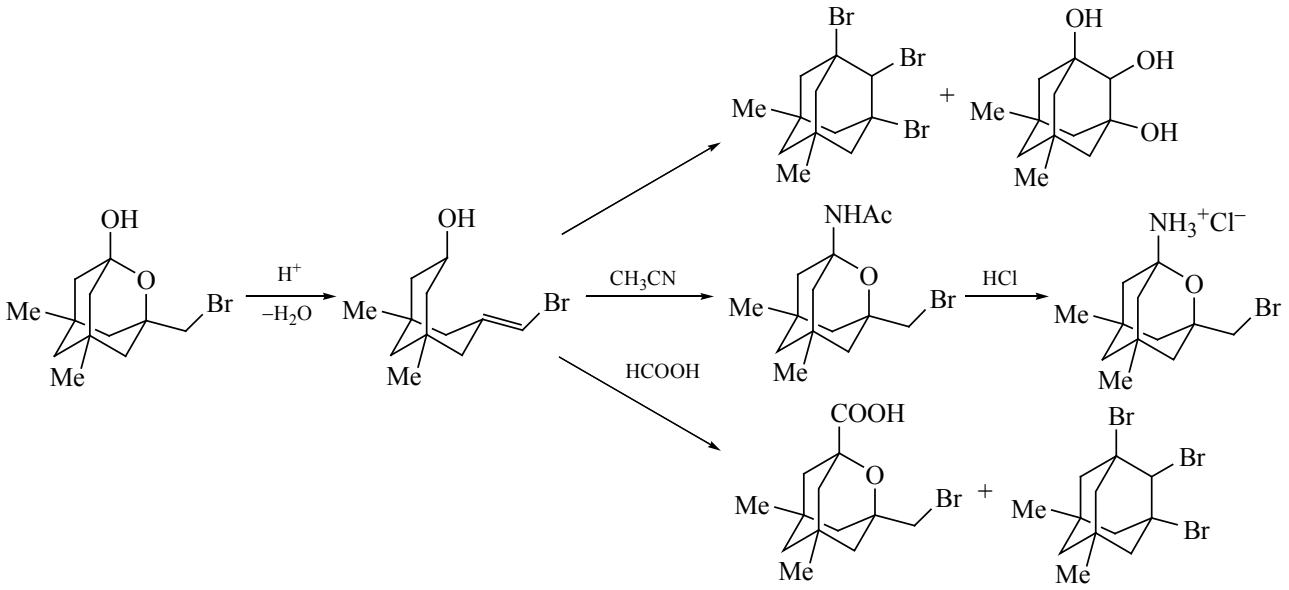
The reduction of 1-[(adamantan-1-yl)-2-oxoethyl]-pyridinium bromides gave 1-[2-(adamantan-1-yl)-2-hydroxyethyl]-1,2,3,6-tetrahydropyridines, which,

Scheme 20.6.



Hal = Br, Cl

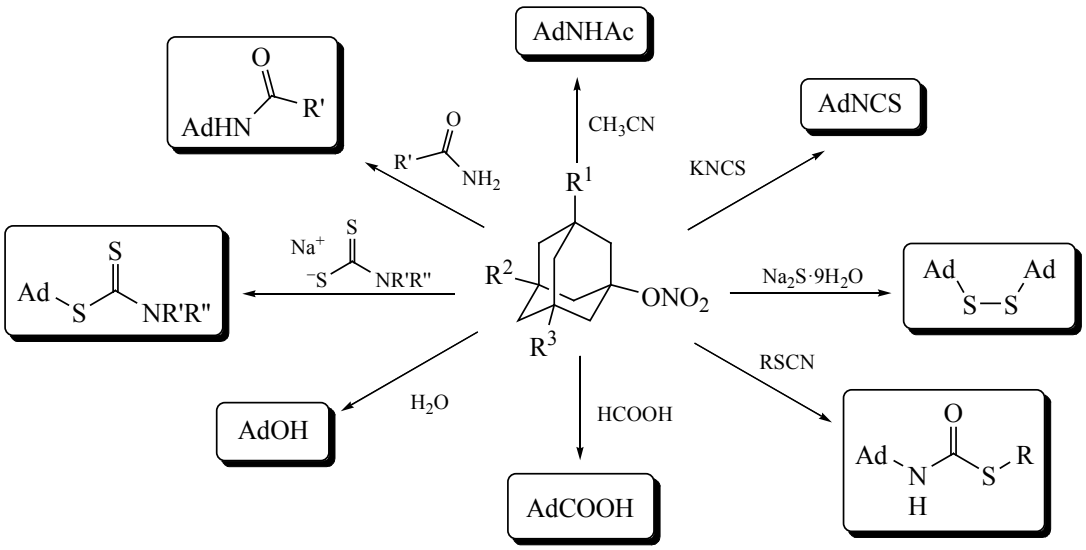
Scheme 20.7.



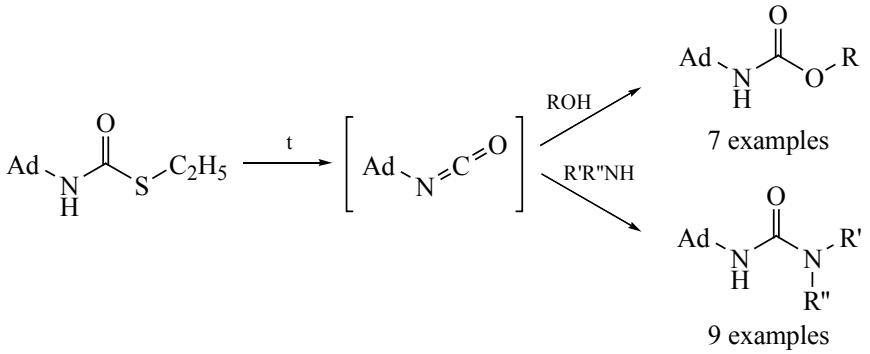
under the action of trifluoromethanesulfonic acid, undergo a carbocationic intramolecular cyclization accompanied by the allowed Wagner–Meerwein rearrangement to form substituted 1-azabicyclo[3.3.1]-non-3-enes annulated with the homoadamantane framework (Scheme 20.12) [1002]. 1-[2-Hydroxy-2-(4-R-phenyl)ethyl]-4-methyl-1,2,3,6-tetrahydropyridines and 1-phenethyl-1,2,3,6-tetrahydropyridines give azabicyclic and azatricyclic structures under the similar conditions [1003, 1004].

At present there are quite a few known homoadamantane compounds, which is largely due to their low synthetic availability. This problem has been successfully solved in recent years owing to the developed method of synthesis of ethyl 5-oxohomoadamantyl-4-carboxylate from readily available reagents. The synthetic attractiveness and potential of this molecule predetermined the development of this direction for many years, since it was just this molecule that made it possible to study properties typical of the chemistry of β-dicarbonyl compounds and obtain, in a few cases, completely unexpected results, may be due to the effect of the framework fragment. 4,4-Disubstituted homoadamantan-5-ones were synthesized by the reactions of ethyl 5-oxohomoadamantyl-4-carboxylate

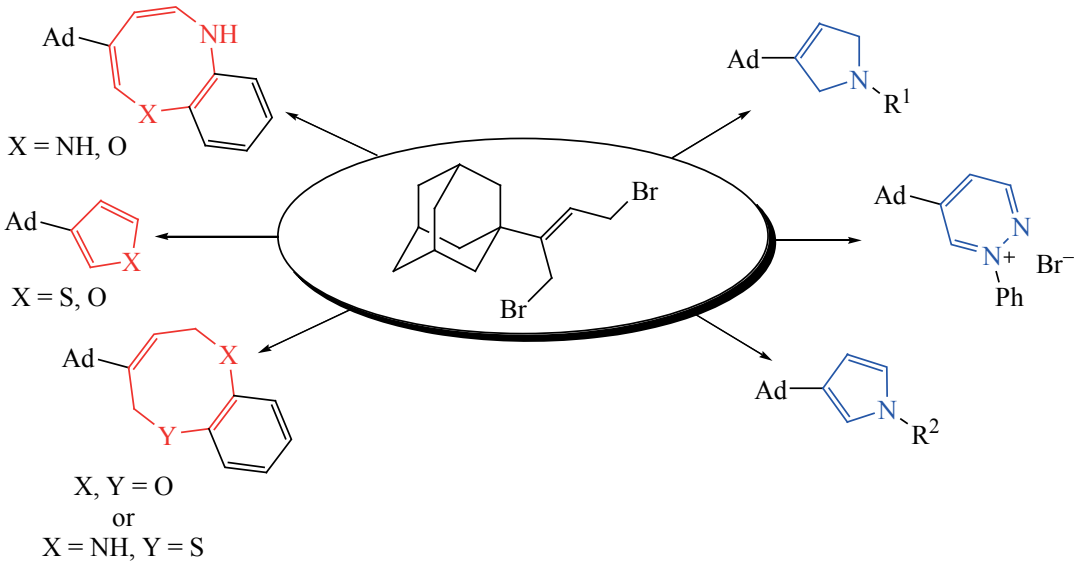
Scheme 20.8.



Scheme 20.9.



Scheme 20.10.

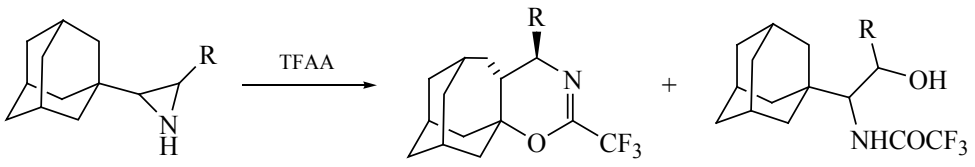


with carbon- and heteroatom-centered electrophilic agents. The reactions of ethyl 5-oxohomoadamantyl-4-carboxylate and its derivatives with dinucleophiles, as well as the Leuckart–Wallach reactions and hydrogenation gave a series of homoadamantane derivatives [4:5]-annulated to nitrogen-containing heterocyclic fragments (Scheme 20.13) [1005].

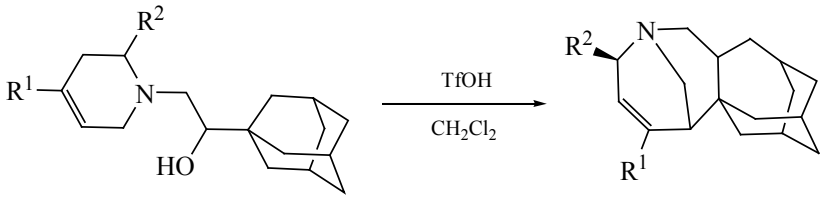
An unexpected acid-catalyzed 1,2-alkyl shift was discovered in a series of 4,4-disubstituted homoadamantan-5-ones. This retropinacol rearrangement leads to tetra- or pentacyclic mono- or bislactones containing a homoadamantane moiety. These transformations open up access to the synthesis of previously unknown 2,4-di- and 2,3,4-trisubstituted homoadamantane derivatives (Scheme 20.14) [1006].

The reactions of 4-substituted ethyl 5-oxohomoadamantyl-4-carboxylates with different nucleophilic agents were studied. The main result was "decarbethoxylation" instead of deacylation by the retro-Claisen reaction. Only one example of the preparation of the desired bicyclo[3.3.1]nonane derivative by the reaction of the α-nitroketo ester with ammonia was reported (Scheme 20.14) [1007].

Scheme 20.11.



Scheme 20.12.



4 examples

As a development of the line of research in the chemistry of bicyclic compounds, convenient synthetic approaches to 3,7-disubstituted derivatives of bicyclo[3.3.1]nonane using 2-adamantanone as the starting substrate were developed (Scheme 20.15) [1008, 1009] and the structural features of the conformational analysis of the synthesized compounds was performed.

Many of the synthesized framework compounds were tested for antiviral activity against hepatitis C virus and poxviruses. A significant number of compounds with pronounced activity were found [1010, 1011].

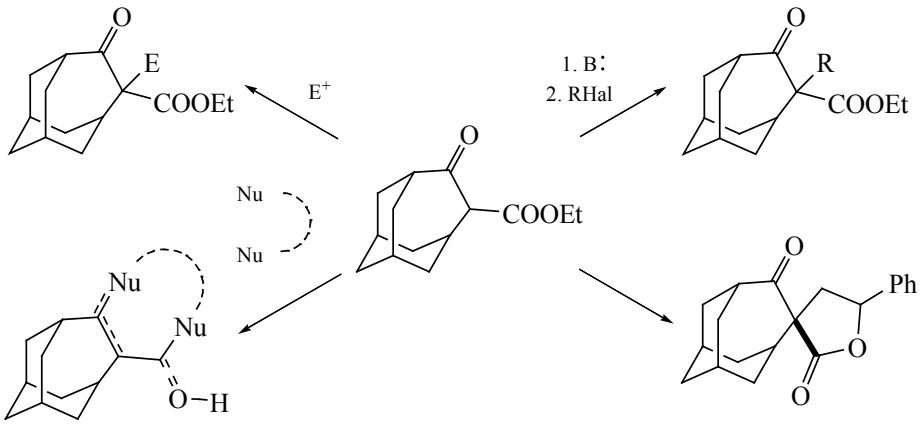
Most of the above studies were carried out during the COVID-19 pandemic, which complicated the creation of both new molecules and new structural types of molecules as potential antiviral agents. Molecular dynamics and molecular docking studies made it possible to refine the structure of the coronavirus helicase NSP13 and propose a number of potential inhibitors containing a framework fragment and clarify the probable mechanism of their action. The proposed approach is also suitable for constructing ligands that interact with other viral helicases [1012]. The reviews systematize information on low-molecular-weight compounds, including well-known pharmaceuticals and natural compounds that have high antiviral activity against coronaviruses [1013], and provide an analysis of the available information on known ion channels of viruses that cause various socially significant diseases [1014].

In collaboration with Volgograd State Medical University, studies were conducted and a series of articles on the neuropsychotropic and cerebroprotective properties of 1-adamantylpyrrolidin-2-ones were published [1015–1019]. Another promising application field of adamantane derivatives is the creation of oil bases for heat-stressed gas turbine engines [1020–1022].

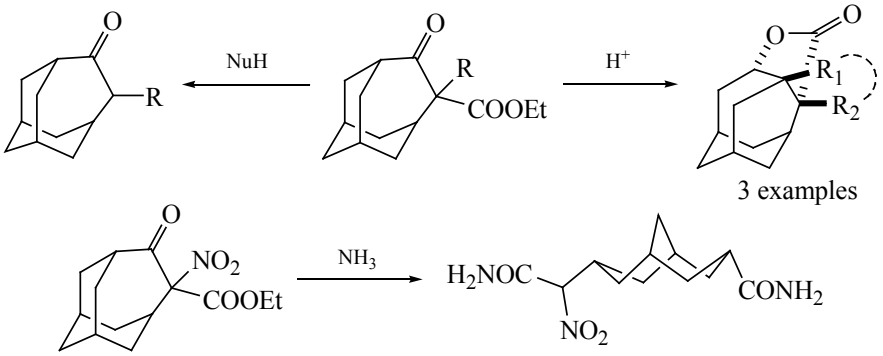
Metal complexes of chiral vicinal diamines containing framework fragments, such as adamantane and homoadamantane, are of undoubted interest as catalysts for asymmetric reactions. Within the framework of this line of research, convenient methods of synthesis of *N*,*N*-donor ligands of this type were developed, and the catalytic properties of their complexes in various asymmetric transformations were studied.

1-(Adamantan-1-yl)ethane-1,2-diamine was prepared in two steps from 1-(adamantan-1-yl)-2-azidoethan-1-one. The resulting racemic diamine was resolved using L-tartaric acid to obtained the *S* isomer (*ee* 96%) (Scheme 20.16) [1023].

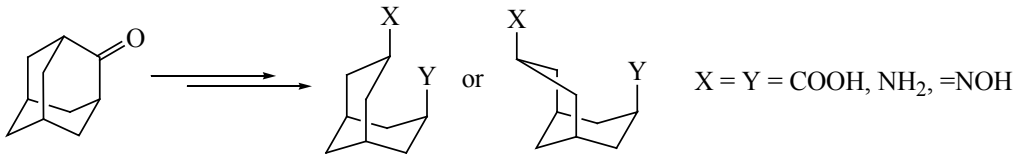
Scheme 20.13.



Scheme 20.14.



Scheme 20.15.



14 examples

The diastereodivergent synthesis of 1-(adamantan-1-yl)propane-1,2-diamine and 1-(adamantan-1-yl)-2-phenylethanediamine from azidoxime was carried out using different reducing systems. The racemic mixtures of *threo* and *erythro* isomers were resolved by crystallization of the diastereomeric salts with (*R*)-mandelic and L-malic acids, respectively, from aqueous alcoholic solutions (Scheme 20.17) [1024, 1025].

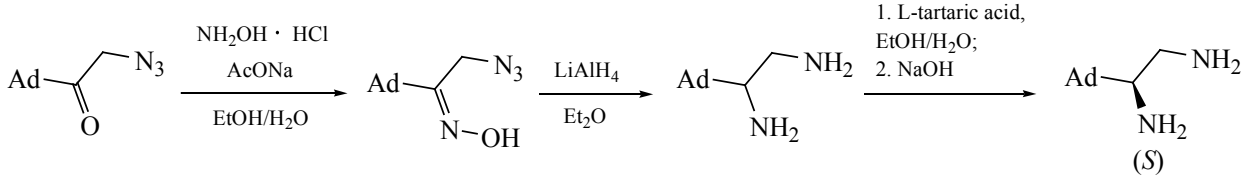
1,2-Diaminoadamantane was obtained from 2-oxoadamantoyl chloride via azidoxime formation, followed by the Curtius rearrangement, hydrolysis, and the Leuckart–Wallach reaction of 1-aminoadamantan-2-one. The *S* isomer was isolated by the crystallization of racemic 1,2-diaminoadamantane with L-tartaric acid (Scheme 20.18) [1026].

4,5-Diaminohomoadamantane was synthesized from homoadamantanone in several steps via the intermediate formation of the corresponding aziridine followed by aziridine ring opening and Red-Al reduction. The subsequent resolution of the racemic product was performed using dibenzoyl-L-tartaric acid (Scheme 20.19) [1025].

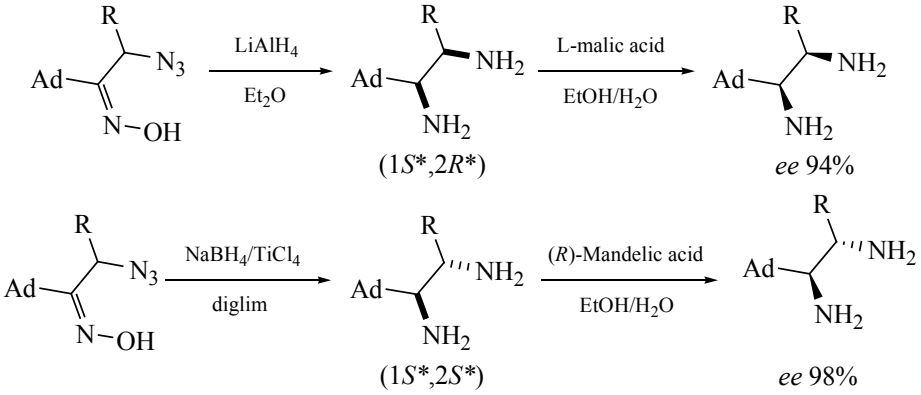
The synthesized vicinal diamines were used to obtain their *N*,*N*'-dibenzylidene and -dibenzyl derivatives, as well as salen-type ligands, as well as Ni(II) and Mn(III) complexes. The catalytic properties of these complexes were studied in model Michael addition of diethyl malonate to ω-nitrostyrene and also in styrene epoxidation. The most interesting result was obtained in the Henry reaction, where the use of the Cu(II) complex generated in situ from *N*,*N*'-dibenzylidene-1,2-diaminoadamantane led to the formation of the *R* isomer of the corresponding nitro alcohol with *ee* 64% (Scheme 20.20) [1026].

The asymmetric Michael reaction catalyzed by metal complexes is a convenient synthetic approach to nonracemic compounds, including those containing a few asymmetric centers. Possible approaches to the synthesis of such compounds are based on stepwise transformations involving chiral Michael adducts and on cascade reactions, when new asymmetric centers of a predetermined configuration are formed under the stereocontrol of those already formed at the Michael addition stage. The latest achievements in this area

Scheme 20.16.



Scheme 20.17.



