

ADVANCES IN THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS IN THE USSR IN THE PAST 50 YEARS

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The contribution of Russian scientists to the world development of the chemistry of heterocyclic compounds, particularly during the years of Soviet power, has been very substantial. Even in the prerevolutionary period, fundamental investigations on a number of heterocyclic compounds now forming part of the "gold reserves" of the history of this enormous division of organic chemistry were carried out in Russia. Of particularly wide significance has been the work of A. P. El'tekov, K. A. Krasuskii, Z. A. Pogorzhel'skii, N. A. Prilezhaev, A. E. Favorskii (the chemistry of the α -oxides), N. D. Zelinskii and E. O. Grishkevich-Trokhimovskii (sulfur-containing heterocycles), N. N. Lyubavin, A. N. Vyshnegradskii, G. K. Dekker, A. A. E. Chichibabin, A. E. Arbuzov, E. S. Khotinskii, V. V. Chelintseva (nitrogen-containing heterocycles with aromatic properties), G. V. Korshun, V. V. Chelintsev (pyrrole derivatives), P. I. Petrenko-Kritchenko (oxygen- and nitrogen-containing heterocycles) and others.

During the past fifty years, investigations in the field of the chemistry of heterocyclic compounds in the USSR have developed on a broad front. From the traditional chemical centers of our country (Moscow, Leningrad, Khar'kov, Kiev, Kazan) they have spread to all its boundaries (Rostov-on-Don, Riga, Sverdlovsk, Saratov, Erevan, Kuibyshev, Voronezh, Lvov, Donetsk, Novosibirsk, Odessa, Dnepropetrovsk, Tomsk, Chernovtsy, Ufa, Irkutsk, Penza, Vladivostok, etc.). In many of these towns (Moscow, Riga, Kiev, Sverdlovsk, Rostov-on-Don, Saratov, Ufa, Odessa) scientific conferences devoted to various aspects of the chemistry of heterocyclic compounds have been held repeatedly.

In the years of Soviet power, Russian scientists have been responsible for a large number of review papers and monographs devoted both to heterocyclic compounds in general [1-19] and to the chemistry of individual representatives of them (alkene oxides [20-25], glycidic ethers [25a], oxacyclanes [26-28], diketene [29], furanones [30], furan [27, 28, 31-37], 2-pyrone [38], 1,4-dioxane [39], oxazoles [40], oxadiazoles [41], alkene sulfides [42], thiolactones [43], thiacyclanes [44], thiophene [45, 46] and other sulfur-containing heterocycles [46], thiamine [47], selenophene [48], ethyleneimine [49, 50], pyrrole [51], pyrrolizidine [52, 85], aza- and diazacyclanes [53], piperidine [54, 55], piperidine [56], pyridine [57-61], indole [62, 63], quinoline [64-66], acridine [67], azoles [68], pyrazoline [69], pyrazolone [70], imidazole [71], piperazine [72], pyrimidine [73], barbituric acids [74], phenazine [75], quinuclidine [76], 1,3,5-triazine [77, 78], alloxazine [79], pteridine [80], hep-

tazine [81], porphine [82], heterocyclic derivatives of carbohydrates [83] and of steroids [84], alkaloids [2, 7, 8, 85-87], heterocyclic vitamins [47, 61], flavins [61a], nucleotides [88], penicillins [88a], inorganic heterocycles [89-93], the spectroscopy of N-heterocycles [93a], etc.).

The incomplete list given above clearly illustrates the wide range of interests of scientists specializing in the field of the chemistry of heterocyclic compounds and the branches of the science in which they have carried out their investigations.

The enormous scientific advances of Soviet investigators specializing in the field of heterocyclic chemistry is also shown by the appearance in 1965 of the new All-Union journal "Khimiya Geterotsiklicheskikh Soedinenii" [Chemistry of Heterocyclic Compounds], which is published by the Academy of Sciences of the Latvian SSR in Riga [English translation by The Faraday Press Inc., New York].

The industrial production of heterocyclic compounds has served as the basis for the creation of a domestic chemical industry, the production of dyes and pharmaceutical preparations. At the present time, many heterocyclic compounds (ethylene oxide, furfural, caprolactam, pyridine, etc.) are multitonnage products of the heavy organic synthesis industry.

It is not possible to list even the names of all the scientists responsible for the enormous rise in the chemistry of heterocyclic compounds in the USSR in the last 50 years, much less, to discuss the results they have obtained, even cursorily. Consequently, in the present short review we shall limit ourselves to a brief account consisting only of the work of Soviet scientists carried out in the years of Soviet power in the field of heterocycles possessing aromatic properties and their hydro derivatives.

Below we shall consider general methods for the synthesis and mutual reactions of oxygen-, sulfur-, selenium-, and nitrogen-containing heterocycles possessing aromatic properties, and also the main directions of investigations in the field of heterocycles containing two and more heteroatoms.

In view of this range of questions considered, a large number of extremely interesting and important investigations devoted to heterocycles mainly of a nonaromatic nature has remained outside the framework of the present paper. Among them are the investigations of V. M. Rodionov [8] (synthesis of heterocyclic compounds based on β -amino acids), I. L. Knunyants [19, 43, 94, 95] (lactams, aza- and thialactones, oxazolones), A. I. Kiprianov [96] and I. I. Levkoev [97] (cyanine dyes), N. S. Vul'fson and M. M. Shemyakin [98] (β -lactones), M. M. Shemyakin

