Journal of Fluorine Chemistry 131 (2010) 123–126

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Editorial

Fluorine chemistry in Ukraine

The beginning of fluorine chemistry in Ukraine is not unique; as many times before in the history of science, it took an overlap of social opportunity and extraordinary personality. As story has it, sometime in 1948, Kiev State University professor, academician Andrey Kiprianov (one of the major contributors to the theory of color of organic compounds), addressing an ambitious request from his graduate student Lev Yagupolskii to try something all-out new as a subject of his PhD dissertation, suggested to take a shot at the virtually unknown at that time chemistry of fluoroorganic compounds, in particular heterocyclic derivatives. Professor Kiprianov also quite reasonably suggested his student to think about less ambitious backup project as chances of success in the uncharted waters of fluoroorganic chemistry were next to nothing. As it turned out, the discussion ushered in the birth of the storied program of fluoroorganic chemistry in Ukraine.

The results obtained by Lev Yagupolskii in the course of his PhD work, and by his group in the following years, clearly underscored the scientific gravity of fluoroorganic chemistry as a distinct discipline, which in 1965 resulted in the establishment of the Department of Fluoroorganic Compounds within the Institute of Organic Chemistry (IOCH) of the Ukrainian Academy of Sciences, Kiev, Ukraine. From that time, Professor Lev Yagupolskii has headed a full-scale research program in various areas of fluoroorganic chemistry spawning new research groups throughout Ukraine and gaining national and international recognition. In 1988 Professor Lev Yagupolskii was succeeded by his son Yurii, as Head of the Department, however, he continued the research work and mentoring PhD students until the last day of his life. His research family includes 87 PhD and 10 Dr.Sc. professionals who contributed their talent to the numerous discoveries and establishment of the Ukrainian brand of fluorine chemistry, the major milestones of which are listed below.

Fluorine containing substituents, the main structural building bricks in the design of complex functional organic molecules, were traditionally in focus of the Ukrainian fluorine chemistry. More than 60 new fluorine-containing groups had been developed and fully characterized from the standpoint of their influence on chemical reactivity and physicochemical properties of organic compounds [1].

In particular, using the reaction of the corresponding chlorine derivatives with SbF_3 or HF the compounds with OCF_3, $-O-CF_2-O-$ and $-CF_2-O-CF_2-$ groups were prepared for the first time, i.e., aryl trifluoromethyl ethers, 1,2-difluoromethylenedioxybenzenes and 1,1,3,3-tetrafluoro-1,3-dihydroisobenzofurans, respectively [2,3]. Thus, aryl trifluoromethyl sulfides and *N,N-bis*(trifluoromethyl)anilines were synthesized by this method [4].

The electronic nature of fluorine-containing substituents manifests itself most notably in effects directing the pattern of aromatic electrophilic substitution reactions. Of particular importance was the discovery that hydroxy- and amino-derived substituents retain their *ortho*- and *para*- directive nature even after replacement of corresponding hydrogen atoms with strong electron-withdrawing groups such as CF_3 and SO_2CF_3 [5].

Thus the OCF_3, OSO_2CF_3 and $NHSO_2CF_3$ groups were shown to direct the incoming nitro group mainly to the *para*-position. On the other hand, the directive nature of trivalent nitrogen atom can be eventually changed by the combined force of two SO_2CF_3 groups; the nitration of *N,N-bis*(trifluoromethylsulfonyl)aniline gives 80% of the *meta*- and 10% of the *para*- substituted products [5].

This fundamental chemistry of new fluorine-containing substituents required the development of the corresponding methodology allowing preparing and introducing a particular substituent in the desired position on aromatic ring.

Particular attention was given to the exploration of sulfur tetrafluoride as fluorinating agent. This direction of research was conducted in the close cooperation with a group of scientists from the Odessa Polytechnic Institute (now the University), headed by Prof. Lyubov Alekseeva. With great contribution from of young enthusiastic chemists (late Prof. Anatolii Burmakov, Prof. Boris Kunshenko) truly practical method of SF_4 production was developed and applied in IOC on the large-scale. Significantly, this collaborative research resulted in the development of new fluorinating system, SF_4–HF–Cl_2/SCl_2, allowing for practical preparation of various aliphatic fluoroorganic compounds [6].

Ion-radical perfluoroalkylation of thiols with perfluoroalkyl iodides via $S_{RN}1$ mechanism is another example of the discovery of new reaction chemistry made by Professor Lev Yagupolskii's group. The generality of this reaction for preparation of aliphatic and aromatic compounds, even amino acids, was clearly demonstrated rendering this process the most convenient and straightforward approach for practical introduction of highly lipophilic -S-R_f groups. It should be noted that the development of this reaction allowed for systematic study of chemistry of SO_2CF_3 group, which can be obtained by oxidation of the corresponding $-SCF_3$ precursors. [7]

Professor Lev Yagupolskii's group was one of the pioneers in and major contributor to the field of electrophilic perfluoroalkylation. The transfer of perfluoroalkyl group from one compound to another as electrophile is one of the most ingenious new paradigms in fluorine chemistry. The work that resulted in this discovery involved the study of new type of compounds, containing perfluoroalkyl substituent bonded to a heteroatom bearing a positive charge. Such synthetically useful reagents as aryl perfluoroalkyliodonium [8] and diphenyl perfluoroalkyl sulfonium

0022-1139/$ – see front matter
doi:10.1016/j.jfluchem.2009.12.011

[9] salts were reported for the first time by Professor Lev Yagupolskii's group and inspired many new and original ideas to further develop the electrophilic perfluoroalkylation.