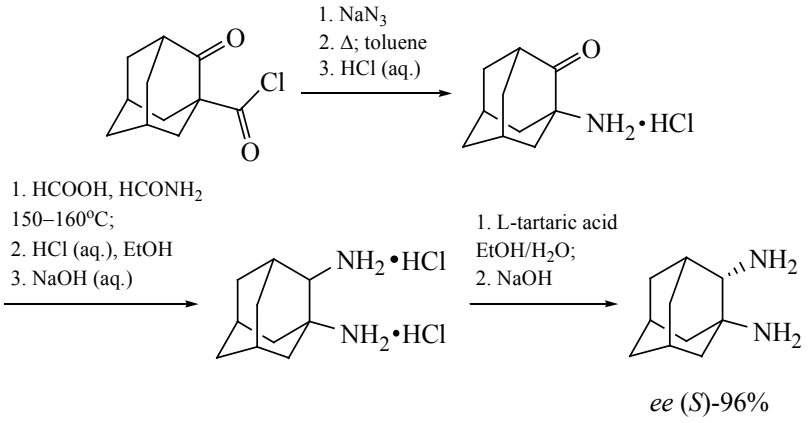
are summarized in our review [1027]. As part of the studies of the asymmetric Michael reaction, conducted at the Department of Organic Chemistry, a series of Ni(II) complexes with new chiral ligands based on (1*R*,2*R*)-1,2-diphenylethane-1,2-diamine, (1*R*,2*R*)-cyclohexane-1,2-diamine, and (1*R*,2*R*)-bicyclo[2.2.2]octane-2,3-diamine were synthesized, which showed a high catalytic activity in this reaction (Scheme 20.21) [1028, 1029]. The enantioselectivity of the addition of diethyl malonate to ω-nitrostyrene and 1-nitropent-1-ene in the presence of these catalysts reaches 96 and 91%, respectively. The reductive cyclization of the chiral adduct with 1-nitropent-1-ene yielded (4*R*)-4-propylpyrrolidin-2-one, a key intermediate in the synthesis of the antiepileptic drug brivaracetam (Scheme 20.22) [1029].

The regularities of the Ni(II)-catalyzed asymmetric addition of 1,3-dicarbonyl derivatives to nitroolefins were studied both by experimental methods and by DFT calculations. The calculation results provide evidence showing that the reaction route involves enolate formation, an intramolecular nucleophilic attack on the coordinated nitroolefin, and protonation of the resulting Ni nitronate complex. The calculated difference in the activation energies of enantiomer formation in the reaction of diethyl malonate with ω-nitrostyrene is close to the experimental values obtained from the temperature dependence of enantioselectivity. The calculations also indicate an important role the hydrogen bond between the amino group of the ligand and the nitro group of the Michael acceptor plays in ensuring the enantioselectivity of the studied reaction [1030].

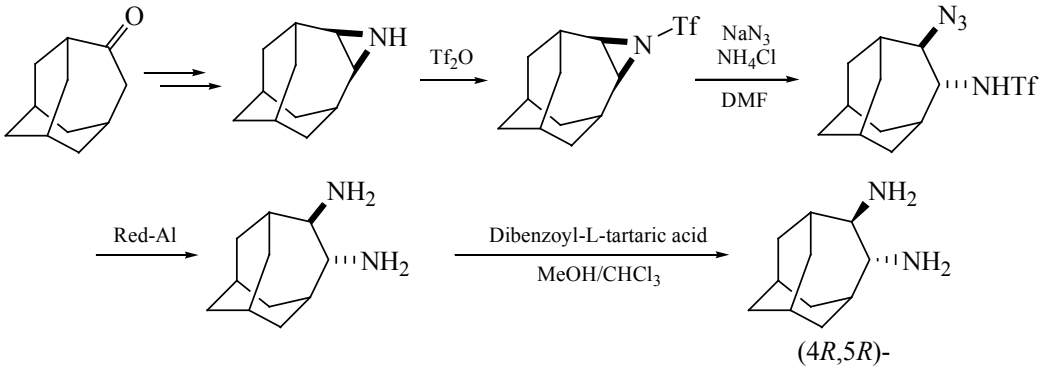
The nickel complex-catalyzed reaction of diethyl malonate with the sterically congested 1-(2-nitroethenyl)adamantane was used to prepare the *R* and *S* isomers of 4-(adamantan-1-yl)pyrrolidin-2-one and 4-amino-3-(adamantan-1-yl)butyric acid, which are of interest due to their potential neurotropic activity (Scheme 20.23) [1031].

Other 3-substituted GABA derivatives were obtained in a similar way. Tetrazole derivatives (Scheme 20.24) [1032] of (*R*)-phenibut, (*R*)-tolibut, and (*R*)-baclofen,

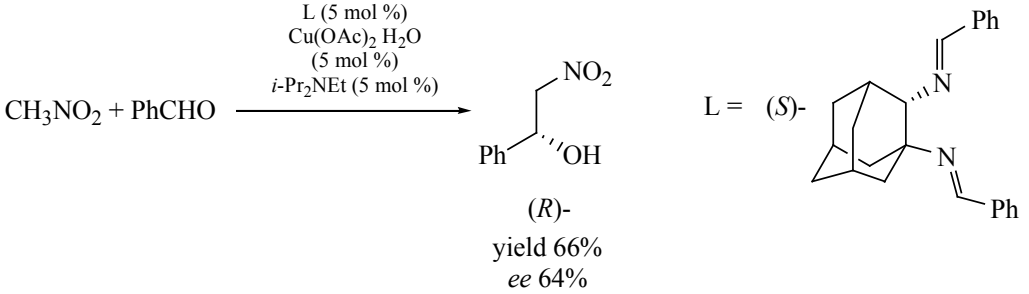
Scheme 20.18.



Scheme 20.19.



Scheme 20.20.



which showed moderate anxiolytic activity in animal experiments, were synthesized [1033].

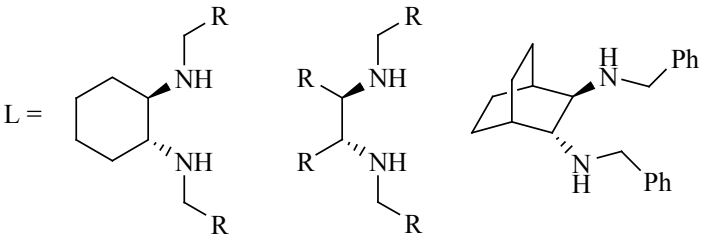
The Ni(II) complex of (1*R*,2*R*)-*N*,*N*-dibenzylcyclohexane-1,2-diamine is an effective catalyst for the addition of β-ketophosphonates [1034] and β-ketosulfones [1035] to nitroolefins, ensuring high enantioselectivity and, in many cases, diastereoselectivity of the asymmetric Michael reaction (Scheme 20.25).

Chiral adducts of β-ketophosphonates and nitroolefins were used to obtain a series of pyrrolidin-3-ylphosphonic acids as individual 2*R*,3*R*,4*S* isomers (Scheme 20.26) [1034].

Individual 2*S*,3*R*,4*S*,5*S*,6*R* isomers of tetrahydropyranylphosphonates were obtained by diastereoselective Henri/hemiacetalization cascade reactions of the chiral adduct of dimethyl (2-oxopropyl)phosphonate with ω-nitrostyrene and various aldehydes (Scheme 20.27) [1034].

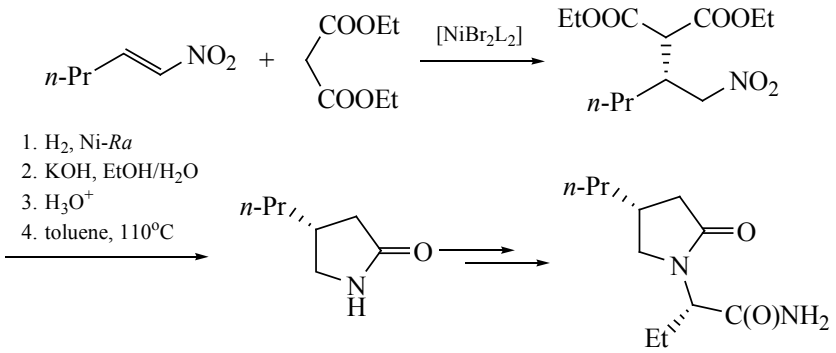
A method for the synthesis of nonracemic 2,3-dihydrofurans has been proposed based on the asymmetric addition of 1,3-diketones, β-ketoesters, and β-ketophosphonates to α-bromonitroolefins. The Michael reaction catalyzed by a Ni(II) complex with (1R,2R)-*N*,*N*'-dibenzylcyclohexane-1,2-diamine leads to the corresponding adducts as a mixture of diastereomers with the *S* configuration of the carbon atom bearing the R^3 substituent. The subsequent 5-*exo*-*tet*-cyclization of these adducts in the presence of 4-dimethylaminopyridine yields 2,3-dihydrofurans with *dr* from 9/1 and higher and up to *ee* 99% (Scheme 20.28) [1036].

Scheme 20.21.



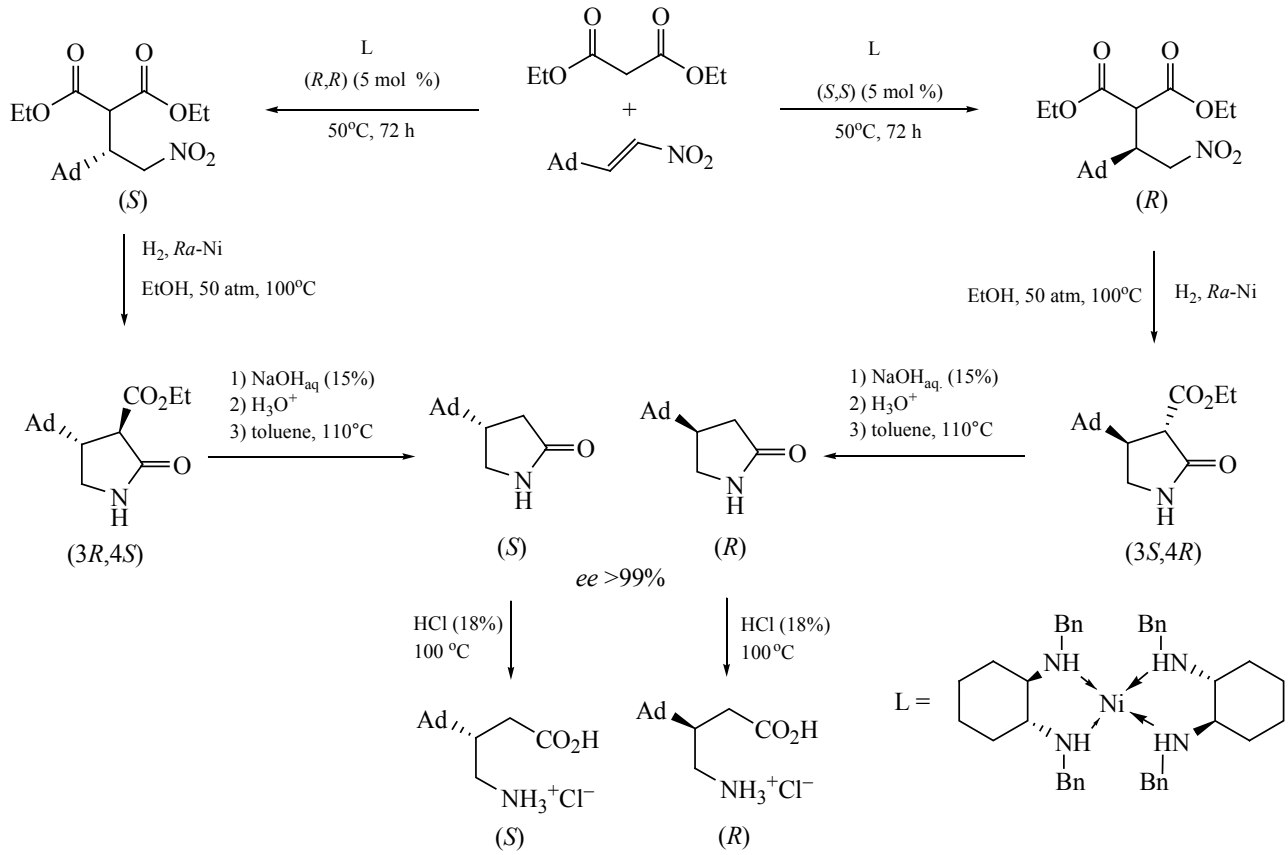
R = aryl, furan-2-yl, thiophen-2-yl, adamantan-1-yl

Scheme 20.22.

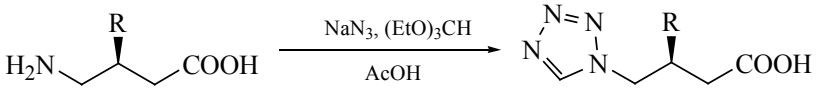


1,3-dicarbonyl compounds, such as 1-(adamantan-1-yl)butane-1,3-dione and ethyl adamantoylacetate were also involved in the asymmetric Michael reaction. A one-pot procedure for the synthesis from acetylacetone and 1,3-diphenylpropane-1,3-dione of 2,3-dihydrofurans as individual 4*R*,5*R* isomers with *ee* 94 and 96%, respectively. 2-Methyl-3-acetyl-4-phenylpyrrole was prepared in a yield of 90% by reduction of 4-acetyl-5-me-

Scheme 20.23.

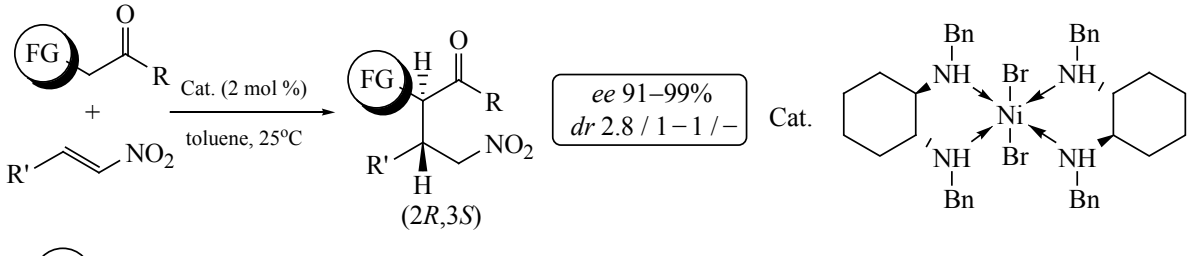


Scheme 20.24.



R = Ph, 4-$CH_3C_6H_4$, 4-ClC_6H_4

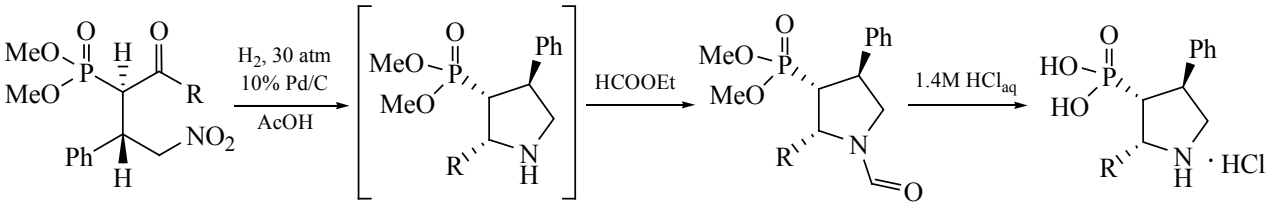
Scheme 20.25.



FG = $(MeO)_2P(O)$, $Ph(MeO)P(O)$, $PhSO_2$

R = Me, Ar; R' = Ar

Scheme 20.26.

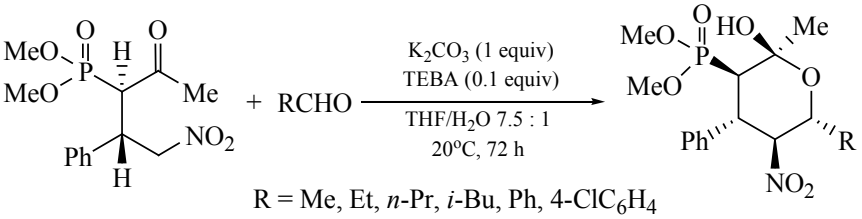


R = Ph, 4-$MeOC_6H_4$, 3-$MeOC_6H_4$, Me

thyl-2-nitro-3-phenyl-2,3-dihydrofuran (Scheme 20.29) [1036].

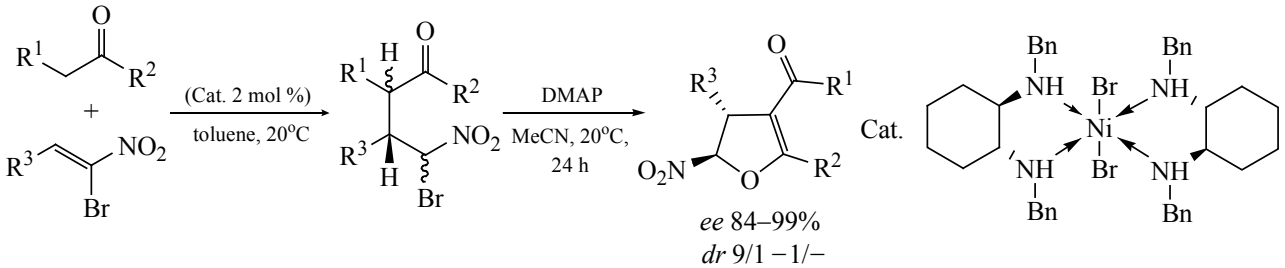
The intramolecular reductive Heck reaction provides a convenient synthetic approach to a wide variety of carbo- and heterocyclic compounds. The latest advances in this area are summarized in our recent review [1037]. In the context of studies on the Heck reductive reaction, conducted at the department, it was shown that nonracemic indanones can be obtained by cyclization of *ortho*-bromochalcones or similar triflate derivatives in the presence of $Pd(dba)_2$/chiral bisphosphine catalytic systems. The highest enantioselectivity was achieved with (*R*)-C_3-Tunephos as a chiral ligand (Scheme 20.30) [1038].

Scheme 20.27.



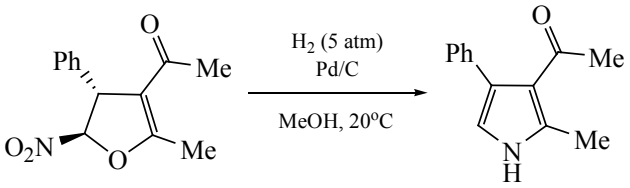
R = Me, Et, *n*-Pr, *i*-Bu, Ph, 4-ClC_6H_4

Scheme 20.28.

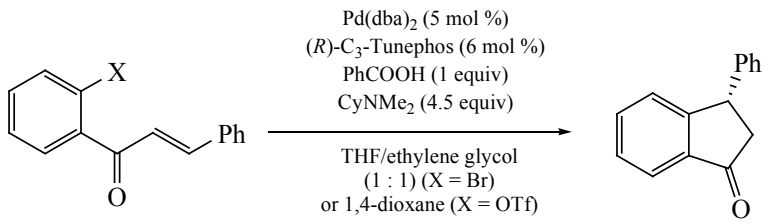


R^1 = MeC(O), 1-AdC(O), PhC(O), C(O)OEt, $(MeO)_2P(O)$; R^2 = Me, Ph, 1-Ad;
R^3 = Ph, 4-MeC_6H_4, 4-FC_6H_4, 5-bromofuran-2-yl

Scheme 20.29.

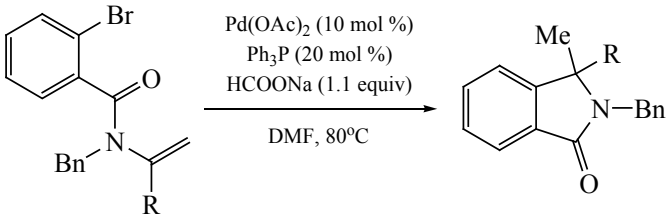


Scheme 20.30.



X = Br, OTf *ee* 76–83%

Scheme 20.31.



R = Ph, 4-MeC_6H_4, 3-$MeOC_6H_4$, 4-FC_6H_4, thiopne-2-yl

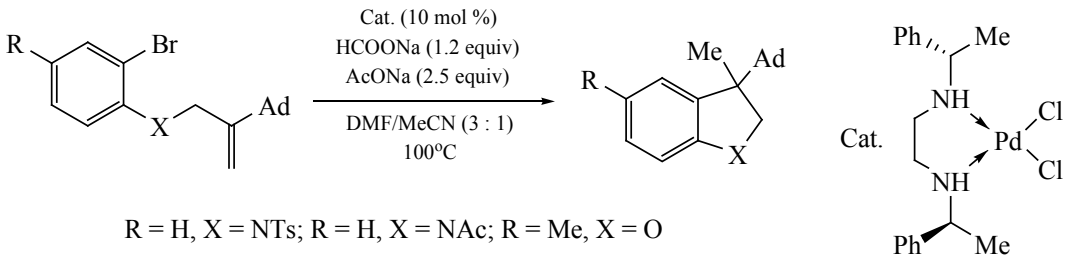
The intramolecular reductive Heck reduction of enamides serves as a convenient approach to the synthesis of isoindolin-1-ones. The use of the Pd(OAc)2/Ph3P catalytic system and sodium formate as a reducing agent allows the preparation of 3,3-disubstituted derivatives with various aromatic and heterocyclic substituents (Scheme 20.31) [1039].

Sterically hindered substrates with exo- and endocyclic C=C bonds, too, were involved in the intramolecular reductive Heck reaction. Thus, the corresponding adamantyl-substituted indolines and 2,3-dihydrobenzofuran were obtained by the cyclization of amides and esters containing a 2-(adamantan-1-yl)-allyl substituent (Scheme 20.32) [1040].

Reductive cyclization of 4-homoadamantenecarboxamide leads to the formation of a spiro derivative containing simultaneously homoadamantane and the oxindole structural fragments (Scheme 20.33) [1040].