and V. K. Antonov [99] (oxacyclanes obtained by the spontaneous isomerization of hydroxy acyl amides), V. G. Yashunskii [100] (sydones and dydnone imines), Yu. A. Arbuzov [101] (production of heterocyclic systems by the diene synthesis and use of nitroso compounds as dienophiles), M. G. Voronkov [102, 103] (chemistry of heteroorganic heterocyclic systems, atranes), M. Yu. Lidaka and S. A. Hiller [104], M. G. Voronkov and L. A. Fedatova [105], G. I. Braz [106], R. G. Kostyanovskii [107], A. Ya. Berlin [108], L. D. Protsenko [109], A. A. Kropacheva [110], N. P. Grechkin [111], N. S. Nametkin and V. N. Perchenko [112] (chemistry of ethyleneimine), P. V. Zimakov [22, 23], M. S. Malinovskii [20, 21], A. A. Petrov [113], G. A. Razuvaev and V. S. Etlis [25], I. L. Knunyants [596], T. I. Temnikova [114], B. A. Arbuzov [113a], P. P. Shorygin and V. I. Isagulyants [114a] (ethylene oxide and its derivatives), G. I. Razuvaev and V. S. Etlis [25], I. L. Knunyants [596], T. I. Temnikova [114], B. A. Arbuzov [113a], P. P. Shorygin and V. I. Isagulyants [114a] (ethylene oxide and its derivatives), G. I. Razuvaev and V. S. Etlis [25] (alkene sulfides), I. N. Nazarov [14]* (six-membered nitrogen-, oxygen-, and sulfur-containing heterocycles), G. V. Chelintsev [116] (synthesis of vitamin B₁), N. A. Preobrazhenskii and M. N. Shchukina [117] (synthesis of pilocarpine), M. V. Rubtsov [53] (azabicyclic compounds), M. A. Prokofiev and Z. A. Shabarova [88, 118] (chemistry of the nucleotides), and many, many others.

Particularly noteworthy are the achievements in the field of natural heterocyclic compounds, the alkaloids. A. P. Orekhov and his pupils [2, 7, 8, 85] have studied the alkaloid composition of about 1000 species of plants of the USSR and have isolated from them and described 65 new alkaloids (the structures of 20 of which have been established). G. P. Men'shikov [85] has discovered a new group of alkaloids containing the previously unknown pyrrolizidine heterocycle.

Extremely important from both the theoretical and the practical aspects is the work of Soviet scientists (A. E. Porai-Koshits, B. A. Porai-Koshits, N. M. Kizhner, A. I. Kiprianov, N. S. Dokunikhin, L. S. Efros, N. S. Vul'fson, B. M. Krasovitskii, G. T. Pil'yugin, etc.) in the field of dyes, materials for cinematography, and luminescent substances of the heterocyclic series.

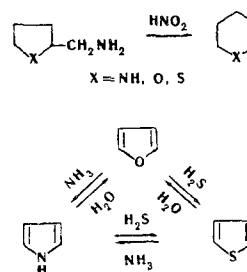
Great interest is also presented by numerous investigations of Soviet chemists in the field of silicon-containing heterocycles (N. S. Nametkin, V. M. Vdovin, V. F. Mironov, E. A. Chernyshev, and M. G. Voronkov) and also organic derivatives of inorganic heterocycles (K. A. Andrianov, L. M. Khananashvili, A. L. Klebanskii, E. G. Kagan, D. Ya. Zhinkin, etc.).

*For a list of 53 papers from the series "Heterocyclic Compounds", see [115].

GENERAL METHODS FOR THE SYNTHESIS OF HETEROCYCLIC COMPOUNDS HAVING AROMATIC PROPERTIES

In 1928–1930, N. I. Putokhin [119] and N. V. Vil'yams [120] showed the possibility of converting aminomethyl derivatives of five-membered heterocycles into the corresponding six-membered heterocyclic compounds by the action of nitrous acid. Thus, the α -aminomethyl derivatives of pyrrole, pyrrolidine, tetrahydrofuran, and thiophene were converted into pyridine, tetrahydropyridine, tetrahydropyran, and thio-pyran, respectively, and β -aminomethylindole was converted into quinoline.

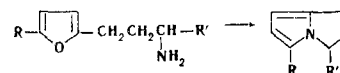
In this way, N. Ya. Dem'yanov's rearrangement was applied for the first time to heterocyclic systems:



In 1935, Yu. K. Yur'ev (for a list of Yur'ev's papers, see [131]) discovered the reaction that now bears his name of the interconversion of heterocycles taking place in the vapor phase over alumina [121–130].

Yur'ev's reaction takes place with the tetrahydro derivatives of aromatic heterocycles even more readily than with the aromatic heterocycles themselves; for example, tetrahydrofuran is converted into thiophan or pyrrolidine and tetrahydropyran into piperidine, thio-tetrahydropyran, or selenotetrahydropyran [122–125].

Yur'ev's reaction enables us to obtain 1-azabicyclic compounds from amines of the furan series containing an amino group in position 3 of a side chain. The synthesis of compounds of the 1,2-dihydropyrrolizidine series is particularly interesting [132]:



The broad possibilities of Yur'ev's reaction in the synthesis of a large number of heterocyclic compounds from 1,2-, 1,4-, and 1,5-diols, mercapto and amino alcohols, and ethylene oxide have been shown on the basis of a large number of examples [5, 125–130].

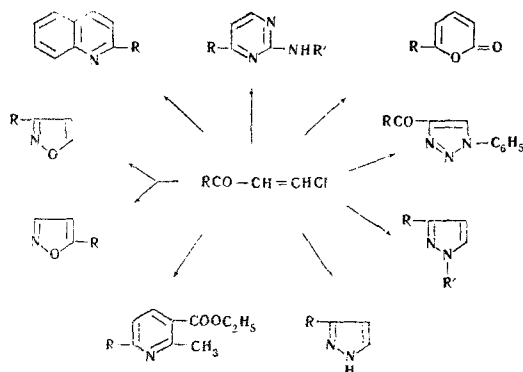
Interesting work on the interconversion of six-membered heterocycles has been carried out in Armenia by S. A. Vartanyan and A. S. Noravyan [133].

Connected with the investigations of Yu. K. Yur'ev, N. I. Putokhin, and N. V. Vil'yams giving the possibility of carrying out the interconversion of heterocyclic systems are the investigations carried out in 1924–1941 by N. D. Zelinskii [134–136], Yu. K. Yur'ev [134, 137], N. I. Shuikin [136, 138], and M. I.