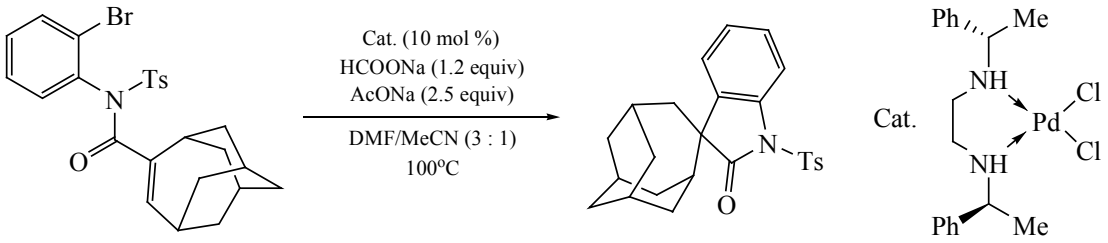
It is interesting to note that the complex of palladium with a vicinal diamine turned out to be an effective catalyst for these reactions. This is the first example of the successful use of a palladium complex with a diamine ligand as a catalyst for the reductive Heck reaction.

Scheme 20.32.



R = H, X = NTs; R = H, X = NAc; R = Me, X = O

Scheme 20.33.



Thus, the staff of the Department of Organic Chemistry of Samara Polytechnic University successfully works in the field of fine organic synthesis with strong emphasis on the development of new methodologies for the synthesis of heterocyclic and framework compounds and on aspects of medicinal chemistry and materials science. The widespread use of metal complex catalysis has made it possible to create simple synthetic approaches to structures combining heterocyclic and lipophilic framework fragments, as well as to enantiomerically enriched polyfunctional derivatives with several adjacent asymmetric centers of a predetermined configuration.

The review was prepared under financial support from the Russian Science Foundation (project. nos. 23-13-20029, 21-73-20096, and 21-73-20103).

21. DEPARTMENT OF ORGANIC CHEMISTRY OF NORTH CAUCASUS FEDERAL UNIVERSITY

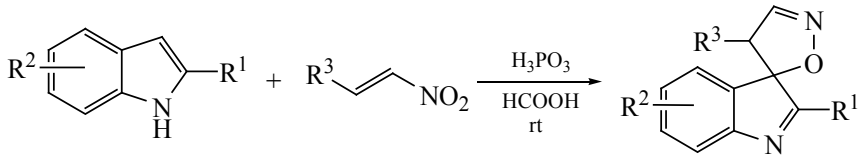
The main direction of scientific research of Stavropol organic chemists under the supervision of Prof. A.V. Aksenov is a study of the possibility to control cascade transformations using the properties of the reaction medium, by varying the reaction conditions, and by introducing modifiers. This approach was given the name "smart reaction media." Also, much effort is being focused on the development of radically new, efficient methods for modifying aromatic systems and preparing on the basis of the resulting systems of heterocyclic compounds with the aim to find new scaffolds and produce large libraries of compounds, primarily of potential use in medicinal chemistry. Surely, of particular interest in this contest are rigid, framework, and spirocyclic compounds, especially those containing important pharmacophoric fragments, among which indole derivatives attract the greatest attention.

The first of these methods is based on the use of nitroalkenes as synthetic equivalents of CCNO-type 1,4-dipoles for a highly diastereoselective formal [4+1]-cycloaddition reaction of indoles in phosphorous acid to obtain 4'*H*-spiro[indole-3,5'-isoxazole] derivatives. Along with ensuring the synthesis of previously unknown heterocyclic system, the transformation makes it possible to gain insight into new aspects of the chemistry of aliphatic nitro compounds (Scheme 21.1) [1041].

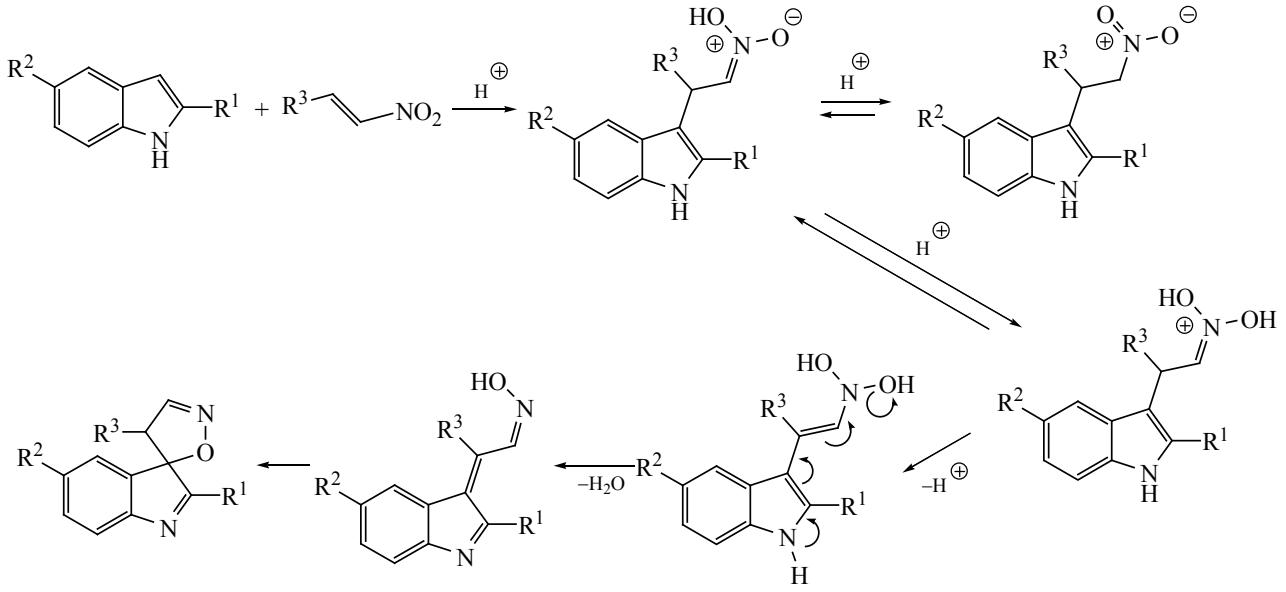
The reaction involves a sequence of Michael addition steps to form a phosphorylated *aci* form of the nitro compound, oxime formation, and 5-endo-trig cyclization, leading to the corresponding spiro compound. The most stable diastereomer is formed, and the key moment of the transformation, like in the Nef reaction, is the generation of a protonated *aci* form, because here, too, 2-(indol-3-yl)nitroethane is unreactive, which completely stops transformation (Scheme 21.2).

The reaction can also be carried out with β-alkyl nitrostyrenes, and in this case, a stronger Brønsted acid, such as MsOH, must be used instead of H_3PO_3 (Scheme 21.3) [1042]. The formation of 3,3'-(4-phenylmethylene)bis(2-phenyl-1*H*-indole) was obtained as a by-product.

Scheme 21.1.



Scheme 21.2.



The formation of the by-product is explained by the fact that *N*-alkylidene-*N*,*N*-dihydroxyammonium formed from the nitronate is in tautomeric equilibrium with *N*-alkyl-*N*-oxohydroxylammonium. The subsequent fragmentation gives rise to highly electrophilic (*E*)-3-alkylidene-2-methyl-3*H*-indole. In the presence of an excess of the slowly reacting starting indole, nucleophilic attack results in the formation of an alkylation product (Scheme 21.4).

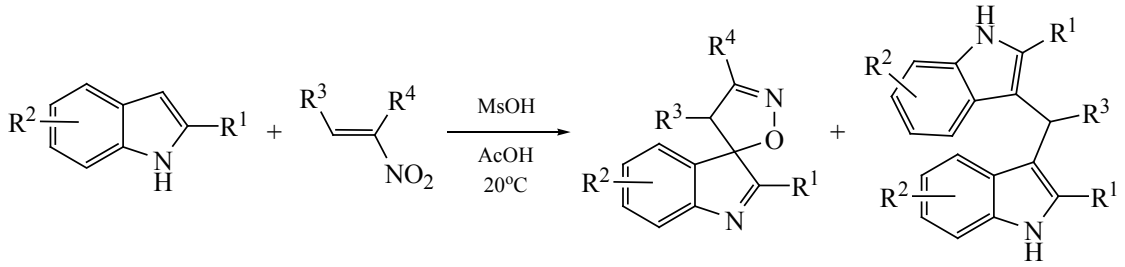
An alternative approach to 4'*H*-spiro[indole-3,5'-isoxazoles] is based on the generation of nitronates by the addition of Grignard reagents to 3-(2-nitrovinyl)indoles followed by Brшnsted acid–assisted spirocyclization. In this case, the process completely reproduces the Nef reaction: the *aci* form is added to HCl at 0°C, while a deviation from the procedure leads to C-protonation to form 1-nitro-2-(indol-3-yl)ethanes. The use of Grignard reagents derived from alkyl halides makes it possible to obtain difficult-to-access 4'-alkyl-substituted derivatives (Scheme 21.5) [1043].

The resulting spiro compounds are quite unstable and easily rearrange into 2-(3-oxoindolin-2-yl)-2-arylacetonitriles. For example, treatment of 4'*H*-spiro[indol-3,5'-isoxazoles] with triethylamine in alcohol produces the corresponding (3-oxoindolin-2-yl)acetonitriles via alpha-ketol rearrangement (Scheme 21.6) [1044].

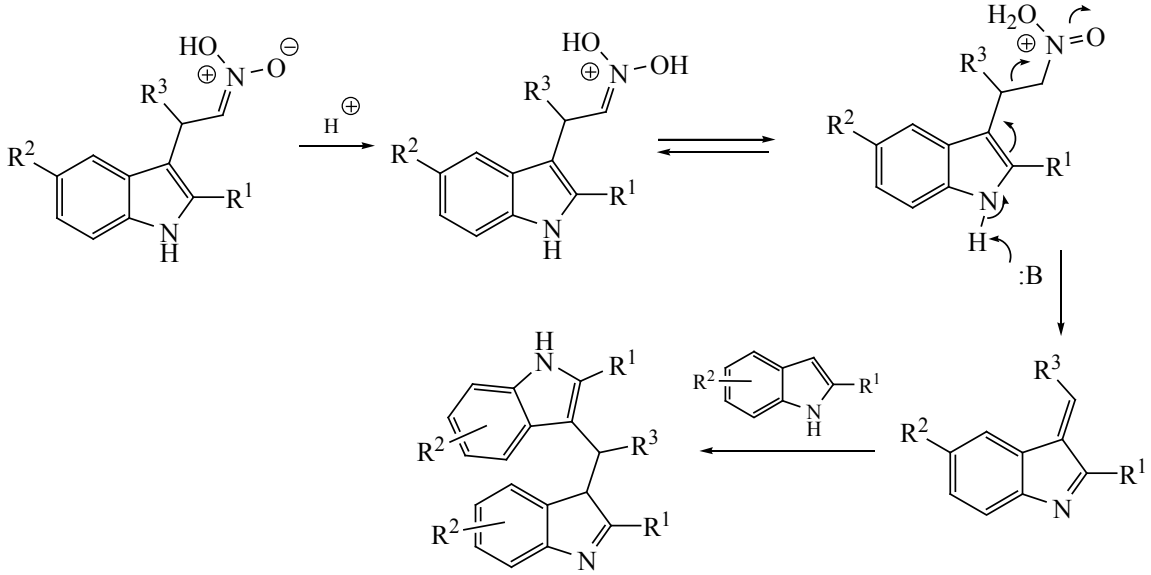
It is known that alpha-ketol rearrangements are also catalyzed by acids, and, therefore, (3-oxoindolin-2-yl)-acetonitriles can be directly synthesized by the reaction of indoles with nitrostyrenes in a mixture of H_3PO_3/EtOAc under reflux (Scheme 21.7).

As mentioned earlier, 1-nitro-2-(indol-3-yl)indoles are inert under the above reaction conditions [1045]. However, the use of $POCl_3/NEt_3$, a standard reagent for generating nitrile oxides from nitro compounds, makes possible the target spirocyclization. In this case, since a sufficiently large amount of base is present in the reaction mixture, the final product is 2-(3-oxoindolin-2-yl)-2-arylacetonitriles (Scheme 21.8). Initially, the reaction of indoles with nitroalkenes in acetic acid

Scheme 21.3.



Scheme 21.4.



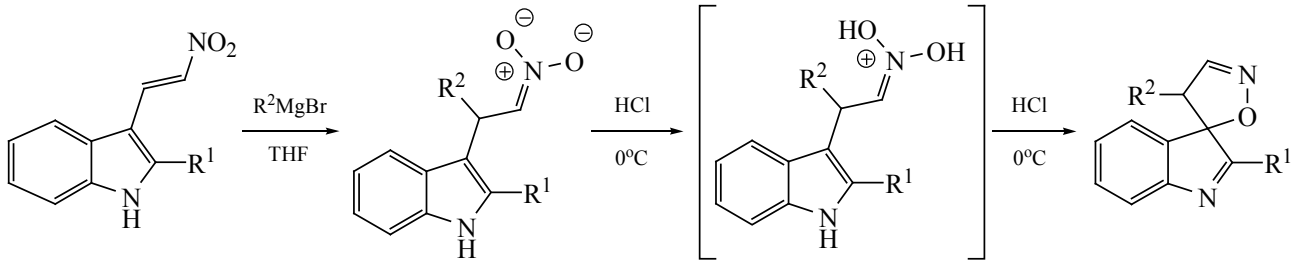
under reflux leads to the corresponding Michael reaction products. The isolated and purified nitroalkane is added to the $POCl_3/NEt_3$ mixture in benzene, which leads to spirocyclization followed by rearrangement.

Subsequent base-catalyzed transformations of 2-(3-oxoindolin-2-yl)acetonitriles are also possible. Acetonitriles with a free nitrogen atom lost an aryl acetonitrile molecule, and then a 1,2-aryl shift occurred to give 3-hydroxyindolin-2-ones. The reactions of *N*-alkyl derivatives of oxoindolines form 1,2,3,3a,4,8b-hexahydropyrrolo[3,2-*b*]indoles (Scheme 21.9) [1046].

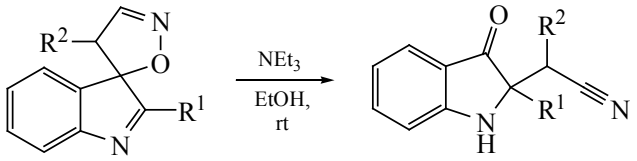
In a further development of this chemistry, 4'*H*-spiro[indole-3,5'-isoxazoles] were converted to 3-aminoindoles by the reaction with hydrazine hydrate under microwave irradiation. The transformation was accompanied by the loss of a benzyl cyanide molecule (Scheme 21.10) [1047].

The microwave-assisted reactions of 2-(3-oxoindolin-2-yl)-2-phenylacetonitriles with 1,2-phenylenediamines afford the corresponding quinoxalines as a single product. This transformation involves an unusual elimination of a phenylacetonitrile molecule and can be accomplished through a short reaction sequence, starting from readily available indoles and nitroolefins (Scheme 21.11) [1048].

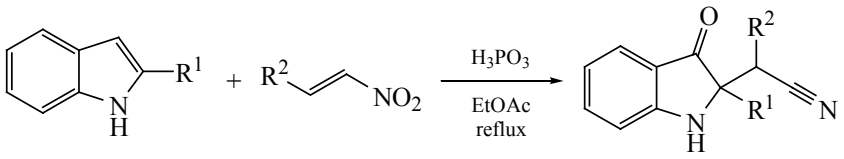
Scheme 21.5.



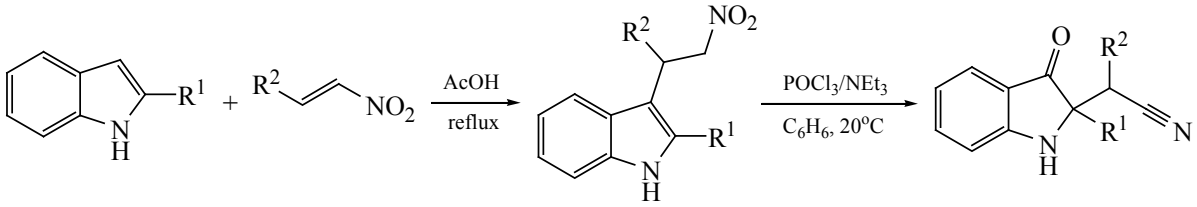
Scheme 21.6.



Scheme 21.7.



Scheme 21.8.



An unusual cascade transformation with ring opening and 1,2-alkyl shift is observed upon the reduction of 4'*H*-spiro[indol-3,5'-isoxazoles] or 2-(3-oxoindolin-2-yl)acetonitriles with sodium borohydride. This reaction allows facile and highly efficient preparation of 2-(1*H*-indol-3-yl)acetamides, which exhibit a high antiproliferative activity against a number of cancer cell lines (Scheme 21.12) [1049].

The related structures, 2-(3-oxoindolin-2-ylidene)-acetonitriles, can be prepared from ortho-nitrochalcones. This process involves Michael-initiated addition of the cyanide anion to the chalcone followed by a cascade cyclization like the Baeyer–Drewson reaction (Scheme 21.13) [1050].

A one-pot synthetic approach to 2-(3-oxoindolin-2-yl)acetonitriles is also possible, which consists of the base-catalyzed aldol condensation of *ortho*-nitroacetophenones followed by hydrocyanation triggering and reductive cyclization (Scheme 21.14) [1051].

Also, 2-(3-oxoindolin-2-ylidene)acetonitriles can be obtained from 4-(2-aminophenyl)-4-oxo-2-phenylbutanenitriles. The described transformation occurs via nucleophilic intramolecular cyclization and involves the oxidation of the aniline fragment with the KOH/DMSO system (Scheme 21.15) [1052].

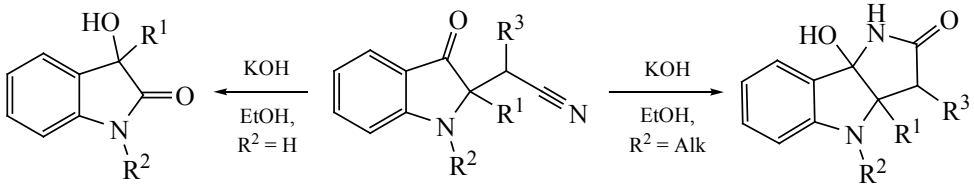
The same reagents in a polyphosphoric acid medium yield 2-aminofurans, which are then undergo recyclization to form 2-(1*H*-indol-2-yl)acetamides. This synthetic route opens up new possibilities for facile assembly of various isotryptamine derivatives for medicinal chemistry (Scheme 21.16) [1053].

The above-described philosophy of fine-tuning the reaction medium allows diversification of the reaction route. For example, 2'-nitrochalcones can be involved in a tandem reaction with aryl(hetaryl)acetonitriles, involving Michael addition followed by ipso-substitution of the nitro group occurs with high diastereoselectivity to form 1-tetralones with two adjacent chiral centers (Scheme 21.17) [1054].

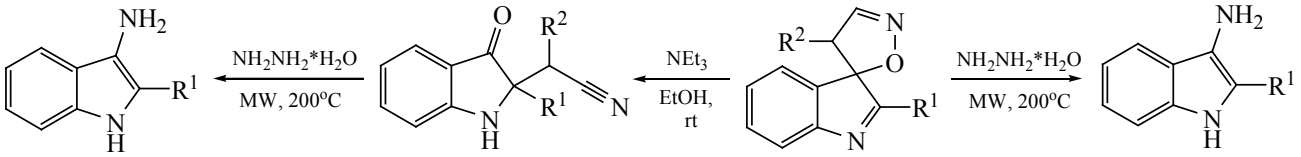
A related annulation reaction of 2'-nitrochalcones with potassium cyanide to form 1-indanones with a quaternary chiral center on C^3 is also possible (Scheme 21.18) [1054].

Another variant of the reaction conditions involves one-pot synthesis of 3-anilino-4-(het)arylmaleimides by heating an aqueous solution of 2'-nitrochalcones in DMSO with potassium cyanide in the presence

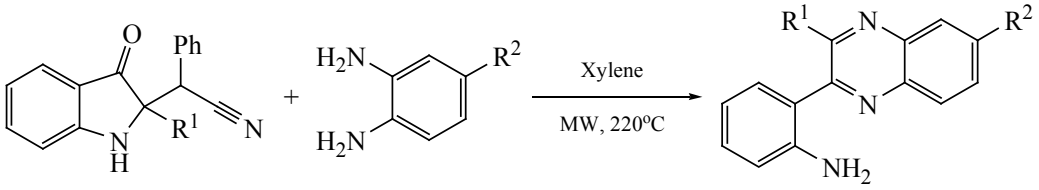
Scheme 21.9.



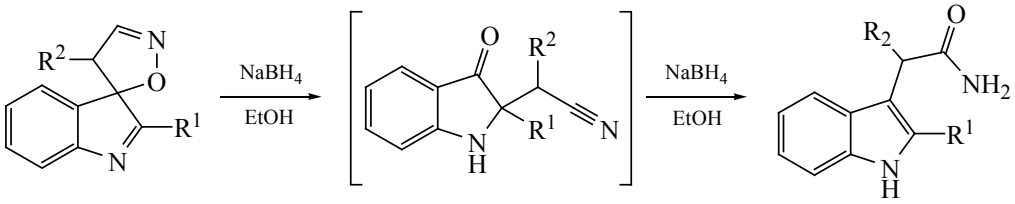
Scheme 21.10.



Scheme 21.11.



Scheme 21.12.



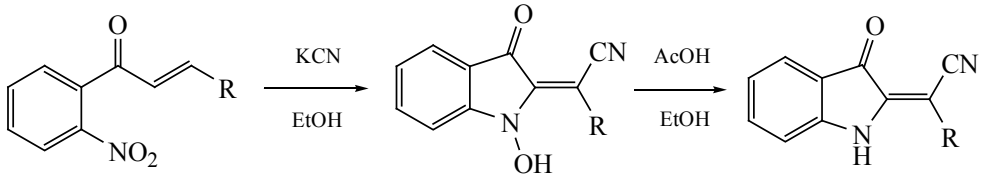
of formic acid (Scheme 21.19) [1055]. This reaction provides efficient access to a variety of β-substituted α-aminomaleimides, which have recently gained interest as small, easily modifiable, and sensitive fluorescent probes.

3,5-Diaryl-substituted 5-hydroxy-1,5-dihydro-2*H*-pyrrol-2-ones can be obtained from 3-cyano ketones by cyclization in the KOH/DMSO system (Scheme 21.20) [1056].

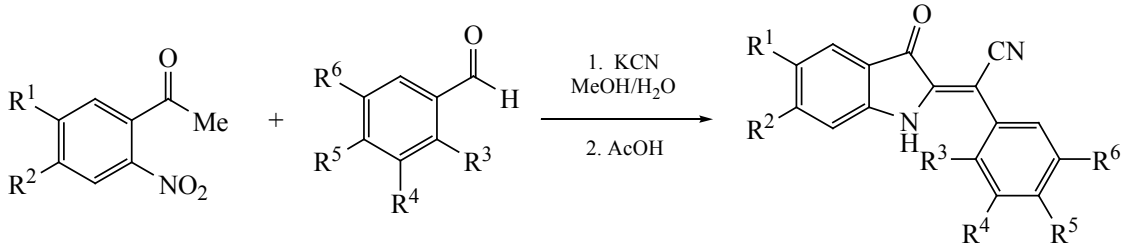
The subsequent modification of lactams is accomplished via their S_EAr reaction with aniline or phenol under heating in solvent-free conditions, which allows the introduction of the third aryl substituent into this heterocyclic scaffold, thus making it attractive in terms of the generation of various compound libraries for drug discovery (Scheme 21.21) [1056].

The condensation of β-cyanoketones with het(aryl)-aldehydes under basic conditions provided 3,5-di-aryl/heteroaryl-4-benzyl-substituted α,β-unsaturated γ-hydroxybutyrolactams, which are of considerable interest for synthetic organic and medicinal chemistry (Scheme 21.22) [1057].

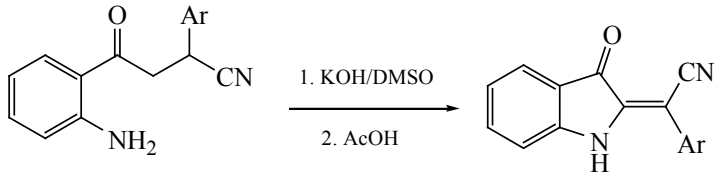
Scheme 21.13.



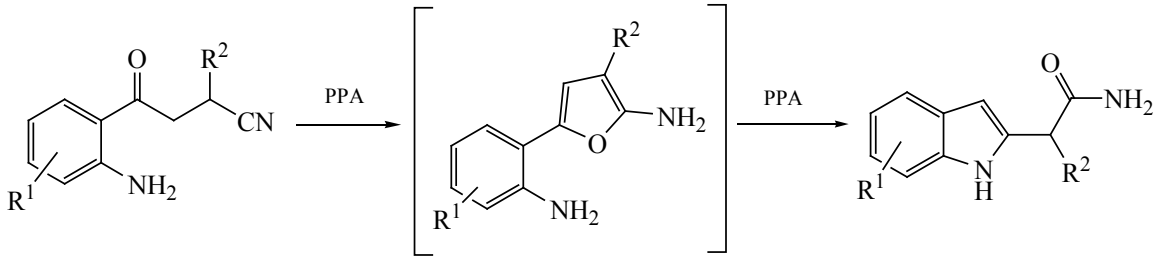
Scheme 21.14.



Scheme 21.15.



Scheme 21.16.



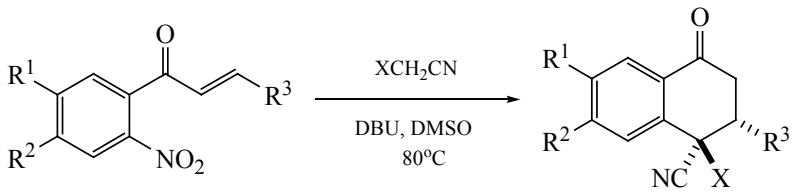
3,5-Diaryl-5-hydroxy-1,5-dihydro-2*H*-pyrrol-2-one, which, according to our previous findings, enters into the Friedel–Crafts reaction with anilines and phenols, can be functionalized with indoles to obtain 4-(indol-3-yl)butyramides (Scheme 21.23) [1058].

The intramolecular version of this reaction allows the synthesis of previously unknown polynuclear indole derivatives structurally similar to ergot alkaloids and, therefore, representing a new class of potential pharmacophores. Thus, the Knoevenagel condensation of indole-4-carbaldehyde with 2,4-diaryl-4-oxobutyronitrile leads to the formation of 4-[(1*H*-indol-4-yl)methyl]-5-hydroxy-3,5-diaryl-1,5-dihydro-2*H*-pyrrol-2-one, whose cyclization on further heating gives 7,9a-diaryl-2,6,9,9a-tetrahydro-8*H*-indolo[7,6,5-*cd*]-indol-8-one (Scheme 21.24) [1059].

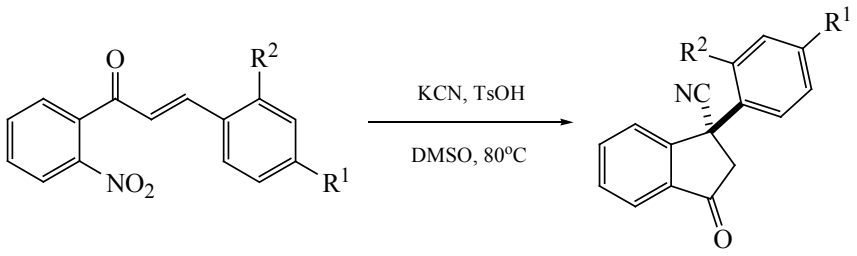
The alkaloid isocryptolepine and its synthetic analogs with a high anticancer activity can be obtained by a three-component polyphosphoric acid (PPA)-mediated reaction (Scheme 21.25) [1060]. This process involves the electrophilic activation of nitroalkanes to form C–C and C–N bonds, and this eliminates unnecessary operations and allows all steps to be carried out in one pot.

The reaction of 2-(2-aminophenyl)indole with nitrostyrene in a PPA medium unexpectedly affords analogous 11*H*-indolo[3,2-*c*]quinolines (Scheme 21.26) [1061]. Furthermore, the same product can be obtained in nearly the same yield, starting from hydrazone or phenylhydrazine, 2-aminoacetophenone, and nitrostyrene. The intermediate indole is formed in situ by the Fischer indolization reaction. In PPA, nitroalkanes

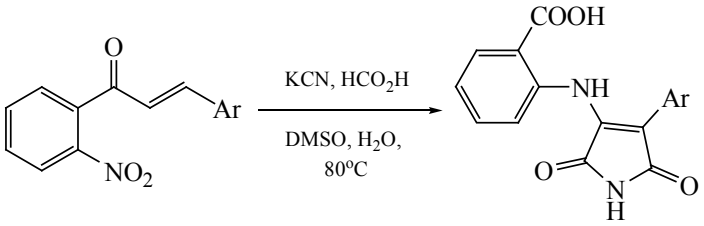
Scheme 21.17.



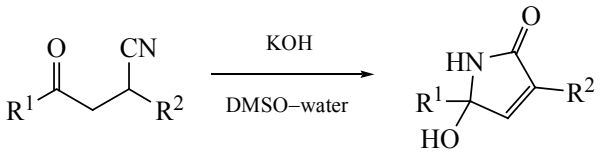
Scheme 21.18.



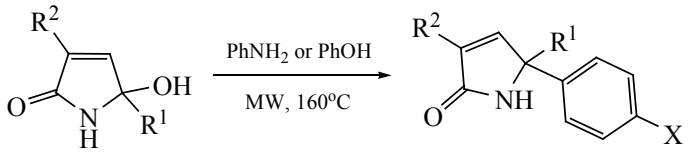
Scheme 21.19.



Scheme 21.20.



Scheme 21.21.



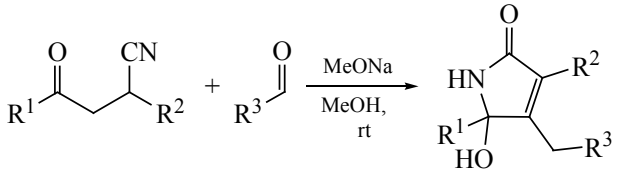
will be present in the phosphorylated *aci* form with a highly electrophilic C=N bond. An intramolecular N-Nef reaction involving the phosphorylated *aci* form occurs.

Benzofuro[2,3-*b*]quinolines, compounds with promising antidiabetic activity, can be obtained by reacting indole with *o*-methoxynitrostyrene. Initially, the reaction is carried out in a PPA medium to form a quinolone, and then pyridine is added to the reaction mixture to form the target product (Scheme 21.27) [1062].

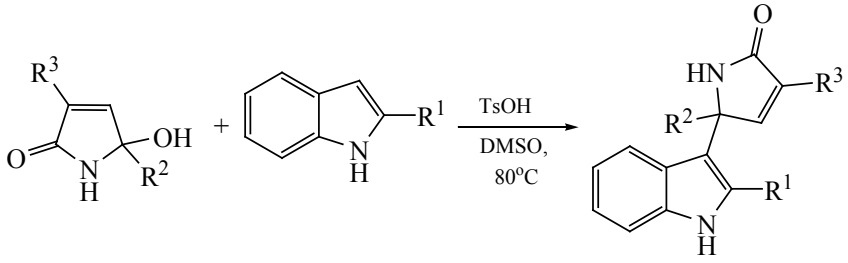
The alkaloid norneocryptolepine, an analog of benzofuro[2,3-*b*]quinolines, was obtained from 1-nitro-2-(2-nitrovinyl)benzene (Scheme 21.28) [1062]. Initially, the electrophilic alkylation of indole occurs, followed by the rearrangement to quinoline. The subsequent reduction of the aromatic nitro group to the amino group leads to ring closure.

When the methoxy group was replaced by a hydroxyl group, instead of the expected rearrangement to 2-quinolone, a 5-*exo*-trig cyclization involving the nucleophilic attack of the phenolic hydroxyl on

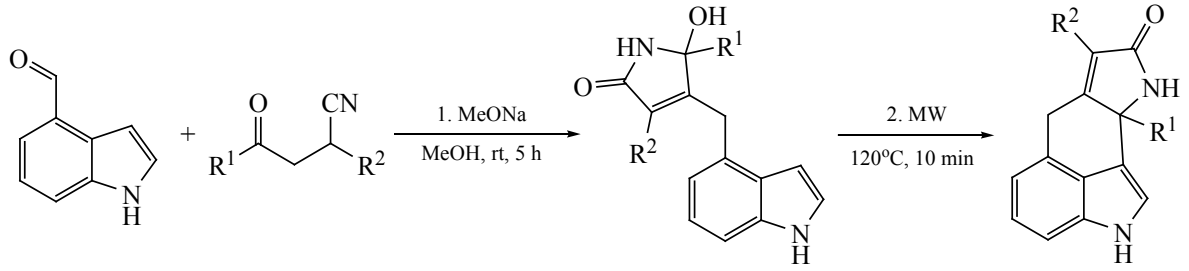
Scheme 21.22.



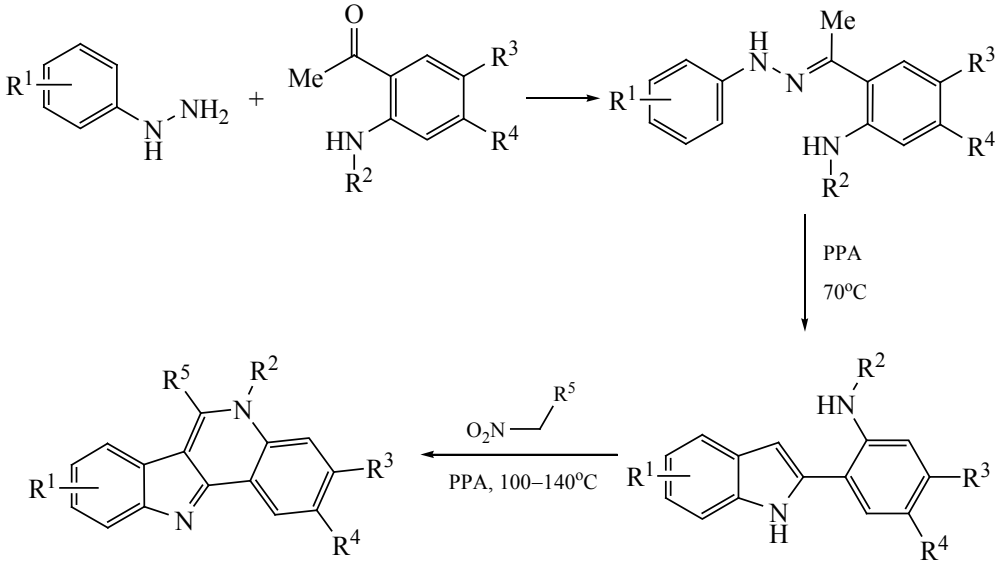
Scheme 21.23.



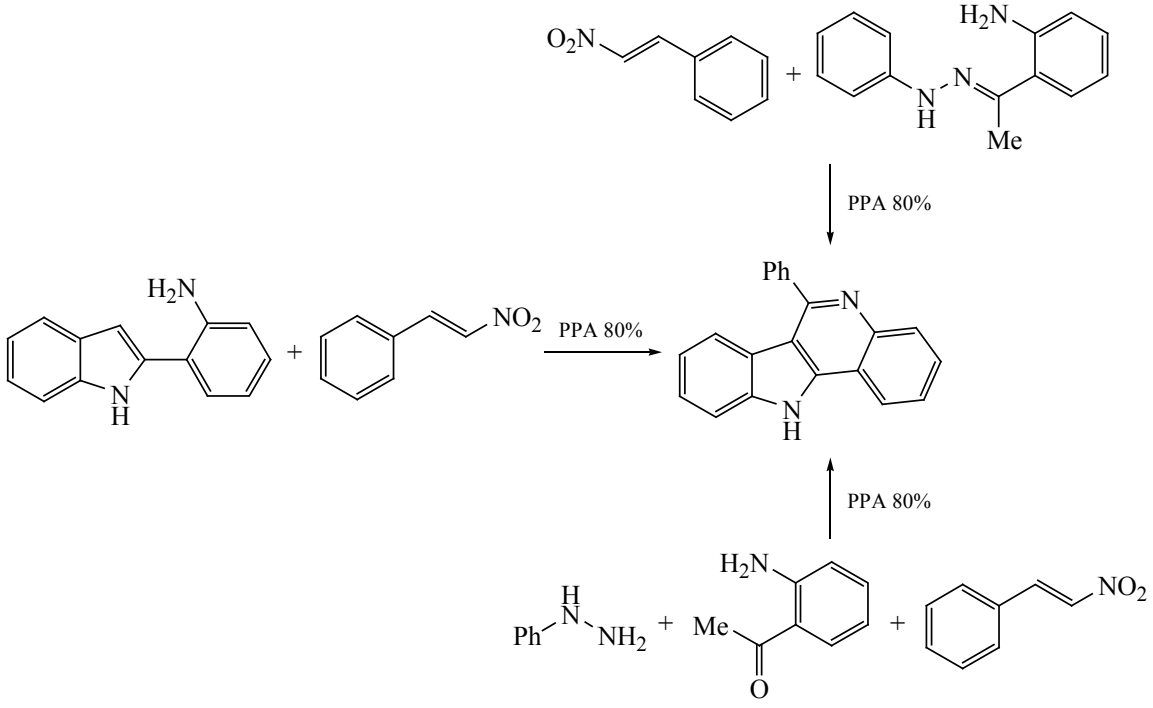
Scheme 21.24.



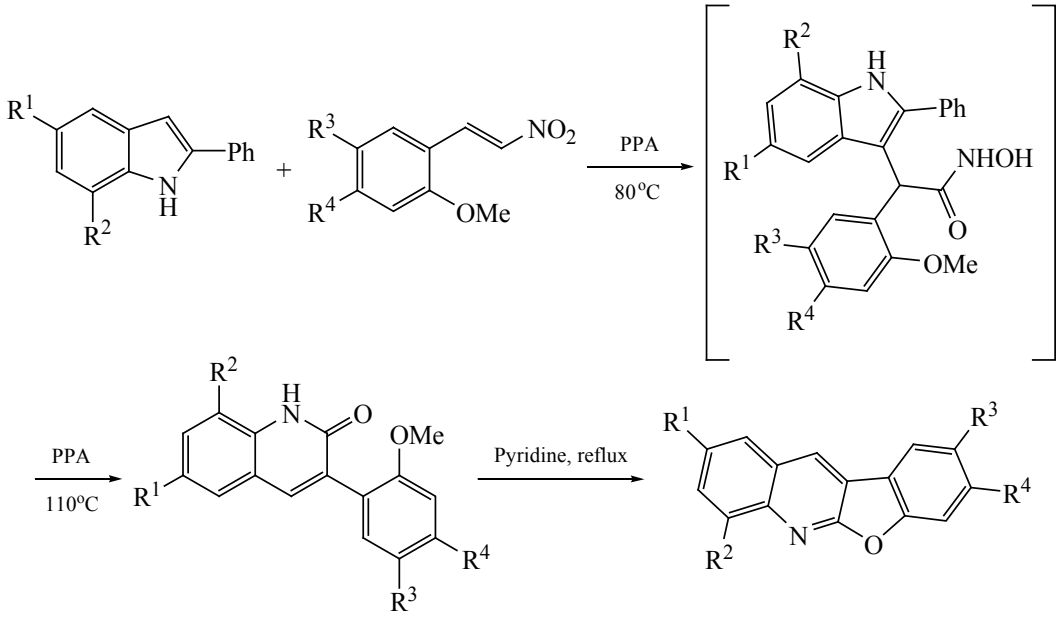
Scheme 21.25.



Scheme 21.26.



Scheme 21.27.



the carbonyl group of hydroxamic acid took place. As a result of this process, benzofuranone oxime was obtained, whose hydrolysis gace rise to benzofuranone (Scheme 21.29) [1063].

The same transformation can also be accomplished by the reaction of 3-(2-nitrovinyl)-1*H*-indole with phenols in methanesulfonic acid (Scheme 21.30) [1063].

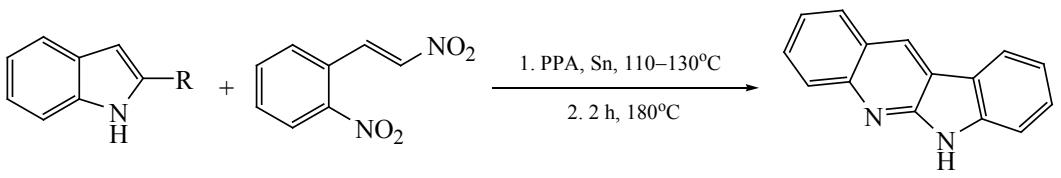
7-Aryl-substituted paullone derivatives can be prepared in 3 steps. This scaffold is structurally similar to 2-(1*H*-indol-3-yl)acetamides, promising antitumor agents, and may therefore be useful for the development of a new class of anticancer drugs. Paullones can be prepared via the cyclization of readily available cyanoketones to keto lactams. To obtain the target keto lactams, we initially prepared the corresponding acids by refluxing in HCl, followed by cyclization step with a CDI/acetonitrile system (Scheme 21.31) [1064].

Fischer indolization occurred in 2 stages. First, 3-phenyl-3,4-dihydro-1*H*-benzo[*b*]azepine-2,5-dione and phenylhydrazine were reacted in ethanol in the presence of acetic acid. Then polyphosphoric acid (80% P_2O_5) was added to the reaction mixture, and the reaction was carried out at 70°C (Scheme 21.32) [1064].

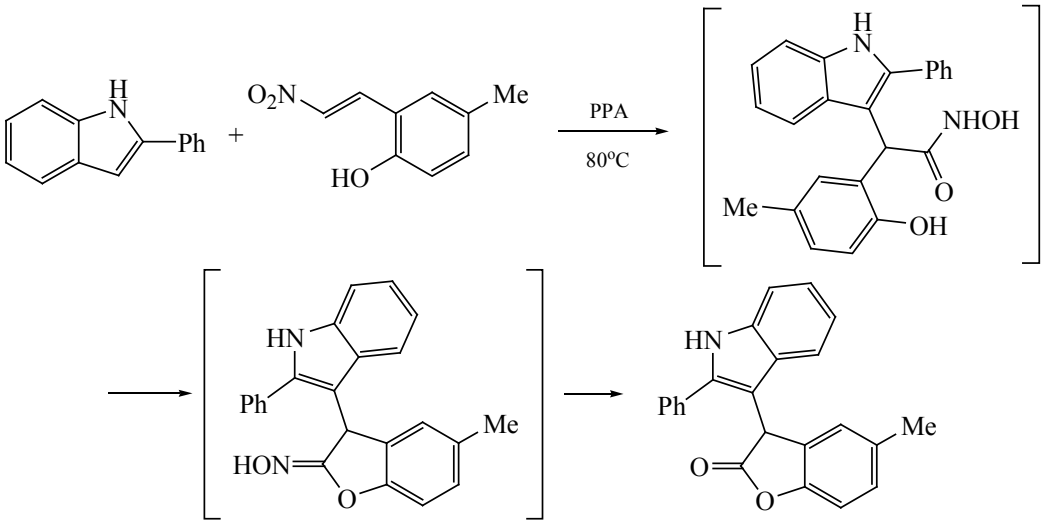
The microwave-assisted cascade transformation involving the electrocyclic reaction of the *aci* (alkylideneazinic acid) forms of nitrovinylindoles acting as heterotrienes leads to the formation of β-carbolines, including several natural products, the alkaloids norharmane, harmane, and eudistomin N. This type of reactivity was not previously known for nitro compounds (Scheme 21.33) [1065].