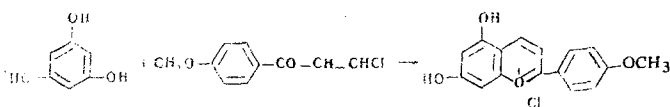
Ushakov [139], and also by N. I. Putokhin [140], on the catalytic hydrogenation and dehydrogenation of five- and six-membered heterocycles, which showed the possibility of the interconversion of aromatic and saturated heterocycles.

A. E. Chichibabin's work [57, 58] on the catalytic synthesis of pyridine and its homologs and analogs from ammonia or amines and acetylene or aldehydes, and of thiophene from hydrogen sulfide and acetylene has become classical.

A general method for the synthesis of various five- and six-membered heterocyclic systems, pyrazole, thiazole, pyridine, quinoline, pyrimidine, isoxazole, and pyrone, from alkyl and aryl β -chlorovinyl ketones (products of the condensation of acid chlorides with acetylene in the presence of aluminum chloride) has been developed by A. N. Nesmeyanov and N. K. Kochetkov:



The reaction of β -chlorovinyl ketones with phenols in the presence of ferric chloride takes place with the formation of benzopyriliium salts [166-169]. For example, starting from phloroglucinol and *p*-methoxyphenyl chlorovinyl ketone the methyl ether of the natural anthocyanidin gesnerin has been synthesized [166]:

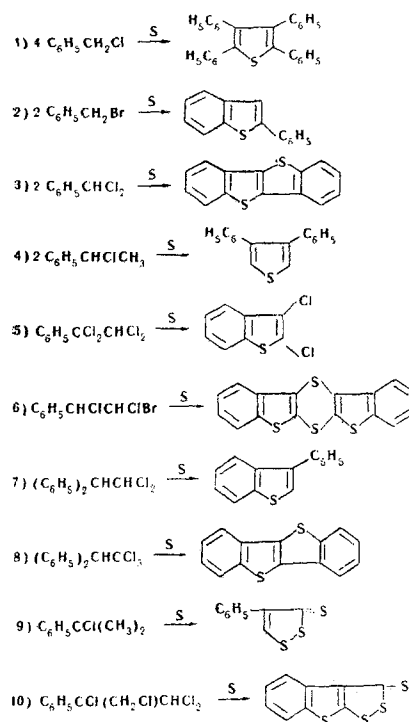


Thus, the highly reactive and readily accessible β -chlorovinyl ketones have become the basis of many new methods for synthesizing heterocyclic compounds.

F. Ya. Perveev [172-183] has developed a general method of synthesizing five-membered heterocycles from acetylenic α -oxides. The reaction of such oxides with hydrogen sulfide, hydrogen selenide, ammonia, or amines, respectively, forms derivatives of thiophene, selenophene, or pyrrole.

An original method for the synthesis of sulfur-containing heterocycles connected or condensed with aromatic nuclei has recently been developed by M. G. Voronkov and his colleagues (V. E. Udre and T. V.

Lapina) [184-186]. It is based on the reaction of elementary sulfur with arylalkyl halides, and can be illustrated by the following examples:



The work of A. P. Terent'ev has made many derivatives of various heterocyclic compounds available. In particular, when using pyridine sulfur trioxide as sulfonating agent he has obtained sulfonic acids of the pyrrole, indole, and furan series and of other acidophobic heterocycles [187].

L. P. Zalukaev's work [188] leading to an original method for the synthesis of nitromethyl derivatives of various heterocycles is also of general importance.

OXYGEN-CONTAINING HETEROCYCLES WITH ONE HETERO ATOM

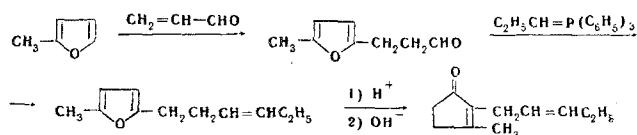
The chemistry of the oxygen-containing heterocyclic compounds and, in particular, the chemistry of furan, is developing extremely vigorously in the Soviet Union. Investigations in the field of the chemistry of furan and its derivatives touch on very diverse aspects of their chemical behavior and practical applications.

Thus, K. Yu. Novitskii and Yu. K. Yur'ev [5, 189-199] have studied in detail the reactivity of various mono- and bis(chloromethyl) furans. On the basis of the reaction of bis(chloromethyl)furans with nucleophilic reagents, general and convenient methods for the synthesis of bifunctional furan derivatives have been developed. Hydroxy- and mercaptoethylamines of the furan series have been used to obtain other heterocyclic systems (piperazines, thiomorpholines, oxazolines, and thiazolines containing furan rings).

Bis (chloromethyl) furans have proved to be convenient starting materials for the synthesis of new nitrogen- or sulfur-containing bi- and tricyclic systems including a furan ring. A case of a rearrangement of the allyl type of halomethylfurans in their reaction with metal cyanides has been found [193, 194], and the kinetics of the solvolysis of chloromethyl derivatives of furan has been studied [198, 199].

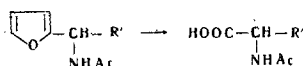
In 1938, S. M. Sherlin and A. Ya. Berlin [200] discovered a substitutive addition reaction in the furan series on the basis of the reaction of furan with acrolein.

I. N. Nazarov and T. D. Nagibina [201, 202], Yu. K. Yur'ev and N. S. Zefirov [203-207], and V. G. Glukhovtsev [208-211] have studied this reaction in detail and have developed it as a new method for the synthesis of carbonyl compounds of the furan series. It has found wide application in the synthesis of amines [212], furylquinones [213], acids, acid chlorides, esters [211], and other derivatives of furan. As an example we may give the synthesis of jasmone effected by Yu. K. Yur'ev and N. S. Zefirov [214] using the substitutive addition reaction:



Using adducts of the diene synthesis in the furan series as model compounds, Yu. K. Yur'ev and N. S. Zefirov have made a broad study of the theoretical questions connected with the Wagner-Meerwein rearrangement [214-217], homoallyl interaction [218, 219], and the stereochemistry of addition reactions to a double bond [218, 220-222].