Nitroalkanes activated by PPA can serve as effective electrophiles in reactions with amines and hydrazines, which allows various cascade transformations to be carried out, involving a wide range of heterocyclic systems. 2-Amino-1,3,4-oxadiazoles and 2-amino-1,3,4-thiadiazoles were synthesized by the nucleophilic attack with semicarbazides or thiosemicarbazides of electrophilically activated nitroalkanes in the presence of PPA (Scheme 21.34) [1066].

Scheme 21.28.



Scheme 21.29.



The chemoselective nucleophilic attack with α-amino acid hydrazides on nitroalkanes electrophilically activated in the presence of PPA leads to the formation of 1,3,4-oxadiazoles containing hydrophilic aminoalkyl substituents, which can enhance the solubility of these compounds in aqueous media and improve their bioavailability (Scheme 21.35) [1067].

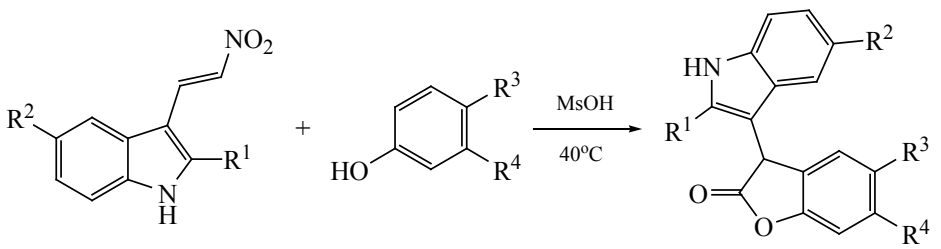
The cyclocondensation of acylhydrazides with nitroalkanes in PPA forms monosubstituted and asymmetrically disubstituted 1,3,4-oxadiazoles (Scheme 21.36) [1068].

Nitroalkanes activated with PPA can be used simultaneous or consecutive annulation of two different heterocyclic rings, which allows one to obtain [1,2,4]-triazolo[4,3-*a*]quinolines with 1,3,4-oxadiazole substituents (Scheme 21.37) [1069].

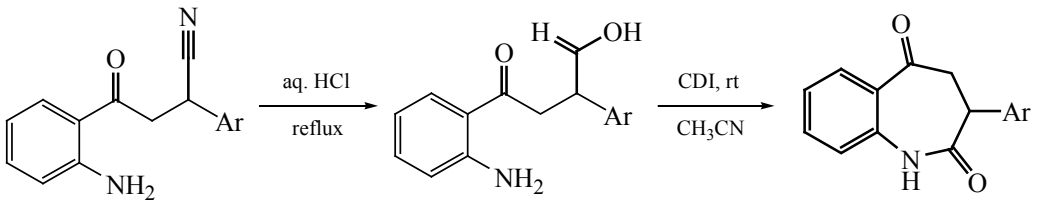
The electrophilic activation of nitroalkanes in the presence of PPA can, at least in some cases, provide nitrile oxides. In the presence of PPA, nitrile oxides tend to transform into hydroxamic acids, but for most stable electron-deficient dipolar species, typical [3+2]-cycloaddition reactions can be studied. Thus, dimerization can occur to form the corresponding 1,2,5-oxadiazole-2-oxides (furoxans) (Scheme 21.38) [1069].

A one-pot assembly of a bicyclic structural core comprising a furoxan fragment coupled to a pyridazine ring is possible. To this end, the nitro ketone was

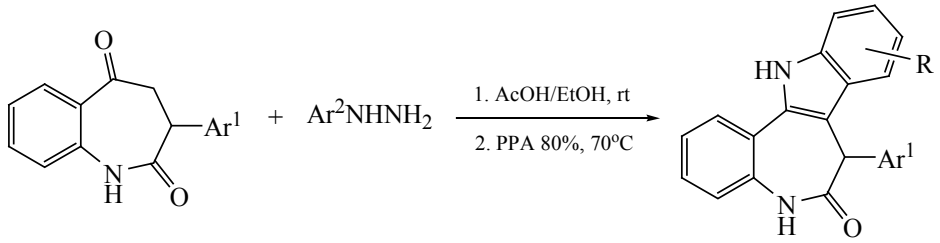
Scheme 21.30.



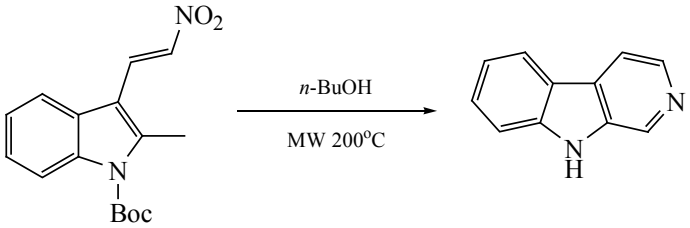
Scheme 21.31.



Scheme 21.32.



Scheme 21.33.



treated with PPA in the presence of hydrazine hydrate (Scheme 21.39) [1070].

The reactions of nitroalkanes with 2-hydrazinylquinolines, 2-hydrazinylpyridines, and bis-2,4-dihydrazinylpyrimidines in PPA give 1,2,4-triazolo[4,3-*a*]quinolines, 1,2,4-triazolo[4,3-*a*]pyridines, and bis-[1,2,4]triazolo[4,3-*a*:4',3'-*c*]pyrimidines, respectively (Scheme 21.40) [1071]. The reaction extends the possibilities of annulation involving phosphorylated nitronates, which are electrophilic intermediates formed from nitroalkanes in PPA.

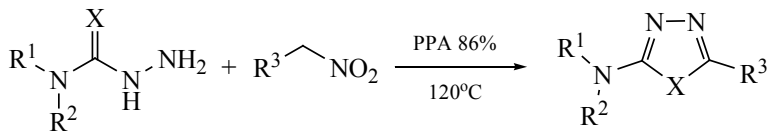
Imidazo[1,5-*a*]pyridines can be obtained by the cyclization of 2-picolylamines with PPA-activated nitroalkanes (Scheme 21.41) [1071].

Imidazolines are formed by the reaction between nitroalkanes and aliphatic 1,2-diamines in the presence of phosphorous acid (Scheme 21.42) [1072].

Nitroalkanes activated by PPA act as effective electrophiles in reactions with nucleophilic amines, which allows the synthesis of various heterocycles. This approach was used to prepare 3,4-dihydroquinazolines from readily available 2-(aminomethyl)anilines (Scheme 21.43) [1073].

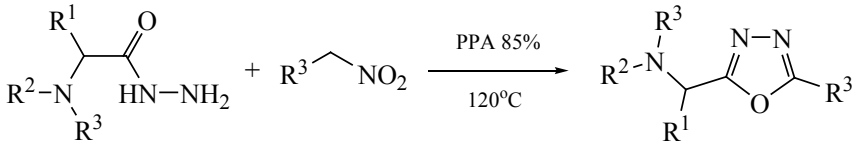
An effective preparative synthetic approach involving the [3+2] cycloaddition of pyridinium ylides to 1-chloro-2-nitrostyrenes has been developed and used to prepare a number of heterocyclic compounds,

Scheme 21.34.

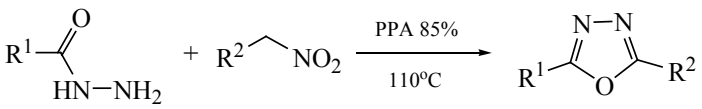


X = O, S

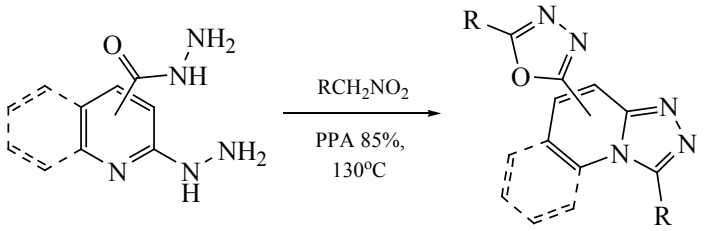
Scheme 21.35.



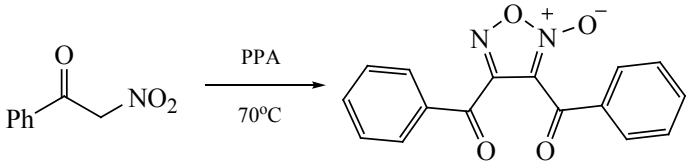
Scheme 21.36.



Scheme 21.37.



Scheme 21.38.



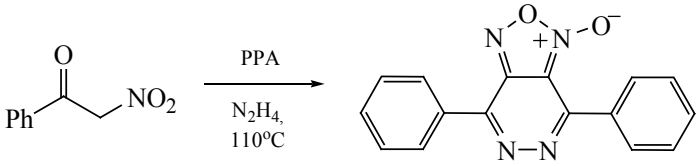
including indolizines and pyrazolo[1,5-*a*]pyridines (Scheme 21.44) [1074].

Section 22 was prepared with the financial support of the Ministry of Science and Higher Education of the Russian Federation (project no. FSRN-2023-0005).

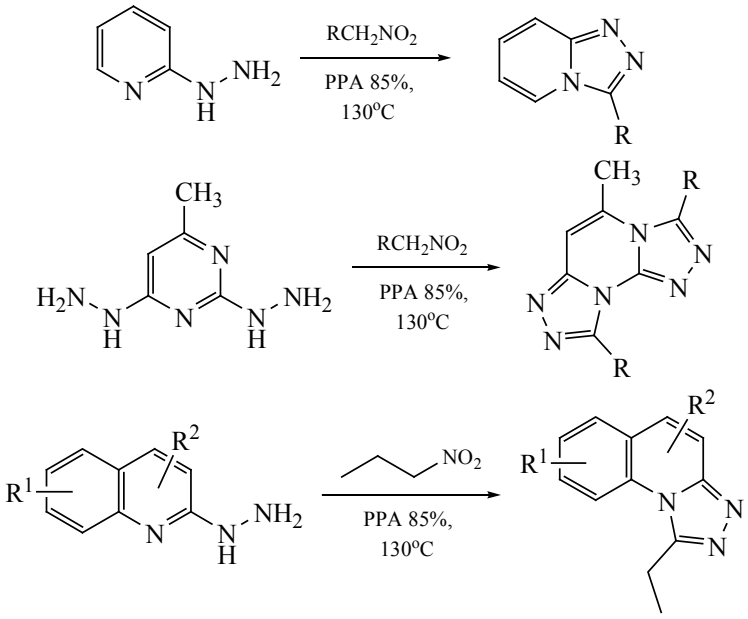
22. DEPARTMENT OF ORGANIC CHEMISTRY OF VOLGOGRAD STATE TECHNICAL UNIVERSITY

Currently, Volgograd State Technical University is developing a fundamental scientific direction at

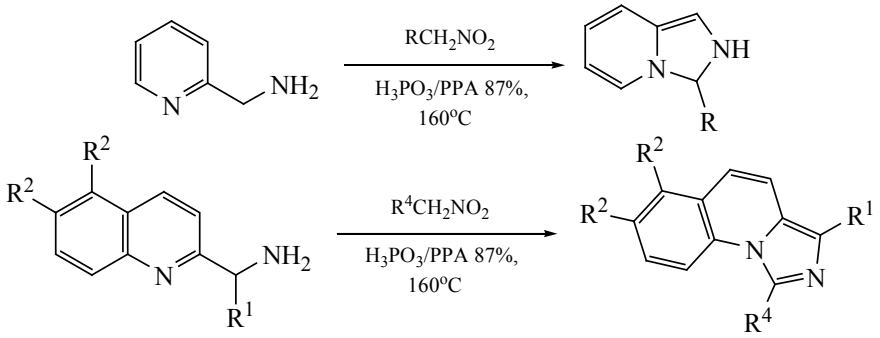
Scheme 21.39.



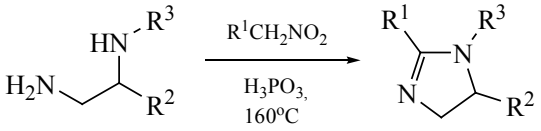
Scheme 21.40.



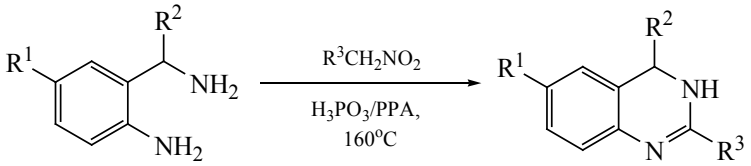
Scheme 21.41.



Scheme 21.42.



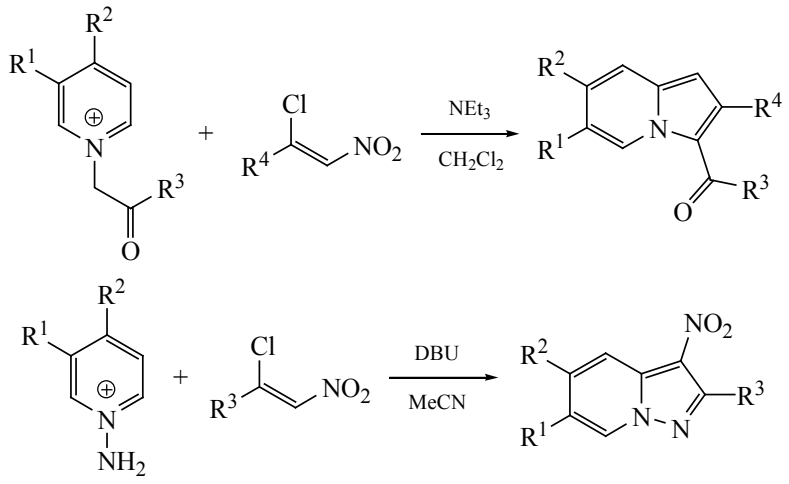
Scheme 21.43.



the intersection of organic, bioorganic, and medical chemistry, which can be titled "Molecular Design of Metabolically Stable and Highly Active Enzyme Inhibitors with Adjustable Lipophilicity of the Framework Ligand for Etiotropic and Symptomatic Therapy of Socially Dangerous Diseases." This direction is being developed under the guidance of **Dr. Chem. Sci., Prof. G.M. Butov** and the new head of the department, **Cand. Chem. Sci., Assoc. Prof. V.V. Burmistrov**.

Since 2012, the university has developed new synthetic approaches to adamantyl-containing heteroallenes and studied their properties in reactions with amines for the synthesis of 1,3-disubstituted ureas. This made it possible to obtain previously unknown bioavailable inhibitors of human soluble epoxide hydrolase (sEH), which are promising in the fight against inflammatory processes and pain conditions of neuropathic etiology [1075–1077]. Original and effective methods of synthesis of 1,3-disubstituted ureas and their S- and

Scheme 21.44.



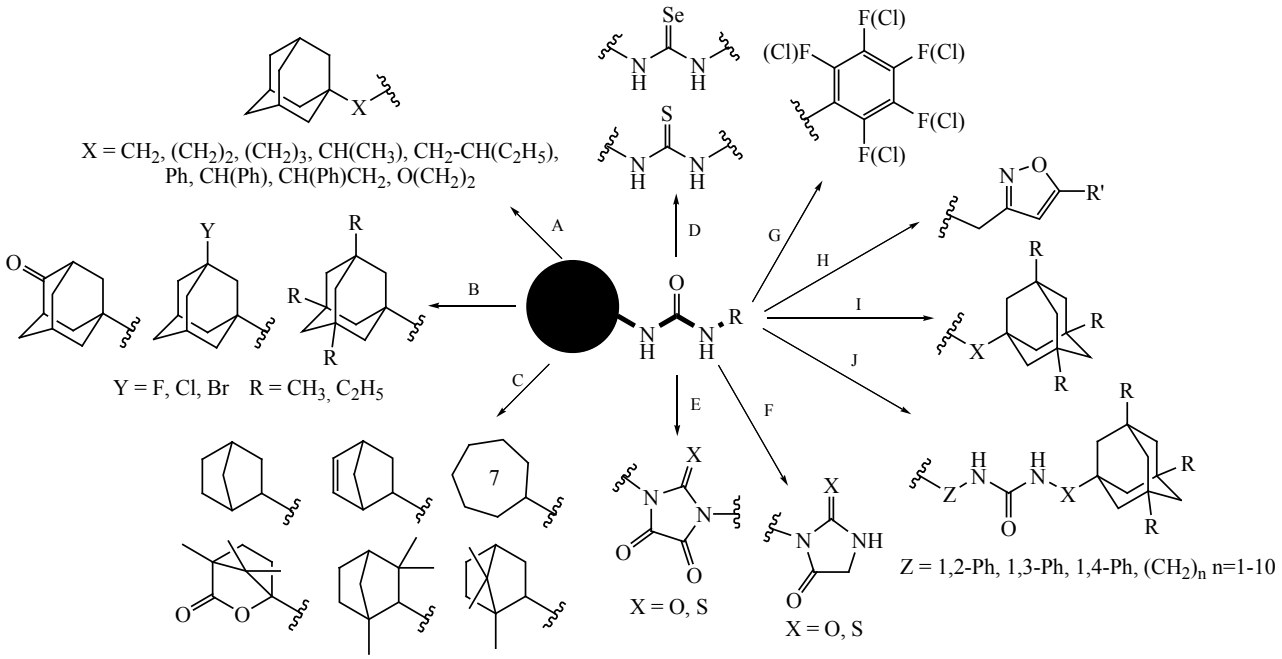


Fig. 22.1. Strategies of synthesis of new sEH inhibitors.

Se-containing derivatives were developed and tested in vitro as potential sEH blockers (Fig. 22.1).

The publications [1078–1082] presented the findings concerning the synthesis of adamantyl isocyanates and isothiocyanates and their derived ureas and thioureas. Recent sEH inhibitory activity studies showed that, due to the structural changes we introduced in ureas, we managed to create inhibitors with a picomolar activity [1083].

The synthesis of ureas was based on reactions of adamantyl isocyanates with amines, which occur with a high atom precision under mild conditions (Scheme 22.1) [1084].

We also proposed and obtained for the first time inhibitors of a new structural type: bis-diadamantyl diureas (Scheme 22.2) [1085, 1086].

Later, we obtained an experimental evidence for the high inhibitory activity of these compounds and found that it depends on the length of the linker Z between the urea groups. Molecular docking demonstrated the possibility of binding of the second urea group to another region of the enzyme (n = 4–6), which had not been previously considered [1085].

Specifically for urea synthesis, we developed methods for the synthesis of previously unknown heteroallenes and amines of the adamantane (or bicyclic) series, which allowed the most important SAR and QSAR dependences for ureas to be established (Fig. 22.2).

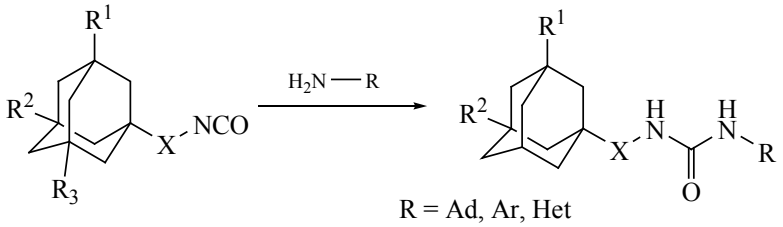
Isocyanates were synthesized by an improved one-pot procedure via the Curtius reaction from carboxylic acid chlorides and sodium azide [1087] or from carboxylic acids (Scheme 22.3) using diphenylphosphoryl azide (DPPA) [1088], as well as via the reaction of amines with *N*,*N*'-carbonyldiimidazole (CDI) [1089].

We found (b. 2017) that heating adamantyl amines with phenyl isothiocyanate given rise to the corresponding adamantyl isothiocyanates (Scheme 22.4) [1090].

It was established that the reaction involves the intermediate formation of an unsymmetrical thiourea. The reaction proceeds in a homogeneous medium, is easily scaled up, and can be extended to any other amines containing donor substituents, for example, bicyclic amines.