An original method for the synthesis of amino acids, including optically active compounds, from amines of the furan series has been developed by A. P. Terent'ev and R. A. Gracheva [223-225]:



A. P. Terent'ev and L. A. Kazitsina [226-228] have shown that furan, 2-methylfuran, and 2,5-dimethylfuran are smoothly sulfonated by pyridine sulfur trioxide with the formation of mono- and disulfonic acids.

The study of the synthesis, reactions, and practical applications of furan derivatives carried out in Riga under the direction of S. A. Hiller [33, 35] has acquired an extremely wide scope. A characteristic of this school is their constant striving to convert the results of their investigation to practical use. Thus, for example, S. A. Hiller and A. Ya. Karmil'chik [229-233] have developed a method for the vapor-phase decarbonylation of furfural to furan over mixed oxide catalysts and have constructed a semi-industrial plant for obtaining furan by this method.

S. A. Hiller and M. V. Shimanskaya [233-239] have developed and introduced into industry a method for the

synthesis of maleic anhydride based on the catalytic oxidation of furfural with air. A large group of studies has been devoted to the creation of new physiologically active and medicinal preparations based on derivatives of furan and other oxygen-containing heterocycles [35, 36, 240-254].

At the present time, many compounds of the 5-nitro-furan series have found use in medicinal practice, for example, 1-[N-(nitro-2-furyl)acrylideneamino]hydantoin, under the name of "furalin".

S. A. Hiller and A. E. Berzin [255] have developed a method for alkylating furan with olefins in the presence of boron trifluoride etherate.

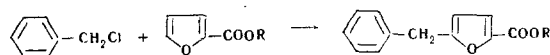
A new direction in the chemistry of furan is represented by the work of E. Ya. Lukevits, M. G. Voronkov, and S. A. Hiller [256-260] on the synthesis of organosilicon derivatives of the furan series. The hydrosilylation of 2-vinylfuran and the reactions of furan amines, alcohols, and mercaptans with chloro- and hydrosilanes have been widely studied, and the reduction of oximes with hydrosilanes has been discovered.

The investigations of Ya. P. Stradyn' and S. A. Hiller [261-265] in the field of the polarography of compounds of the furan series are interesting.

At Rostov-on-Don under the direction of Z. A. Nazarova [266-271] the behavior of the halogen atom in 5-halofurfurals and related compounds in relation to various nucleophilic agents has been studied systematically.

A study of the pharmacological activity of derivatives of the furan series is being carried out in Erevan under the direction of A. L. Mndzhoyan [13, 272-279, 597]. As a result of the investigations, the antitubercular agent "Armazid" and an agent for the treatment of glaucoma "Dimefur" have been sent for clinical trials.

A systematic study of the chloromethylation of derivatives of pyromucic acid has made possible the development of methods for the synthesis of very diverse functional derivatives of furan [5, 272-279]. Of particular interest is the synthesis proposed by A. L. Mndzhoyan and V. G. Afrikyan [274] of derivatives of benzylpyromucic acid by the following reaction:



Original investigations in the field of furan chemistry have been carried out at Saratov under the direction of A. A. Ponomarev [280-295]. He has studied the reductive amination of furan ketones and has performed the synthesis of a number of amines, diamines, and hydroxy amines of the furan and tetrahydrofuran series. With Z. V. Til [286-290] he has studied hydrazides of mixed furan acids with the object of finding physiologically active compounds.

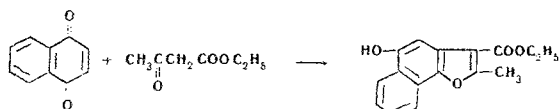
Of particular interest is the work of A. A. Ponomarev, Z. V. Til, and I. A. Markushina [286, 291, 292] in the field of 1,6-dioxaspiroonane, derivatives of which can be readily obtained by the hydrogenation of γ -carbonyl and γ -hydroxy derivatives of furan.

Dioxaspiroonane has been obtained by A. A. Ponomarev and V. V. Zelenkova [293-296] in the optically

active form by the hydrogenation of 1-(α -furyl)-3-propanol over nickel deposited on *d*- or *l*-quartz. This is a fairly rare example of absolute asymmetric synthesis in the case of reactions conducted in the presence of a heterogeneous catalyst.

Of great practical importance is the work of P. A. Moshkina, V. I. Lutkova, and N. N. Shmagina [297-299] on the synthesis of furan-2,5-dicarboxylic acid, which is a valuable starting material for the synthesis of a large range of synthetic fibers, resins, and other polymeric materials.

In the field of the chemistry of benzofuran, the work of A. N. Grinev and A. P. Terent'ev [300-302] is particularly interesting; they have developed a method for the synthesis of compounds of this series from *p*-quinones and acetoacetic ester:



The substances obtained have proved to be effective plant growth stimulators.

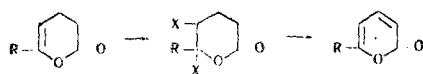
Ya. L. Gol'dfarb and L. D. Tarasova [303] have recently developed a new method for the synthesis of β -substituted furans (starting from the 4,5-dibromo derivatives formed by the bromination of furfural or 2-acetylfuran in the presence of aluminum chloride) and also of biheterocyclic condensed furan systems.

N. D. Zelinskii and N. I. Shuikin have found conditions for the smooth hydrogenation of the furan ring [136]. The hydrogenolysis of furan compounds has subsequently been widely used by N. I. Shuikin et al. [138, 304-311]. A systematic study of the use of metals of the eighth group as catalysts has shown that in the presence of platinum, iridium, and ruthenium the double bonds in the furan series are not hydrogenated but the furan ring undergoes hydrogenolysis at C-O bond with the formation of aliphatic ketones [138, 304, 305].

N. I. Shuikin, I. F. Bel'skii, and R. A. Karakhanov [306-319] have made a detailed study of the isomerization reaction of β - and γ -oxides in the presence of platinum on carbon and also of the thermal and catalytic conversions of dihydrofurans.

Of great interest is the work of N. M. Kizhner [320], who has obtained 2-(2-methylcyclopropyl) furan and an unstable isomer of 2-methylfuran, 2-methylene-2,3-dihydrofuran, and also dyes of the furan series.

The chemistry of the six-membered oxygen-containing heterocycles has been studied mainly in the case of the pyrones. Basically new methods for the synthesis of α -pyrone derivatives have been developed in the USSR. Thus, for example, R. Ya. Levina and N. P. Shusherina [38, 321-329] have proposed a method for obtaining α -pyrones by the bromination or chlorination of δ -enol lactones and the dehydrogenation of the dihalides formed:



L. I. Zakharkin and L. I. Sorokina [330] have developed another new method for the synthesis of 2-pyrones, based on the condensation of dichloroacrolein with various compounds having a labile hydrogen atom: alkyl aryl ketones, acetoacetic ester, and pyrotartaric acid.

N. P. Shusherina, R. Ya. Levina, and N. D. Dmitrieva [38] have made a detailed study of electrophilic substitution in a number of pyrones (nitration, bromination, sulfonation, chloromethylation), and have also investigated the various reactions of functional acids of derivatives of this series.

The aromatic nature of 2-pyrones has been confirmed by a study of their IR spectra (Yu. N. Sheinker et al. [331, 332]).

In conclusion, we must mention the interesting work of G. N. Dorofeenko and Yu. A. Zhdanov [333-338] on the synthesis of pyrilium salts condensed with furan or thiophene rings.

SULFUR- AND SELENIUM-CONTAINING HETEROCYCLES WITH ONE HETEROATOM

The greatest contribution to the chemistry of thiophene has been made by the work of Ya. L. Gol'dfarb.

Starting from thiophene and its simplest homologs and making wide use of chloromethylation, metalation, alkylation, and other substitution reactions, Ya. L. Gol'dfarb and his coworkers [339-367] have synthesized numerous thiophene derivatives. Their subsequent hydrogenolysis on a skeletal nickel catalyst with the elimination of sulfur has led to acyclic derivatives which would be difficult to synthesize by a different route. As an example, we may mention the synthesis of ketones and diketones with 14-28 members in a ring, for example exaltone and dihydrocivetone [368-370]. Of other investigations in the field of thiophene we must mention a convenient method for acylating thiophene with the silicoanhydrides of organic acids developed by Yu. K. Yur'ev, G. V. Elyakov, and Z. V. Belyakova [371-377].

The synthesis of reference compounds is of fundamental importance for the identification of the tetrahydrothiophenes of the sulfur-containing mineral oils of the USSR. For this purpose a large number of tetrahydrothiophenes of very diverse structure has been synthesized and studied spectroscopically by Yu. K. Yur'ev and E. G. Rozantsev [377].

M. G. Voronkov and A. S. Broun [378-380] have developed a convenient method for the synthesis of phenylthiophenes which is based on the reaction of elementary sulfur with isomeric phenylbutenes, phenylbutanes, or phenylbutadienes and also their *exo*-alkyl substituted derivatives.

The study of the degradation of the thiophene and tetrahydrothiophene rings is of great practical importance, since these reactions are at the basis of industrial methods for desulfurizing mineral oils. In this area, we must mention the work of N. D. Zelinskii [381], and also that of I. N. Tits-Skvortsova, T. A. Danilova [382, 383], and R. D. Obolentsev [384] (Ufa).

Of investigations in the field of heterocyclic systems containing one sulfur atom, we must also mention the work of N. I. Putokhin's school [385, 386] devoted to the chemistry of aryl- and thienyl-substituted thiophenes, the original method for synthesizing hydrothioxanthenes developed by V. G. Kharchenko [387], and the synthesis of thiobicyclanes proposed by G. D. Gal'perin et al. [388].

The investigations of A. I. Shatenshtein and A. G. Kamrad [389] on the reactivity of thiophene and its derivatives (and also furan) by the deuterioexchange method must be regarded as particularly valuable for the chemistry of thiophene. The kinetics of the solvolysis of chloromethyl derivatives of thiophene (and furan) has also been studied [396].

A great contribution to the chemistry of selenophene, a little-studied five-membered heterocycle very closely analogous to thiophene, has been made by Yu. K. Yur'ev, N. N. Magdesieva (Mezentseva), N. K. Sadova, and M. A. Gal'bershtam [390-397]. They have developed a convenient method for the synthesis of selenophene by the reaction of butenes with metallic selenium. A detailed study of electrophilic substitution in the selenophene nucleus [394] has permitted the synthesis of various functional derivatives of the selenophene series, the study of their reactivities, and the finding of methods for their practical utilization (as antioxidants, complex-forming agents, and physiologically active compounds). The kinetics of the mercuriation of selenophene derivatives [396], the kinetics of the isotope exchange of deuterioselenophenes [397], and the kinetics of the solvolysis of chloromethyl derivatives of selenophene have been studied. As a result of these investigations it has been possible to elucidate the position of selenophene among the five-membered heterocyclic compounds with aromatic properties.

NITROGEN-CONTAINING HETEROCYCLES WITH ONE HETEROATOM

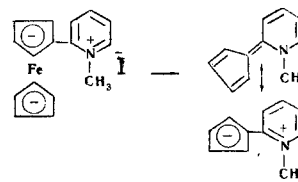
In the field of nitrogen-containing heterocycles, Soviet chemists have made a number of basic scientific discoveries.

Even in the years immediately after the revolution, V. V. Chelintsev and his colleagues A. P. Terent'ev, B. V. Tronov, and B. V. Maksorov carried out investigations in the field of chemistry of pyrrole [398-401]. T. N. Godnev has also devoted himself to the study of derivatives of this heterocycle, including chlorophyll [402].

At the present time, syntheses of natural compounds of the porphyrin series (based on pyrrole derivatives) are being carried out and their properties are being studied under the direction of N. A. Preobrazhenskii, R. P. Evstigneeva, and A. F. Mironov [403-409]. Thus, for example, the study of the question of salt formation in the acetylpyrroles has enabled our knowledge of the mechanism of the synthesis of dipyrromethenes from aldehydes of the pyrrole series to be extended [408, 409].