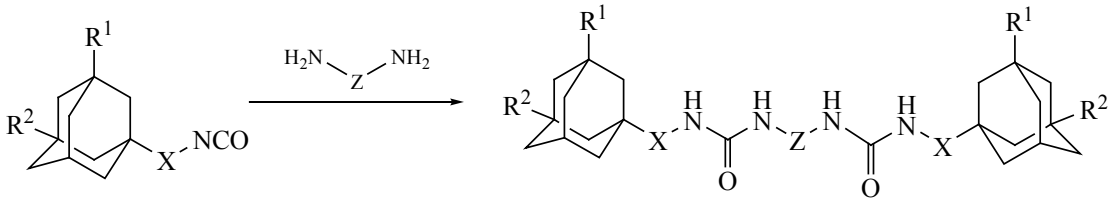
To prepare difficult-to-access adamantyl isocyanates and thioisocyanates, new one-step reactions of alkyl(aryl)iso(isothio)cyanates or esters with 1,3-dehydroadamantane were carried out (Scheme 22.5) [1091]. Methods for the synthesis of some amines of the adamantane series, including difficult-to-access branched amines at the bridgehead position of the adamantyl substituent, were developed [1092]. The

Scheme 22.1.



R = Ad, Ar, Het
R^1, R^2, R^3 = H, CH_3, CH_3CH_2, F, Cl, Br
X = "–", CH_2, CH_2CH_2
150 examples

Scheme 22.2.



R^1, R^2 = H, CH_2, CH_3CH_2, F, Cl, Br
X = "–", CH_2, CH_2CH_2
Z = $(CH_2)_n$, 1,2-Ph, 1,3-Ph, 1,4-Ph, $(CH_2CH_2\text{-}O)_n\text{-}CH_2CH_2$
n = 2–10 n = 2–4
100 examples

ureas obtained from these amines made it possible to establish for the first time the differences between the sEH inhibitory activities of the nodal and bridgehead positions of the adamantyl substituent.

Under the supervision of **Acad. I.A. Novakov** and **Dr. Chem. Sci., Prof. M.B. Nawrozkij**, methods of synthesis of more conformationally flexible amines containing framework fragments (bornyl, norbornyl,

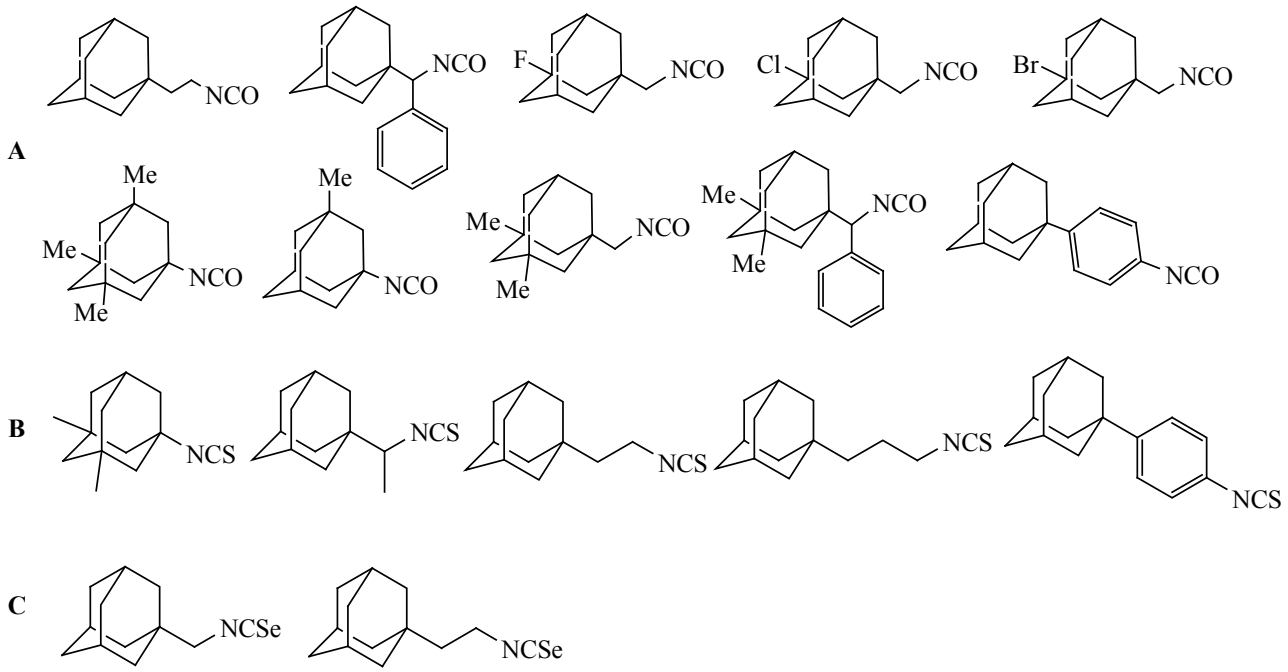
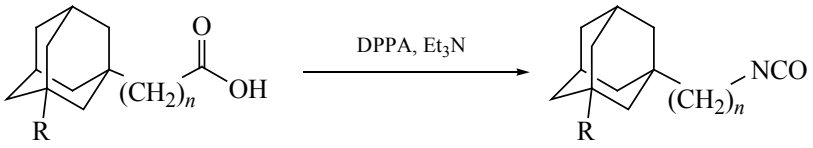


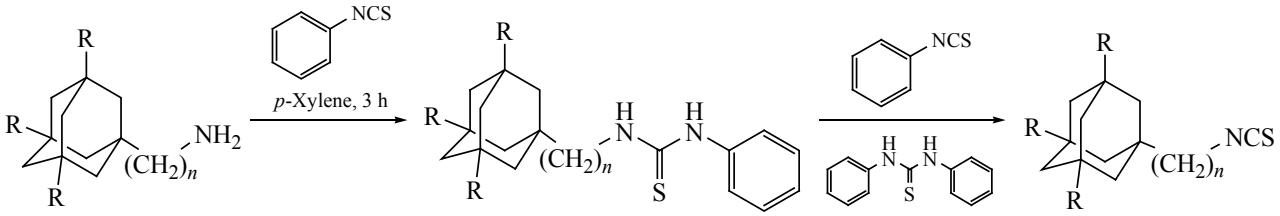
Fig. 22.2. Structures of some synthesized isocyanates (**A**), isothio- (**B**) and isoselenocyanates (**C**).

Scheme 22.3.



n = 1, 2, R = H, CH_3, CH_3CH_2, F, Cl, Br
12 examples

Scheme 22.4.



n = 1–3, R = H, CH_3
10 examples

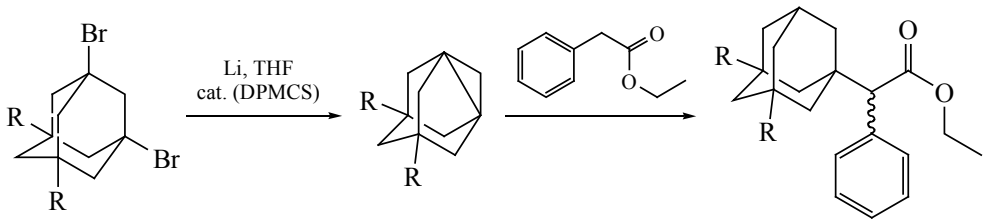
etc.) by the reduction of the corresponding high-purity oximes (≥ 99%) under Schwenk–Papa reaction conditions were developed (Scheme 22.6) [1093–1097], and the products were used to obtain sEH inhibitors. Such structural changes in urea molecules made it possible to obtain the first evidence showing that sEH is enantiomerically specific. Thus, ureas obtained from the L-enantiomer of bornylamine are up to 14 times more active than their D analogs. Therewith, the activity of racemates is mid-range between the L and D enantiomers [1098, 1099].

A few ureas were tested as mouse and rat sEH inhibitors. Differences in binding to human (hsEH), mouse (msEH), and rat (rsEH) soluble epoxide hydrolases were demonstrated for the first time. It was found that the steric load of the lipophilic substituent stronger affects binding to msEH than to other sEHs, which suggests that the results obtained for rodents should be treated with greater caution when assessing the impact on humans [1080].

As a result of our research, new original methods for the synthesis of isocyanates, isothiocyanates, and isoselelonocyanates containing framework fragments were discovered and studied. The sythesized hetero-allenes were used to develop synthetic approached and prepare 1,3-disubstituted ureas, thioureas, and their cyclic analogs: imidazolidine-2,4,5-diones, pyrimidine-2,4,6-triones, and hydantoins. The products showed inhibitory activity against human soluble epoxide hydrolase at nanomolar concentrations.

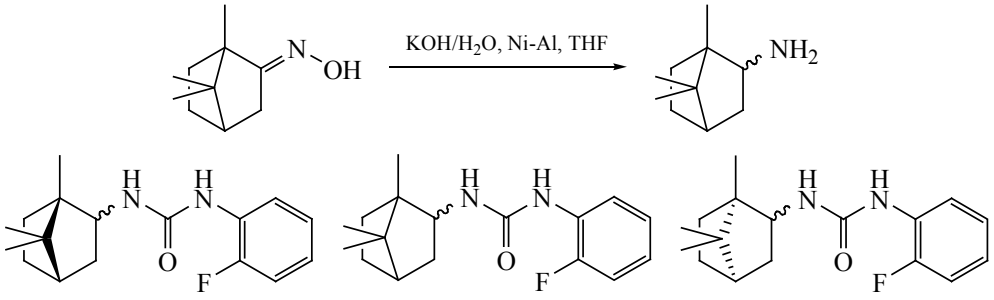
This work was financially supported by the Russian Science Foundation (projects nos. 16-13-00100, 19-73-10002, 21-73-20123 and 22-13-20062), the Russian Foundation for Basic Research (project nos. 20-03-00298-A, 18-43-343002-r_mol_a, 16-43-340116-r_a, 16-33-00172-mol_a, and 12-03-33044-mol_a_ved), and the Ministry of Education and Science of the Russian Federation within the framework of the basic part of the state assignment for 2017–2019 (project no. 4.7491.2017/BCH).

Scheme 22.5.



R = H, CH_3

Scheme 22.6.



IC_{50} = 3.7 nM IC_{50} = 27.0 nM IC_{50} = 50.6 nM

23. KEY ACHIEVEMENTS AND MAIN BASIC AND APPLIED SCIENTIFIC RESULTS OF THE DEPARTMENT OF ORGANIC CHEMISTRY OF VORONEZH STATE UNIVERSITY, FROM 2018 TO 2023

To the 100th anniversary of the Department of Organic Chemistry

The main direction of basic research at the Department of Organic Chemistry of Voronezh State University, headed by **Dr. Chem. Sci., Prof. Kh.S. Shikhaliev**, is the development of highly selective methods of synthesis and study of functional derivatives of mono- and polynuclear N,O,S-containing heterocyclic compounds with a wide range of practically useful properties, including diverse biological activity, inhibitory effect on corrosion of nonferrous and ferrous metals, as well as luminescent and surface-active properties. Within the framework of this direction, new types of cascade reactions have been developed, for example, a one-pot combination of 2-4 processes, including multicomponent variants; new recyclization processes of cyclic derivatives of carboxylic acids have been proposed; and a series of rearrangements have been explored.

Cyclic imides of unsaturated dicarboxylic acids are versatile substrates for the synthesis of various heterocyclic compounds, including potential new pharmaceuticals. Research in this area is being conducted by **Cand. Chem. Sci., Assoc. Prof. Yu.A. Kovygin**, **Cand. Chem. Sci., Assoc. Prof. A.V. Zorina**, **Cand. Chem. Sci., Assoc. Prof. N.V. Stolpovskaya, and Assoc. Prof. A.L. Sabynin**. The reactions of thioacetamide with *N*-arylmaleimides were studied under different conditions (Scheme 23.1).

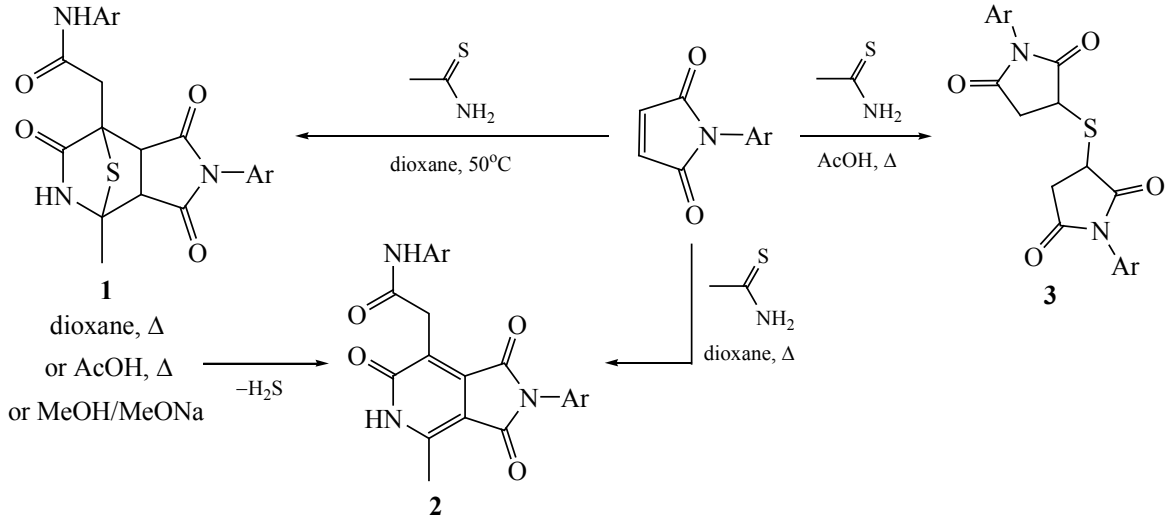
It was established that heating the starting reagents under reflux in acetic acid provides 3,3'-thiobis(1-arylpyrrolidine-2,5-diones) **3** as major products. The reaction of thioacetamide and *N*-arylmaleimide in dioxane at 50°C gives epithiopyrrolo[3,4-*c*]pyridines **1**, which release hydrogen sulfide under heating to form pyrrolo[3,4-*c*]pyridines **2** [1100].

A series of tandem reactions of itaconimides (synthetic analogs of maleimides) with various 1,3-N,N- and 1,3-C,N-binucleophiles (Scheme 23.2), leading, respectively, hydropyrimidines **4** and **5**, and hydropyridines **6**, **7** were studied [1101, 1002].

The traditional direction of research related to the functionalization of 2,2,4-trimethyl-1,2-dihydroquinolines **8** and 2,2,4-trimethyl-1,2,3,4-tetrahydroquinolines **9** is being developed by **Cand. Chem. Sci., Assoc. Prof. S.M. Medvedeva** and **Dr. Chem. Sci., Assoc. Prof. A.Yu. Potapov**, and also formed the basis of dissertation for the degree of Candidate of Chemical Sciences by Lecturer **N.P. Novichikhina**. Among functionally substituted 2,2,4-trimethylhydroquinolines, derivatives with neuroprotective, antioxidant, hepatoprotective, and growth-regulating activities were found. Particularly significant advances are associated with the synthesis and transformations of tricyclic systems derived from 2,2,4-trimethylhydroquinolines **10**–**24** (Scheme 23.3), which are described in the monograph published in 2023 [1103].

Among the newly synthesized substituted 2,3-dithiolo[5,4-*c*]quinoline-1-thiones **10** and (1,2-dithiolo[3,4-*c*]quinolin-1-ylidene)arylamines **11**, protein kinase inhibitors, as well as antibacterial, antifungal, anti-inflammatory, and antiviral (wild-type SARS-CoV-2) agents were found [1105–1105]. Various derivatives of pyrano[3,2-*g*]quinolin-2-ones **12** and **13** were synthesized and comprehensively studied for the first time. The most extensive studies have been carried

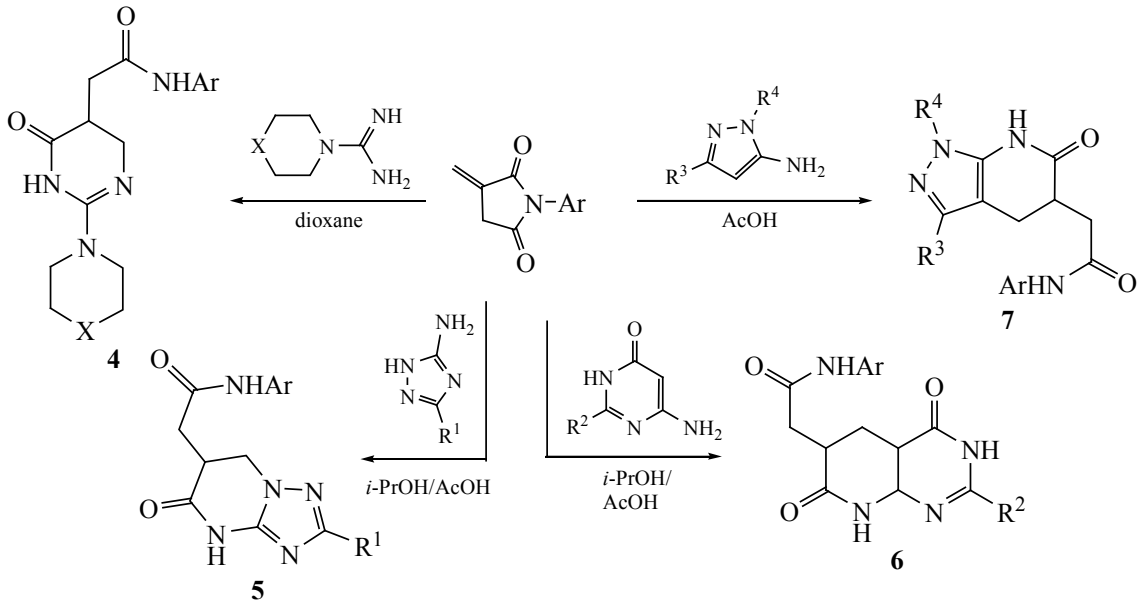
Scheme 23.1.



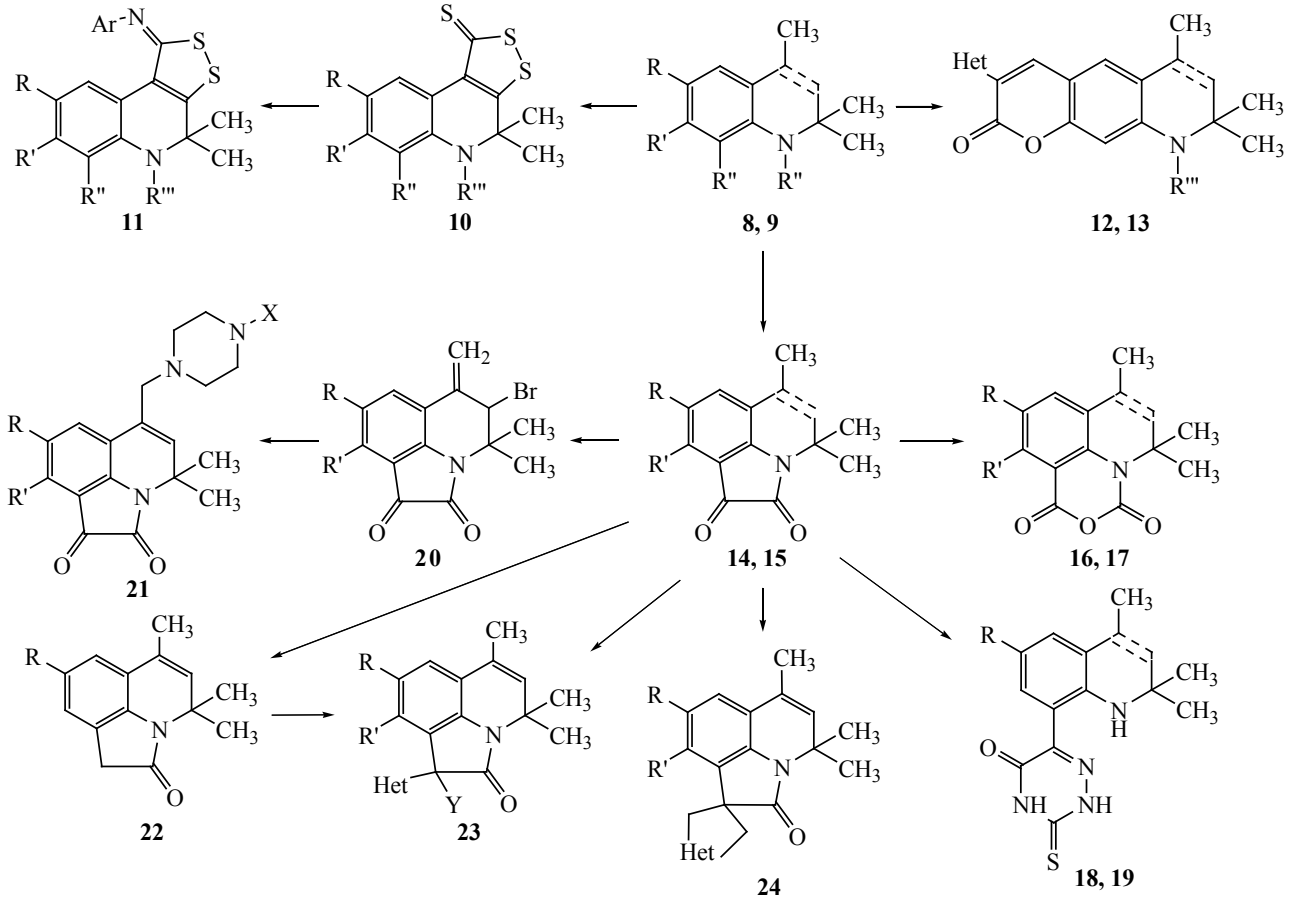
out on transformations of pyrrolo[3,2,1-*ij*]quinoline-1,2-diones **14** and their hydrogenated analogs **15**. Oxidative recyclization and decyclization reactions of these compounds, leading to 1,3-oxazino[5,4,3-*ij*]-quinoline-1,3-diones **16** and **17** and (hydroquinolin-8-yl)-3,4-dihydro-1,2,4-triazines **18** and **19**, respectively, have been performed. In addition, the products have been introduced into reactions with the retention of the pyrrole. Among compounds **20–24**, effective blood coagulation factor Xa, XIa, and XIIa inhibitors have been found [1103, 1106].