An extremely interesting synthesis of a heterocyclic derivative of ferrocene, ferrocenylpyrrole, has been

effected by A. N. Nesmeyanov and V. A. Sazonova [410, 411]. Later, they found that heterocyclic ferrocene derivatives (for example, ferrocenylpyrimidine methiodide) readily decompose with the formation of ylides:



In a number of papers, A. P. Terent'ev and L. A. Yanovskaya [187, 412] have shown that compounds of the pyrrole series are smoothly sulfonated by pyridine/sulfur trioxide to α -monosulfonic acids.

In the field of six-membered nitrogen-containing heterocycles we must particularly mention the classical studies of A. E. Chichibabin [11]. The reaction discovered by Chichibabin and O. A. Zaide in 1914 of the amination of pyridine and its derivatives with sodium amide [57], which has acquired the name of the "Chichibabin [Tschitschibabin] reaction" in world literature, is widely used to obtain very diverse amino derivatives of pyridine, quinoline, isoquinoline, pyrimidazole, etc. [57, 413, 414]. The use of this reaction by A. E. Chichibabin himself and his pupils (O. A. Zaide, A. V. Kirsanov, I. L. Knunyants, N. N. Vorozhtsov, F. N. Stepanov, et al.) has enabled them to bring about a wide development of the chemistry of many nitrogen-containing heterocyclic systems.

From 2-aminopyridine, I. Ya. Postovskii [415-419] has synthesized various sulfonamide preparations (including Sulfidine).

The tautomerism of derivatives of 2-aminopyridine discovered by A. E. Chichibabin is extremely interesting [420].

Work in the field of the tautomerism of amino derivatives of heterocyclic compounds has been carried out by Yu. N. Sheinker. By making use of the law of tautomeric equilibria discovered by M. I. Kabachnik [421-422], he found a connection between the position tautomeric equilibrium and the nature of the substituents on the nitrogen atoms of the amino groups [423, 424]. The method of synthesizing pyridine derivatives from acetaldehyde and ammonia developed by A. E. Chichibabin is being studied at the present time by M. I. Farberov [425] (Yaroslavl) in order to use this reaction for the industrial synthesis of pyridine derivatives.

Investigations in the field of pyridine chemistry are also being carried out by Yu. I. Chumakov [58, 426] (Kiev). He has developed a general method for the synthesis of pyridine bases which is founded on the saponification of 2-alkoxy-3, 4-dihydropyrans to 1, 5-dicarbonyl compounds and the reaction of the latter with hydroxylamine.

An interesting synthesis of pyridines by the dehydration of γ -piperidols with subsequent catalytic dehydrogenation have been developed by N. S. Prostavkov [427-429].

The Chichibabin reaction can be used successfully for the synthesis of quinoline bases. Thus, when a mixture of acetaldehyde and aniline vapors is passed over alumina at 450–500° C, lepidine is obtained in good yield. An original method for synthesizing quinolines has been developed by N. S. Kozlov [430–432] (Perm). It is based on the reaction of aniline and other aromatic amines with acetylene in the presence of salts of copper, mercury, or silver.

A convenient method for obtaining 4,4-bipyridyl has been proposed by A. E. Arbuzov [433].

A valuable contribution to the chemistry of indole has been made by the investigations of A. E. Porai-Koshits [485, 593] and V. I. Minaev [594] in the field of indigo and indigoid dyes.

We may also mention the work of Z. V. Pushkareva [434–435] on quinaldine derivatives. This question is directly connected with the chemical utilization of coal tar bases.

Under the direction of G. Ya. Vanag [436–442] (Riga), detailed studies of the chemistry of β -dicarbonyl compounds and, in particular, of indandione derivatives has been carried out. The synthesis of various complex heterocyclic systems from this diketone has been effected. Thus, for example, the Beckmann rearrangement of 2-nitroindandione oxime gives 1-chloro-3-nitro-4-hydroxyisoquinoline.

L. S. Povarov and B. M. Mikhaiov [443–449] have found that vinyl alkyl esters react with Schiff's bases in the presence of catalytic amounts of Lewis acids (BF_3 , AlCl_3 , AlBr_3). This gives rise to derivatives of 4-alkoxy-1,2,3,4-tetrahydroquinoline, which can easily be converted into the corresponding quinolines. This method opens up wide possibilities for the synthesis of quinoline and tetrahydroquinoline derivatives.

The new method for synthesizing compounds of the pyridine series developed by G. Ya. Kondrat'eva [450, 451] is worthy of attention. It is based on the reaction of oxazoles with various philodienes under the conditions of the diene synthesis and leads to the formation of substituted cinchomeronic (pyridine-3,4-dicarboxylic) acids.

Indole derivatives play an essential role in vital processes. It is sufficient to mention such compounds of the indole series as tryptophan, serotonin, bufotenin, reserpine, strychnine, and psilocine. Extremely intensive studies of the chemistry of indole are proceeding in the Soviet Union at the present time.

Many of them are devoted to the synthesis of indoles from arylhydrazones by means of the Fischer reaction as modified and improved (as early as 1910) by A. E. Arbuzov (cf. [452]) [62].

Using this, N. N. Suvorov et al. [452–461] (Moscow) have developed the synthesis of the known stimulator of plant root formation, 3-indolylbutyric acid. Acids containing substituents in various positions of the pyrrole and benzene rings have been obtained in the same way.

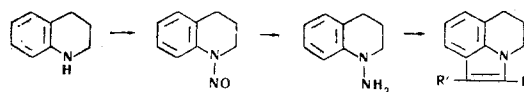
N. N. Suvorov et al. have developed a new method for obtaining serotonin from γ -formylpropionic acid and *p*-benzoyloxyphenylhydrazine.

N. N. Suvorov's work [62] has played a great part in establishing the mechanism of the Fischer reaction.

A. N. Kost and L. G. Yudin [462, 463] have found that keto nitriles can take part in the Fischer reaction and, depending on the reaction conditions and the structure of the initial compounds, nitriles of both the indole and the indolenine series, and also substituted indoles, can be obtained.