The research of **Cand. Chem. Sci., Assoc. Prof. D.Yu. Vandysheva**, **Dr. Chem. Sci., Assoc. Prof. A.Yu. Potapova**, **Dr. Chem. Sci., Assoc. Prof. M.Yu. Krysina**, **Cand. Chem. Sci.**, **Researcher A.A. Kruzhilina**, and **Cand. Chem. Sci., Assoc. Prof. N.V. Thus** is focused on such a relevant subject as heterocyclization reactions involving polynucleophilic reagents: biguanidines, amidinothioureas, 3-amino-1,2,4-triazoles, 1,2-diaminoimidazoles, 2-amino-1,3,5-triazines, etc. For example, a general diastereoselective method for the synthesis of pyrimido[1,2-*a*][1,3,5]-triazine derivatives **25** (Scheme 23.4) via the Biginelli

Scheme 23.2.



Scheme 23.3.



reaction of amidinothiourea, arylaldehydes, and acetoacetic ester was developed [1107].

It was shown that 1,2-diamino-4-phenylimidazole acts as a C,N-nucleophile in the multicomponent processes leading to the formation of an imidazo[1,5-*b*]-pyridazine core (Scheme 23.5). Tricyclic systems **29** exhibit luminescent properties and hold promise for the design of biological probes on their basis [1108].

Over the past years, the department's staff has developed and studied a series of radically new inhibitors of corrosion of nonferrous and ferrous metals in different media. The basic heterocyclic core of such inhibitors is 1,2,4-triazole (Scheme 23.6). The introduction of various functional groups into this molecule, including hydrophobic long-chain alkyl groups, makes it possible to obtain inhibitors with predetermined properties. An innovative approach to effective corrosion inhibitors, which involves the use of plant materials and the synthesis of twin molecules, has been developed [1109–1111].

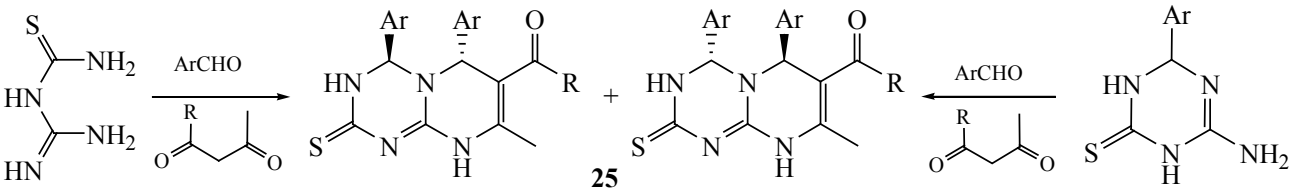
A number of processes for modifying epoxidized derivatives of fatty acids of sunflower and soybean oils have been studied, and products with potential applications as emulsifiers, plasticizers, lubricant components, bioepoxy resins, etc., have been obtained [1112, 1113].

Currently, developments in the field of the targeted synthesis of organic additives used in chemical and electrochemical metal deposition for microelectronics are undeway [1114].

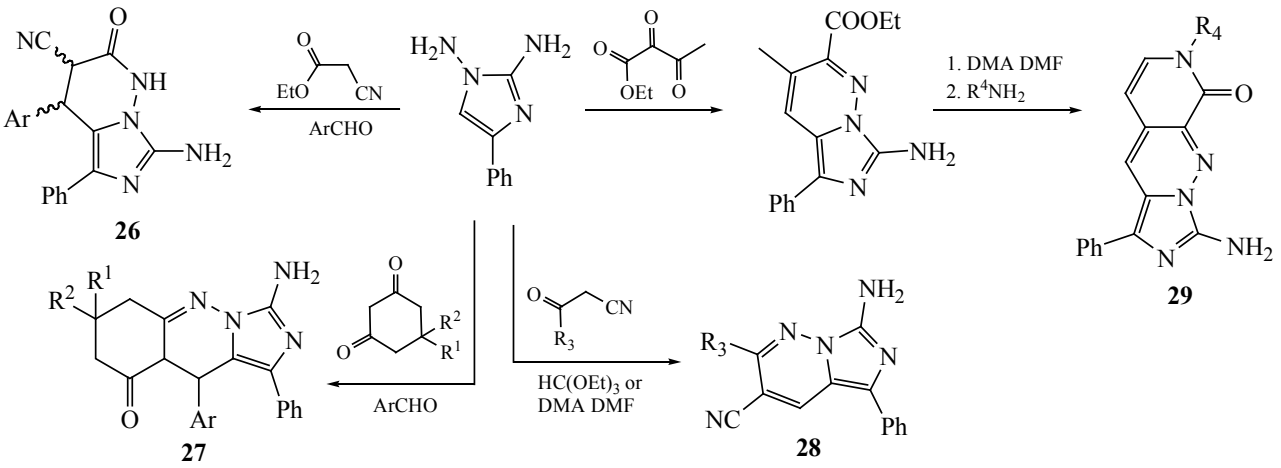
Thus, the Department of Organic Chemistry of Voronezh State University carries out research in different areas both with the aim to find biologically active compounds and promising candidates for diverse industrial applications, from oil production to microelectronics.

The work was financially supported by the Ministry of Science and Higher Education of the Russian Federation within the framework of the state assignment to universities in the field of scientific activity for 2022–2024 (project no. FZGU-2022-0003).

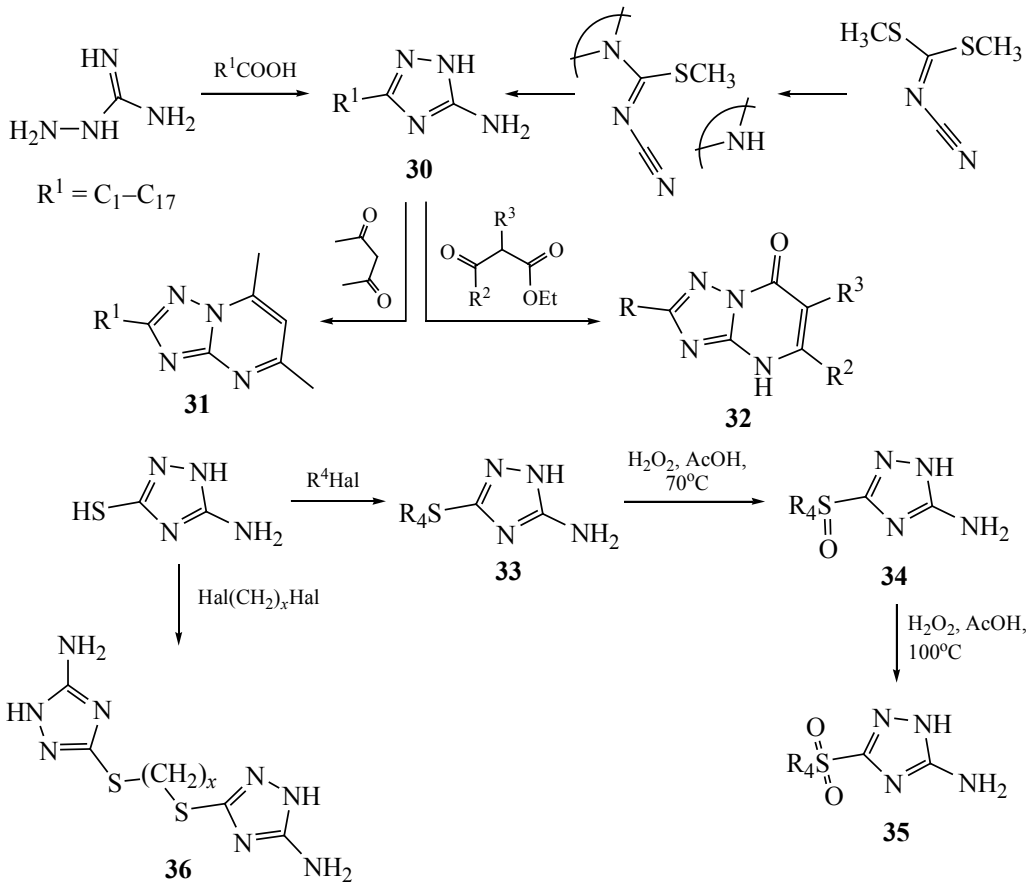
Scheme 23.4.



Scheme 23.5.



Scheme 23.6.



24. RESEARCH INSTITUTE OF ANTIOXIDANT CHEMISTRY OF NOVOSIBIRSK STATE PEDAGOGICAL UNIVERSITY: 20 YEARS OF HISTORY

In 2023, the Research Institute of Antioxidant Chemistry (RIAC) established at Novosibirsk State Pedagogical University (NSPU) on the initiative and under the guidance of Dr. Chem. Sci., Prof. A.E. Prosenko celebrated its 20th anniversary. The decision to establish a research institute at the pedagogical university rested on the long-term and fruitful work of the employees of the Chemical Department of NSPU on the molecular design and development of industrially acceptable methods for the synthesis of thermal stabilizers for polymeric materials [1115]. The priority area of activity of RIAC was applied research aimed at developing the chemistry of phenolic compounds and creating innovative antioxidants for practical use in various industries, biology, and medicine.

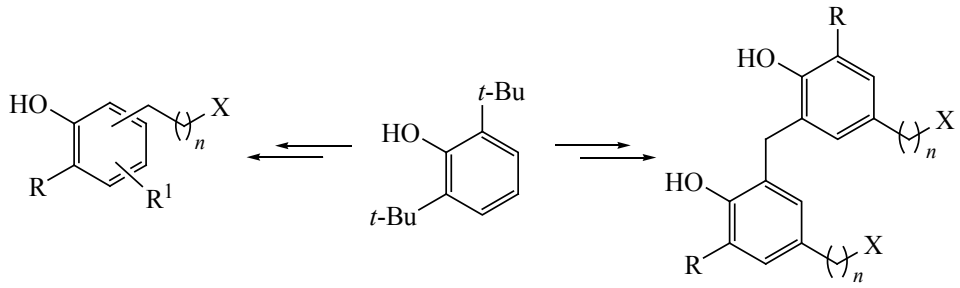
A distinctive feature of the antioxidants created at RIAC is the bifunctional mechanism of their antioxidant action, based on the synergistic combination of the antiradical activity of phenolic fragments with the antiperoxide activity of functional sulfur-, selenium-, tellurium-, phosphorus-, or nitrogen-containing groups. The antioxidant activity of such inhibitors, unlike the activity of classical alkylphenols, is difficult to model mathematically, and this required a large body of empirical evidence concerning the antioxidant properties of such closely structurally related compounds to be accumulated, and, accordingly, effective approaches to their synthesis should be developed. Under the supervision of Prof. A.E. Prosenko, effective ways of transforming industrially available phenols, primarily 2,6-di-*tert*-butylphenol, into halogenated alkylphenols of different structures and further into S,N,P-containing phenolic antioxidants were developed (Scheme 24.1), and the inhibitory activity of the synthesized compounds toward polymers, mineral oils, and lipid substrates was systematically studied [1116].

Later, the proposed methods showed good reproducibility in the synthesis of derivatives of polyatomic [1117] and isobornyl-substituted phenols [1118], imidazole oxides [1119], as well as in the synthesis [1120] and modification [1121] of natural physiologically active substances.

Many antioxidants synthesized at RIAC are characterized not only by high efficiency, but also by the versatility of their antioxidant action. Thus, bis[3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propyl] sulfide was designed as a polymer stabilizer and, as such, was highly appreciated by specialists [1115, 1116], but it also demonstrated high bioantioxidant activity in vivo, as well as pronounced protective properties with respect to free-radical pathologies, including cardiovascular and oncological diseases [1116], and is considered to hold promise for drug design [1122].

Another leading area of RIAC activity is the development of pharmacologically active S,Se-containing phenolic antioxidants with hydrophilic properties, which ensures their high bioavailability and suitability for injection and infusion medications, including in cases of acute emergency. This determines the particular attractiveness of such compounds for medical and biological research. Sulfur- and selenium-containing hydrophilic phenols are successfully used in studies on redox-sensitive processes in healthy and tumor cells and on the effect of the Keap1/Nrf2/ARE

Scheme 24.1.



R, R^1 = H, Me, *i*-Pr, *t*-Bu, Cy, OMe, Br; *n* = 0–5; X = Cl, Br, I; NH_2, SH, SAlk, SSAlk, SAr, NHAlk, $NAlk_2$; NH_3Cl, NH_2AlkCl, $NHAlk_2Cl$, $OP(OAlk)_2$, $SP(SAlk)_2$

signaling system on cellular metabolism and free-radical pathologies [1123]. The methods of synthesis of hydrophilic chalcogen-containing antioxidants of different structures and their properties were reviewed in [1124].

Among the water-soluble antioxidants synthesized at RIAC, the most well-known is sodium *S*-[3-(3-*tert*-butyl-4-hydroxyphenyl)propyl]sulfothioate (antioxidant TC-13), whose biological effects are associated with its ability to effectively activate the Keap1/Nrf2/ARE (antioxidant response element) transcription pathway. The anticancer activity of TC-13 [1125] revealed in MCF-7 cell cultures was confirmed in vivo: TC-13 not only inhibited the growth of transplantable Lewis lung carcinoma in mice, but also enhanced the therapeutic effect of doxorubicin [1126]. At the dose (30–60 mg/kg), TC-13 by itself did not inhibit the development of P-388 lymphocytic leukemia in mice, but significantly enhanced the effect of cisplatin: the average life expectancy index of experimental animals with their combined use increased to 397% vs 109% with cisplatin monotherapy [1127]. In laboratory experiments, TC-13 also neutralized the side effects of cytostatic agents, in particular, it reduced the cardiotoxicity of doxorubicin [1128]. TC-13 also exhibited neuroprotective activity in a model of Parkinson's disease in mice [1129] and increased the survival rate of laboratory animals with tuberculous granulomatosis [1130].

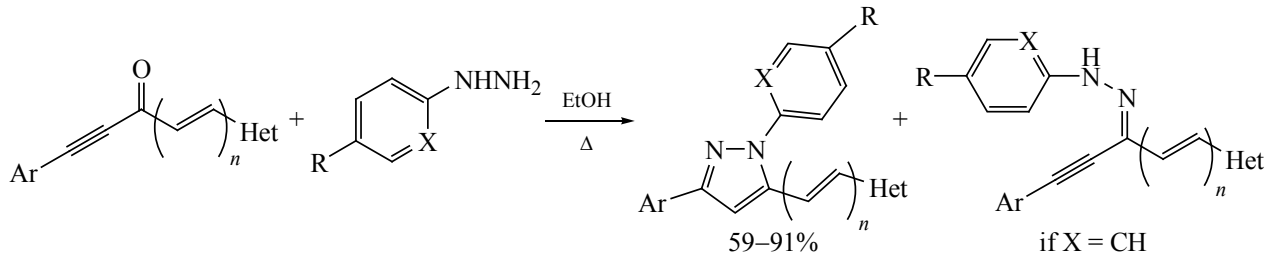
The replacement of the divalent sulfur atom in TC-13 by selenium resulted in a decrease in biological activity [1131], while in the case of compounds containing carboxyl as a hydrophilic group, on the contrary, the selenium-containing derivative was more effective [1132]. The ambiguous effect of selenium on the activity of phenolic inhibitors may be associated both with the different activity of S, Se-containing groups of different structures and with the degree of expression of the synergistic component. Using 2-dodecylsulfanyl(selanyl)methyl-substituted 5-hydroxy-2,3-dihydrobenzofurans and the corresponding binary compositions as an example, we found that selenium-containing phenols can much surpass their sulfur-containing analogs in efficiency of lipid oxidation just due to high synergistic effects [1133, 1134]. These findings explain the significant interest of the RIAC team in the development of the chemistry of selenium-containing phenolic antioxidants in their future activities.

25. ORGANIC CHEMISTRY AT TOGLIATTI STATE UNIVERSITY: CONJUGATED ENYNONES AND THEIR ANALOGUES IN THE SYNTHESIS OF CARBO- AND HETEROCYCLIC COMPOUNDS

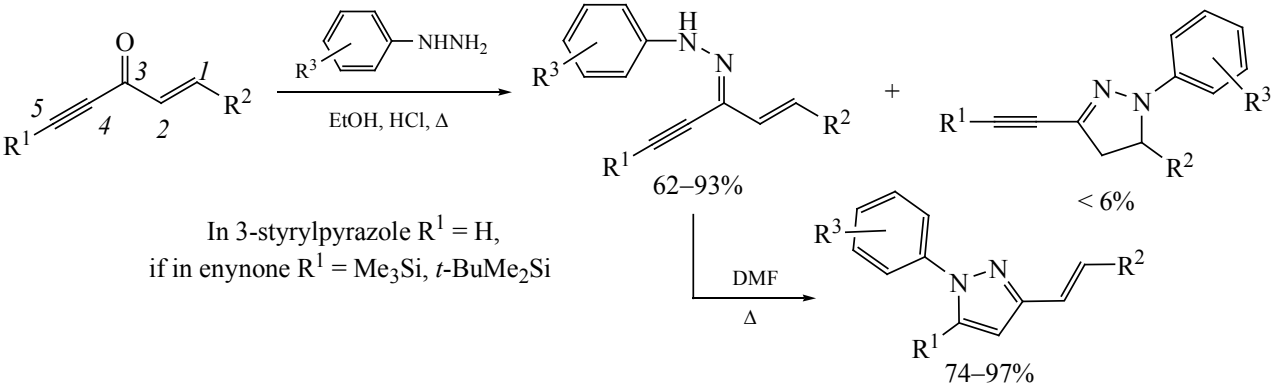
Research in the field of acetylene chemistry has been conducted at Togliatti State University (TSU) since the 1960s and is associated with the name of Prof. Sergei Petrovich Korshunov (1934–2003). Professor Korshunov, being a student of I.A. D'yakonov (Leningrad State University) [1135], completed his postgraduate studies under the supervision of L.I. Vereshchagin in Angarsk. After defending his candidate dissertation, he worked in Togliatti since 1968. Until the early 1990s, under the supervision of S.P. Korshunov and in scientific collaboration with I.V. Bodrikov (Nizhny Novgorod), V.D. Orlov (Kharkov), Ya.P. Stradins (Riga), and other organic chemists, research into the kinetics, stereochemistry, and mechanism of Ad_N reactions of unsaturated compounds was performed [1136–1138]. Since 2010, the scientific interests of the staff of the Korshunov Research Laboratory-13 of TSU have been focused on the chemistry of conjugated enynones and their structural analogs: multifunctional compounds with reaction centers of various chemical natures [1139–1142]. The latter factor allows us to consider conjugated enynones as starting materials for the selective synthesis of carbo- and heterocyclic compounds that are difficult to access by alternative methods. Thus, the introduction of a π-excess heterocycle into position 1 of cross-conjugated enynones reduces the relative activity of the C=C bond and directs cyclocondensation with monosubstituted hydrazines to the triple bond (Scheme 25.1).

The synthesis of 5-(het)arylethenylpyrazoles under mild conditions (in ethanol under reflux) is accompanied by the formation of arylhydrazone impurities in cases, where arylhydrazines (X = CH) are used as reagents. Based on this reaction, a method for the preparation of 1-(pyridin-2-yl)-5-styrylpyrazoles, luminescent probes (X = N) suitable for the detection and quantitative analysis of Hg^{2+}, Pb^{2+}, and Cd^{2+} ions, was developed. Due to the protonation of the keto group and the resulting activation of the C^3 atom of the substrate, increasing the acidity of the reaction medium makes it possible, to change the direction of the reaction with hydrazine toward the formation of the corresponding arylhydrazones, which undergo cyclization in boiling DMF to form 3-(het)arylethenylpyrazoles [1143, 1144].

Scheme 25.1.



Ar = Ph, 4-MeC_6H_4, 5-bromofuran-2-yl; Het = furan-2-yl, 5-methylfuran-2-yl, 5-bromofuran-2-yl, 5-iodofuran-2-yl, 5-nitrofuran-2-yl, benzofuran-2-yl, pyrrol-2-yl, thiophen-2-yl, thiophen-3-yl; X = N, CH; *n* = 1–3



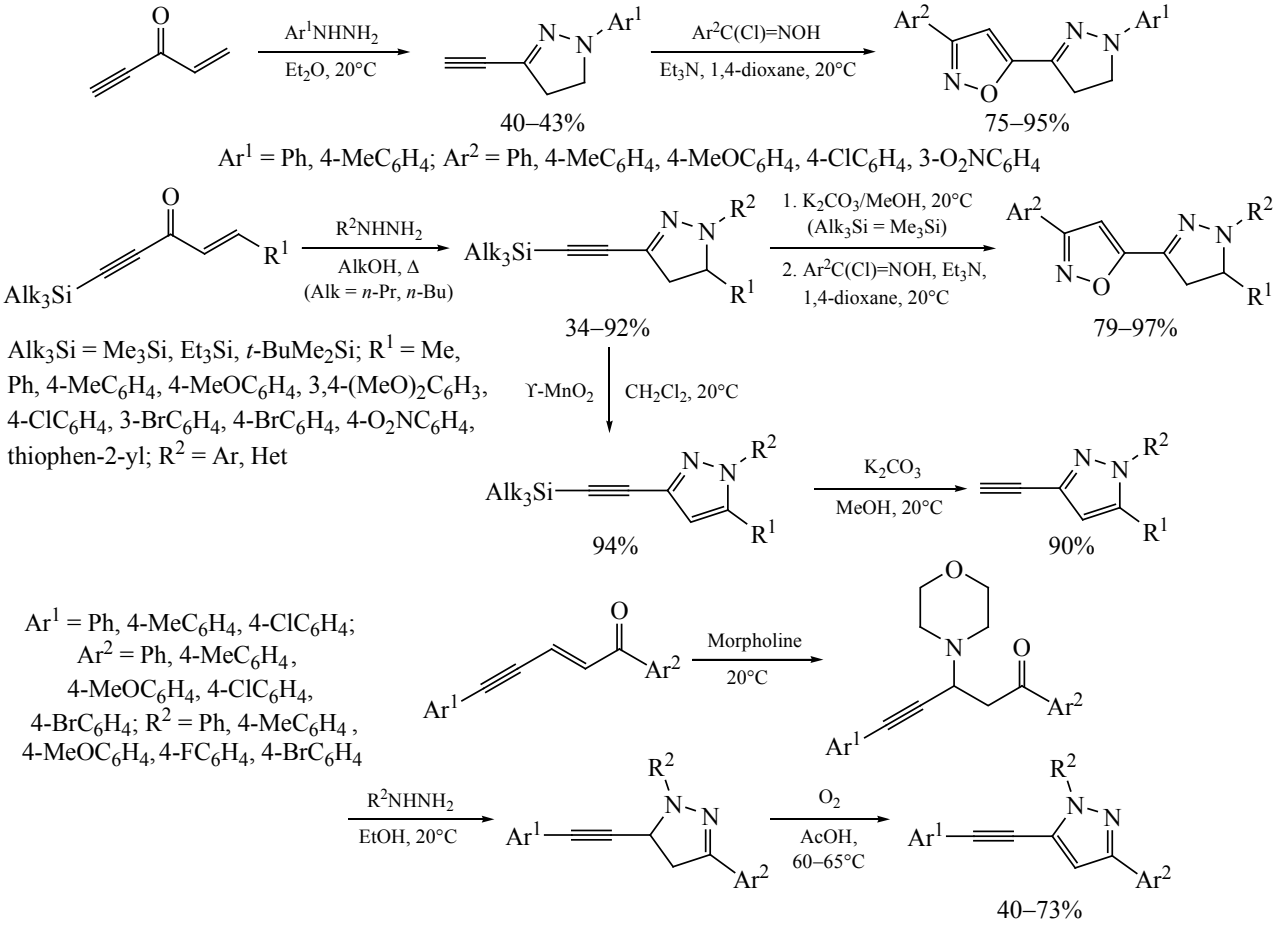
Conjugated enynones can be used to synthesize 4,5-dihydro-1*H*-pyrazoles. The simplest representative of the series, pent-1-en-4-yn-3-one, reacts with arylhydrazines at the propenone fragment to give 3-ethynyl-3-yl-4,5-dihydro-1*H*-pyrazoles in moderate yields. In a more general case, it is possible to redirect cyclocondensation toward the synthesis of 4,5-dihydro-1*H*-pyrazoles by introducing a bulky trialkylsilyl group at the triple bond. Unlike cross-conjugated ones, linearly conjugated enynones scarcely react with arylhydrazines, but the ketone derivatives of propargylamines, obtained on their basis, are synthetic equivalents of pent-2-ene-4-yn-1-ones and easily react with arylhydrazines, providing the corresponding 5-arylethynyl-4,5-dihydro-1*H*-pyrazoles in high yields. The developed methods open up new routes to a series of luminescent azole derivatives (Scheme 25.2) [1145–1148].

The main factors that control the directions of reactions of conjugated enynones with hydrazines were established in [1143–1148]. Thus, varying the substituents in the substrates and reagents, changing the acidity of the medium, and changing some other conditions make it possible to obtain luminescent polysubstituted azoles from preparatively available typical starting compounds [1140]. Together with the research group of A.V. Vasil'ev (St. Petersburg State University), the reactions of linearly conjugated enynones with CH acids were studied (Scheme 25.3) [1149–1152]. For example, the three-component reaction of 1,5-diarylpent-2-en-4-yn-1-ones with malononitrile and sodium alkoxides in the corresponding alcohols leads to the *E* and *Z* isomers of arylethenylnicotinonitriles, as well as arylethynylpyridines as by-products [1150]. In THF, in the presence of $(i\text{-Pr})_2NLi$ (LDA), the same enynones react with malononitrile via a stereoselective dimerization route to form polysubstituted cyclohexanes [1151]. The resulting nicotinonitrile derivatives and cyclohexanes have luminescent properties. Benzoylacetonitrile in the presence of bases adds to the C=C bond of enynones, and the resulting 1,5-diketones react with hydrazine to give 5,6-dihydro-4*H*-1,2-diazepines [1152].

Another type of reactions of conjugated enynones and their structural analogs, leading to aza-heterocyclic compounds, is 1,3-dipolar cycloaddition (Scheme 25.4) [1153–1155].

Reactions of pent-2-ene-4-yn-1-ones with potassium azide (the Huisgen reaction) give excellent yields of 1,2,3-triazole analogs of chalcone and their acyl derivatives, which showed antibacterial activity against *Staphylococcus aureus* [1152]. In addition, these chalcones were used to prepare luminescent 1,2,3-

Scheme 25.2.



triazole derivatives of nicotinonitrile [1153]. Linearly conjugated enynes (ketones, dicarboxylic acids, and Meldrum's acid derivatives) react with diazomethane at the double bond to form pyrazolines or cyclopropanes. The structure of the products depends on the number of acceptor groups in the substrate: ketones form 2-pyrazolines, esters of propargylidene malonic acids form 1-pyrazolines, and enyne derivatives of Meldrum's acid form 5,7-dioxaspiro[2.5]octane-4,8-diones [1154].

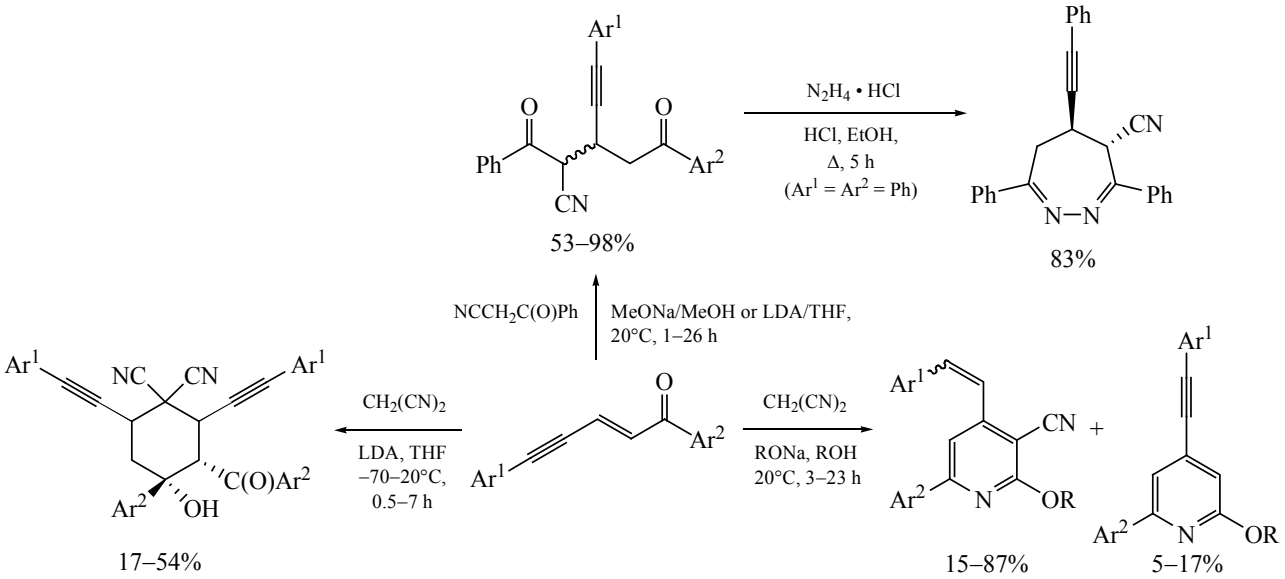
26. POLYMER CHEMISTRY AT BERBEKOV KABARDINO-BALKAR STATE UNIVERSITY: MODERN TRENDS

The Center for Advanced Materials and Additive Technologies (CAMAT) at Kabardino-Balkarian State University (KBSU) dates back to the 1960s–70s, when one of the leading scientists of our country in the field of polymer science, Prof. A.K. Mikitaev (1942–2017), created the industry laboratory named "Heat-Resistant Polymers in Electronic Engineering", the Research Institute of High-Molecular Compounds, and the Mars Design and Development Bureau. In 2008, these structures formed the basis for the establishment of the Polymers and Composites Research and Education Center (REC) of Kabardino-Balkar State University (KBSU). In 2014, the Laboratory of Advanced Polymers was created on the basis of the REC together with the Advanced Research Foundation. In 2017, this laboratory was transformed into CAMAT headed by **Dr. Chem. Sci., Prof. S.Yu. Khashirova**.

The work of CAMAT is aimed at ensuring the technological sovereignty of the country in the field of strategically important polymer materials. To date, 27 highly qualified young specialists in the materials science of polymer materials and in additive technologies have been involved in the research work (including 3 doctors and 20 candidates of chemical and technical sciences in the specialty "High-molecular compounds").

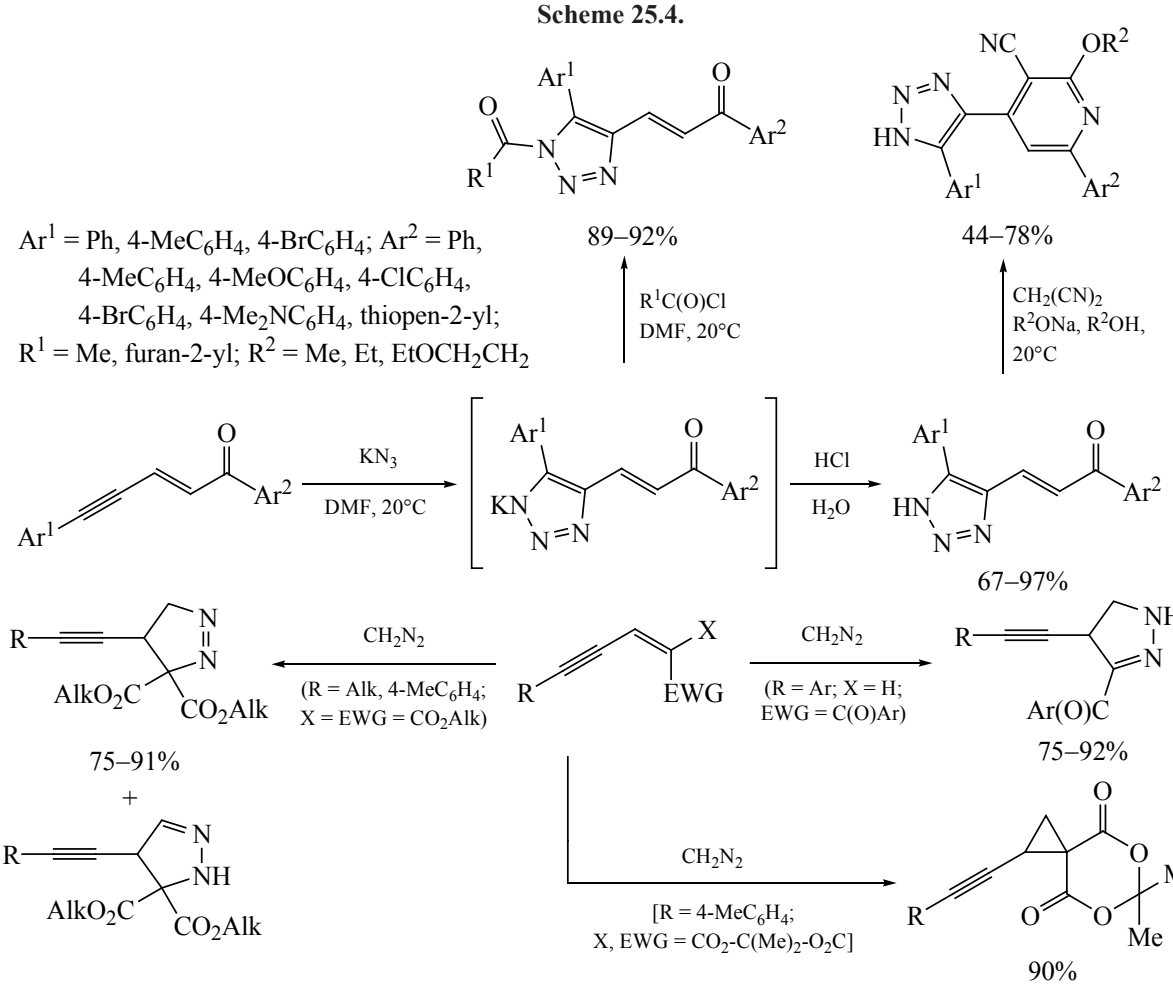
The scientific interests of the center are focused on six main areas: synthesis and study of the properties of new monomers and their oligomers and polymers;

Scheme 25.3.



Ar^1 = Ph, 4-MeC_6H_4, 4-$MeOC_6H_4$, 4-ClC_6H_4; Ar^2 = Ph, 4-MeC_6H_4, 4-$MeOC_6H_4$, 4-ClC_6H_4, 4-BrC_6H_4, 4-$O_2NC_6H_4$; R = Me, Et, *n*-Pr, *n*-Bu

Scheme 25.4.



synthesis of structural and superstructural polymers (polyether ketones, polyether sulfones, polyimides, and polyphenylene sulfide) and their composites; design of binders, plasticizers, dressings, and functional composite materials; development and improvement of 3D printing technologies for polymer materials; additive manufacturing of products from structural and superstructural polymers for the aerospace and defense industries and medicine and the synthesis and study of polymers for medical purposes (polyelectrolytes and biocompatible polymers).

Currently, laboratories specializing in polymer synthesis, special purpose polymers, functional polymers, biomedical polymers, polymer composite materials, structural and thermal studies, polymer physicochemistry, chromatography, polymer rheology, polymer fire resistance, additive technologies and modeling, and additive manufacturing of personalized implants are functioning at CAMAT.

The center carries out a full innovation lifecycle: from polymer synthesis and creation of composite materials to their processing into products by extrusion, casting, 3D printing, milling, pressing, and comprehensive testing of performance. In 2023, a new research area was opened to develop the technology for creating cell-based 3D models by 3D printing, and an equipped 3D bioprinting laboratory was established.

The center operates more than 10 3D printers (Fig. 26.1), based on different printing technologies (FDM, SLS, SLA). For the first time in the Russian Federation, a 3D printer for laser sintering of superstructural polymers was developed, which successfully passed tests for sintering polyether ether ketones (PEEK) together with Kompozit AO.

The center has a pilot production site (Fig. 26.2), which houses semi-industrial equipment for the synthesis and processing of polymers and composites. Small-scale PEEK production is carried out.

The center closely cooperates with research centers of our country. Joint projects are being implemented with Kompozit AO (Roscosmos), the Topchiev Institute of Petrochemical Synthesis of the Russian Academy of Sciences (INPS RAS), the Federal Research Center for Problems of Chemical Physics and Medicinal Chemistry of the Russian Academy of Sciences (FRC PCP MC RAS), network laboratories of polymer composite materials have been created with Peter the Great St. Petersburg Polytechnic University (SPbPU) and Tula State University (TSU).

In particular, together with colleagues from St. Petersburg Polytenic University, PEEK formulations with improved tribological properties were developed [1156].

In collaboration with colleagues from INPS RAS, highly permeable ultrafiltration hollow fiber membranes based on polysulfones of various structures, synthesized at CAMAT, were developed and studied for the first time [1157]. Their nitrogen permeability is 9.5 times higher than the respective value for membranes from commercial PSF Ultrason S 6010 [1158], and their selectivity for the H_2/CH_4 gas pair is 7 times higher compared with membranes from imported PFSF Ultrason. The new domestic polymers were shown to hold promise as membrane materials for hydrogen and helium separation [1159, 1160].

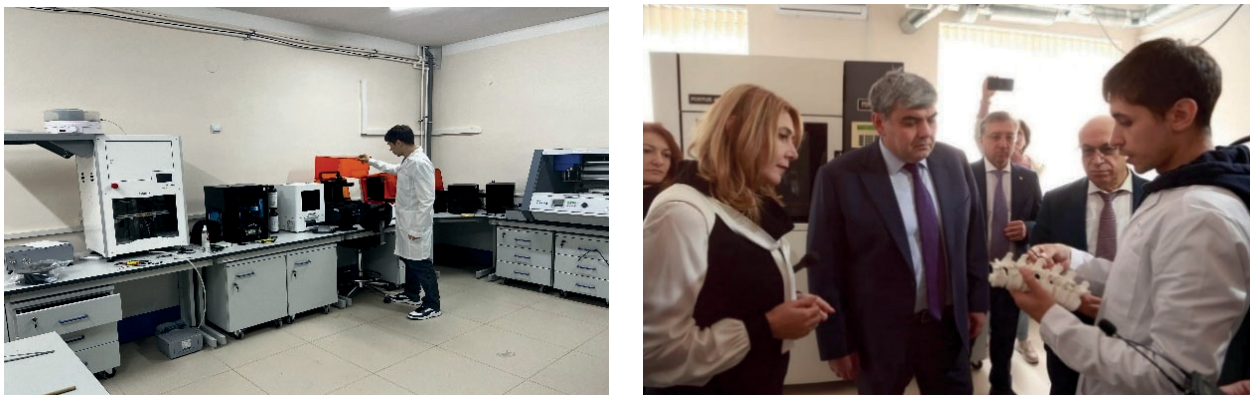


Fig. 26.1. Laboratory of Additive Technologies and Modeling: 3D printers (FDM, SLS, SLA) and a new product made from 3D-printed polymer materials.

Fig. 26.2. Pilot plant of CAMAT.

Together with colleagues from FRC PCP MC RAS, an analysis of the cytotoxicity of 3D products made from PEEK samples synthesized at KBSU and modified by different physical and chemical methods was performed to reveal a lack of cytotoxicity and cell growth. The implants printed from PEEK produced at KBSU successfully passed preclinical tests. For the first time, Technical Specifications for medical PEEK and its biosafety passport were developed.

The CAMAT team has successfully completed more than 10 major research projects and is currently implementing 4 projects on the synthesis of highly effective polymer materials and development of polymer composite materials and polymer additive technologies. Within the framework of the project with the Advanced Research Foundation and 2 projects with Komposit AO (Roscosmos) within the framework of the Federal Target Program of the Ministry of Education and Science of the Russian Federation, a unique scientific and technical reserve has been formed in the field of creating new progressive domestic superstructural polymers and technologies for their 3D printing. Over the past 3 years, a number of new materials for 3D PEEK materials for 3D printing by selective laser sintering (SLS) have been developed with Komposit AO [1161].

New polymer–polymer composites based on the ethylene–vinyl acetate copolymer (EVA) and high-density polyethylene (PE) have been developed for use in 3D printing by the FDM method [1162].

According to the Decree of the Government of the Russian Federation no. 1130-r of May 7, 2022, the center, together with the Titan Group, is an executor of the Petrochemical Cluster Scientific and Technical Program and is developing technologies for the creation of new catalysts and environmentally friendly and highly efficient poly(ethylene terephthalates) (PET) and their composites. Over 1.5 years of project implementation, new complex catalysts based on titanium-containing chelate compounds and layered silicates have been developed [1163, 1164]. The regularities of the synthesis of poly(ethylene terephthalate) and its copolymers in the presence of newly developed titanium catalysts have been established. For the first time, the problem of the yellowish color of products synthesized under titanium catalysis has been solved, and a number of poly(ethylene terephthalates) for film and fiber applications have been synthesized. It has been shown that the new titanium catalysts are five times more efficient than antimony catalysts, which provides ample opportunities for controlling the range of grades of PET materials.

A new method and device for studying the barrier properties of polymer films have been developed, which makes it possible to evaluate the gas permeability of films simultaneously for three gases over a wide time interval (from 1 to 24 h, depending on the quality of the film), and also to increase the accuracy of gas chromatographic analysis [1165].

Over the past 5 years, the employees of the center have published more than 100 papers in highly rated journals, received more than 150 Russian patents and know-how, and have received a number of awards at major international industry exhibitions.

The center is the organizer of the “New Polymer Composite Materials. Mikitaev Readings”, the largest international scientific and practical conference in our country, which annually brings together leading scientists and specialists in polymer chemistry. In 2023, more than 700 people took part in the conference. The Polymer School for Young Scientists has been held for the second year within the framework of the conference.

The future plans of CAMAT include expanding consortiums with scientific and scientific educational organizations to develop, along with established scientific areas, interdisciplinary projects at the intersection of polymer chemistry and physics, biology, microelectronics, agriculture, and information technologies, as well as active involvement in the development of low-tonnage chemistry of advanced polymer materials to strengthen the technological sovereignty of our country.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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