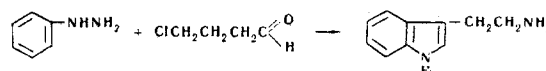
A convenient method for the synthesis of 2-methyltryptophol has been proposed by I. I. Grandberg, A. N. Kost, and A. P. Terent'ev [464, 465].

A. N. Kost, L. G. Yudin, A. P. Terent'ev, and B. A. Dmitriev [466, 467] have developed the following elegant method for the synthesis of lilolidene structures:



Of other methods of constructing the indole ring we may mention the method for the synthesis of indoles developed by A. I. Grinev and A. P. Terent'ev [468–473], which is based on the Nenitzescu reaction and consists in the condensation of quinones with aminocrotonic ester and its analogs.

I. I. Grandberg et al. [474] have discovered a new and simple method for the synthesis of tryptamines starting from arylhydrazones and γ -halogeno carbonyl compounds [422, 423]:



We must mention the work of A. N. Kost, L. G. Yudin, and V. A. Budylin [475], who have studied electrophilic substitution in 2,3-substituted indoles as a function of the acidity of the medium.

L. N. Yakhontov [476, 477] (Moscow) has carried out general investigations in the field of azaindoles in a search for new physiologically active substances.

It is impossible not to mention the work of A. E. Arbuzov [478] on the tautomerism of isatin.

In the field of acridine chemistry, the largest number of investigations (I. L. Knunyants, G. V. Chelintsev, and O. Yu. Magidson) has been devoted to the synthesis of the valuable antimalarial agent acrichine and its analogs [479–482]. Of the other investigations in this field, we may mention those of A. M. Grigorovskii [484] devoted to a study of the chemistry of the luminescence reactions of lucigerine (10,10-dimethylbisacridilium nitrate).

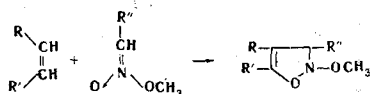
Of other synthetic studies in the field of heterocycles containing one nitrogen heteroatom we must also refer to the work of A. E. Porai-Koshits [485], O. Yu. Magidson [488], M. V. Rubtsova [486], I. Ya. Postovskii [487], N. N. Vorozhtsova [488], B. P. Lugovkina [489], P. A. Petyunina [490], Ts. V. Tsukerman [491], and others.

HETEROCYCLES CONTAINING SEVERAL HETEROATOMS

Heterocycles having aromatic properties and containing two or more heteroatoms form such a large group of compounds that to mention even briefly all the directions of work in this field is not possible. The main achievements of Soviet chemists in the field of the chemistry of heterocyclic compounds are connected with the study of derivatives of isoxazole, pyrazole, imidazole, pyrimidine, and their hydrides.

Thus, N. K. Kochetkov, E. D. Khomutova, and S. D. Sokolov [492, 493] have studied the chemical properties of isoxazole. On the basis of a large number of examples they have shown that electrophilic substitution (nitration, sulfonation, halogenation, mercuration) takes place in position 4 of the oxazole ring. It has also been shown that isoxazole somewhat resembles pyridine in its properties.

An original method for the synthesis of isoxazolidine derivatives has been developed by V. A. Tartakovskii and S. S. Novikov [494-497] (Moscow) on the basis of the 1,3-dipolar addition of esters of the aci form of nitro compounds to olefins, for example:



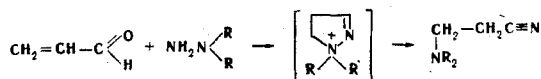
A great contribution to the chemistry of heterocyclic derivatives of hydrazine has been made by A. N. Kost and I. I. Grandberg. A series of their first papers was devoted to the synthesis of pyrazolines from amines under the action of acid catalysts [498, 499].

After these workers had found a convenient method for dehydrogenating pyrazolines to pyrazoles using sulfur as dehydrogenating agent [500-507], broad and systematic work on the chemical properties of pyrazole systems was begun. Particular interest is offered by a study of electrophilic substitution in the pyrazole ring [508-510]. I. I. Grandberg [508] showed that electrophilic substitution takes place in position 4 of the pyrazole ring.

A series of I. I. Grandberg's investigations was devoted to the physicochemical properties of various pyrazole derivatives. Thus, for example, dipole moments were determined [511], IR, UV, NMR, and fluorescence spectra were studied, etc.

I. I. Grandberg [512, 519] has also described the rearrangement of pyrazolines and their derivatives into β -amino nitriles.

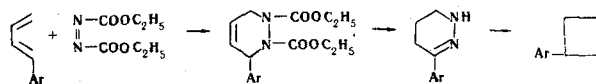
B. V. Ioffe and K. N. Zelenin [520] have found that the reaction of α, β -unsaturated carbonyl compounds with dialkylhydrazines leads to an amino nitrile rearrangement which takes place via the intermediate stage of the formation of quaternary pyrazoline derivatives:



Yu. A. Naumov, I. I. Grandberg, and A. N. Kost [521, 522] have studied the factors affecting reactions

taking place with the cleavage of the N-N bond and have shown the kinetic relationship of the amino nitrile rearrangement to reactions of various hydrazine derivatives.

The chemistry of the tetrahydropyridazines has been developed by R. Ya. Levina, Yu. S. Shabarov, and M. G. Kuz'min [523-527]. They have synthesized numerous compounds of the tetrahydropyridazine series, especially from azodiaryls by means of the diene synthesis with axodienophiles. The latter were used as starting materials in the development of an interesting method for synthesizing cyclobutanes in the following way:



O. S. Otroshchenko and A. S. Sadykov [528-532] have carried out systematic investigations in the field of bipyridyl chemistry. They have developed methods for synthesizing bipyridyls and have studied the direction of electrophilic substitution reactions.

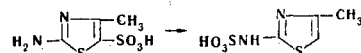
A large range of derivatives of thienopyridine, thienothienopyridine, thienothiazole, and thienopyrrolenine has been synthesized by V. G. Zhiryakov [533-536].

One of the directions of the studies carried out by M. A. Prokof'ev and Yu. P. Shvachkin [537-548] is connected with the study of pyrimidylamino acids. These compounds are of interest as potential antimetabolites, substances that participate in the nuclein-protein metabolism of the organism. The authors succeeded in developing general methods for synthesizing previously unknown pyrimidylamino acids of all theoretically possible types. Very recently, pyrimidylamino acids have been detected in natural materials.

By using methods which they had developed, M. A. Prokof'ev and Yu. P. Shvachkin [540-542] have performed the first chemical synthesis of a new amino acid derivative, lathyrine.

Here we must also mention the fact that derivatives of tetrahydropyrimidine are readily obtained from acylated β -amino acids by a method developed by V. M. Rodionov, V. V. Kislova, and V. K. Zvorykina [549].

O Yu. Magidson and K. A. Chkhikvadze [550, 551] have synthesized and studied a number of new derivatives of orotic acid and its 2-amino and 2-thio analogs. A large contribution to the chemistry of heterocycles with two heteroatoms has been made by the work of I. Ya. Postovskii [552]. He has made a detailed study of various sulfo derivatives of thiazole and has found an unusual case of the isomerization of an amino sulfonic acid of thiazole into the corresponding sulfamic acid [552, 553]:



Yu. N. Sheinker and I. Ya. Postovskii [553-560] have studied questions of thione-thiol and amino-imino tautomerism in a number of mercapto and amino derivatives of thiazole.

V. L. Nirenburg and I. Ya. Postovskii [559] have found a peculiar thermal isomerization of 5-hydrazino-1-phenyltetrazole into 1-amino-5-anilinetetrazole.

The so-called tetrazole-azide tautomerism, which has been studied in relation to azide derivatives of nitrogen-containing heterocycles, is of great theoretical interest [560, 561].

An interesting series of investigations in the field of benzazoles and naphthazoles has been carried out by N. P. Bednyagina, I. N. Getsova, and I. Ya. Postovskii [562].

Broad and extremely interesting investigations have been carried out in the imidazole series by P. M. Kochergin [68, 563] and A. M. Simonov [68, 71], in the benzimidazole series by A. M. Simonov, B. A. Porai-Koshits, and L. S. Efros [565, 566], and in the pyrimidine series by V. P. Mamaev [567].

We must also mention the work of I. L. Knunyants [568] in the thiazole field. A new class of heterocyclic compounds—the pyrimidineamidazolones—has been discovered by M. A. Prokof'ev and Yu. P. Shvachkin [569].

A. S. Elina and O. Yu. Magidson [570-572] have carried out investigations in the field of the N-oxides of quinoxaline, pyrazine, and their derivatives. Original methods for the synthesis of derivatives of these heterocycles and their N-oxides, previously difficult to obtain, have been proposed. Practicable routes for obtaining the N-oxides of quinoxaline containing amino, hydroxy, carboxy, alkylcarboxy, alkyl, hydroxyalkyl, and other substituents in the pyrazine part of the molecule have been found.

Interesting studies on 5-amino-1,2,4-triazoles have been carried out by V. Ya. Grinshtein and G. I. Chipen [573-577], who have developed a method of obtaining aminotriazoles and their derivatives.

A large number of heterocycles of various structures containing several heteroatoms has been synthesized by I. K. Korobitsina and Yu. K. Yur'ev [578-582] from 1,2-diketones of the tetrahydrofuran series.

I. K. Mushkalo [583] has synthesized a large number of derivatives of benzothiazine, benzoheptathiazine, thiazoline, and thiazolidine, and has studied their reactivities.

Heterocyclic systems containing two or more heteroatoms have also been the subject of investigations by S. N. Baronov [80, 584, 585], V. G. Pesin [586] (thia- and selenadiazoles), V. A. Krasovskii and S. I. Burmistrov [587] (2-aminothiazole), B. M. Turkevich [588] (rhodanine derivatives), S. V. Zhuravlev [589] (phenothiazine), and others.

In 1944-1947, M. G. Voronkov and A. S. Broun [378, 590, 591] discovered a new class of five-membered heterocycles possessing aromatic properties containing two sulfur atoms in the ring, aryl- and alkyl-substituted 1,2-dithiol-3-thiones, and a method for their production based on the sulfonation of the corresponding arylalkenes and alkenes. On recently continuing these investigations, M. G. Voronkov and T. V. Lapina [186, 592] have developed a convenient catalytic method for synthesizing 4- and 5-aryl-1,2-dithiol-3-thiones starting from elementary sulfur and

1- and 2-arylpropanes and their derivatives. The dipole moments and electronic structures of these compounds have been studied [595]. R. O. Matevosyan et al. [598-600] have studied the stable free radicals obtained from heterocyclic hydrazines (derivatives of thiazole, imidazole, oxazole, etc.). These investigations have fundamental importance for elucidating the fine chemical structure and reactivity of such heterocyclic systems.

In this review, the end of which we are now approaching, we have repeatedly emphasized that it is not possible to give any kind of exhaustive account of all the interesting results of investigations in the field of the chemistry of heterocyclic compounds having aromatic properties obtained by Soviet scientists in the last 50 years. We have mentioned only the most fundamental of them and have shown the main directions being followed in this field of investigation.

The material given above clearly indicates the vigorous development of the chemistry of heterocyclic compounds in many Soviet scientific centers. A fundamental role in the advances achieved has been played by the leading organic chemists of the USSR who have constantly devoted great attention and interest to this very important branch of organic chemistry.

The broad investigations discussed have, on the one hand, enabled basically new features in the reactions of organic compounds to be discovered and thus have made possible the development of new theoretical ideas in organic chemistry and the solution of important problems of modern bioorganic chemistry, biochemistry, and pharmacology.

On the other hand, the enormous practical importance of these investigations is quite obvious, since they form the basis of the creation of new medicinal and veterinary agents, dyes, plant growth regulators, pesticides, intermediates of fine organic synthesis, polymeric materials, etc.

A characteristic feature of the development of the chemistry of heterocyclic compounds in the Soviet Union is the ever-increasing use of physical and physicochemical methods of studying these substances and their reactions.

All this shows the unusually wide range of problems that Soviet scientists working in the field of the chemistry of heterocyclic compounds have set themselves and successfully solved.

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