Noteworthy to mention the family of some "exotic" compounds developed by Professor Lev Yagupolskii's group, such as $PhPF_4$ (via oxidative fluorination of $PhPCl_3$ with SbF_3) [10]; *tris*(perfluoroalkyl)difluorophosphoranes (via electrochemical fluorination of trialkylphosphine oxides, despite the well-known fact of poisoning of electrodes by P-derivatives) [11]; fluorine containing pentavalent iodine compounds [12]; convenient methods of $CuCF_3$ and $Hg(SCF_3)_2$ generation [13].

A separate chapter in the history of Ukrainian fluorine chemistry is the design and synthesis of first organic compounds containing perfluorinated unsaturated chains bearing aromatic moieties on the ends of the chain, generally, diarylperfluoropolyenes [14] as analogs of cyanine dyes. Over 50 years ago it was necessary to prove the currently well-established fact that perfluorinated C=C chains are capable of transferring the electronic effects and therefore can be used in the design of organic dyes. As a highlight of this work is the original method for preparation of carbocyanine dye with perfluorinated trimethine chain; the synthesis of analogues dicarbocyanine dye derivatives is presented in this Special Issue.

Among the novel structures of organic molecules developed by Professor Lev Yagupolskii's group, one can mention the σ-complexes of aromatic compounds with perfluoroalkylsulfonyl and fluorosulphonyl groups [15], hexasubstituted benzenes with trifluoromethylthio groups [16], the class of strong and superstrong –OH, CH– and NH-acids. Systematic work in the latter area resulted in the discovery of new principle, bearing the name of Professor Yagupolskii, consisting in substitution of the oxygen in a E=O function with NSO_2CF_3 [17]. This principle allows for the rational design of super strong electron withdrawing groups reaching σ_p values 1.4–1.75 [conversion of SO_2CF_3 into $S(O)(=NSO_2CF_3)CF_3$ and SO_2F in $S(O)(NSO_2CF_3)F$ and $S(=NSO_2CF_3)_2F$]. On the basis of this principle the most powerful organic acid, *bis*(trifluoromethylsulfonylimino)trifluoromethansulfonic acid $CF_3S(=NSO_2CF_3)_2OH$ was synthesized and its acid strength ($-H_o$ is about 24) was estimated by ^{29}Si–NMR spectroscopy [18].

The area of fluorine-containing biologically active compounds has also received due attention in the realm of scientific interest of Professor Lev Yagupolskii's group. One of the most noticeable achievements (in collaboration with Riga Institute of Organic Chemistry, Academy of Sciences of Latvia) was the development of hypotensive medicinal product Foridon, which has $OCHF_2$ group in the *ortho*-position on the benzene moiety and acting as a calcium channel modulator. The therapeutic success of the drug suggested general application of this new fluorine-containing group as pharmacophore unit in the design of pharmaceutical products. For instance, a similar low-toxicity drug Flocalin, acting as calcium channel activator and showing cardiotonic activity, is currently in the phase of preclinical trials.

The results of systematic study of the effect of fluorine-containing substituents on the property of drugs used for treatment of cardiovascular diseases were summarized in the monograph Organofluorine Compounds in Medicinal Chemistry and Biomedical Application (Elsevier, 1993) edited by R. Filler (USA), Y. Kobayashi (Japan) and L.M. Yagupolskii (Ukraine).

Study of trimethyltrifluoromethylsilane chemistry and its application as trifluoromethylating reagent was conducted by Professor Yurii Yagupolskii's group in collaboration with Prof. D. Naumann and Dr. W. Tyrra (Cologne University, Germany). This work allowed for preparation of variety of trifluoromethyl containing compounds and made a significant contribution to the understanding of new mechanistic aspects of this chemistry [19].

One of the most important directions in the chemistry of organo-fluorine compounds, originated in another Department of the Kiev's Institute of Organic Chemistry under guidance of late Academician Prof. Leonid N. Markovsky and his successor Prof. Dr. Yury G. Shermolovich, is a study of the tetra-coordinated sulfur (+4), containing one or several fluorine atoms near sulfur atom-fluorosulfuranes. These compounds are widely used in the organic synthesis as fluorinating agents and as starting materials for the synthesis of various sulfur-containing compounds [20].

Most recently, a synthetically simple and generalized method for preparation of fluorosulfuranes was developed, making use of the reaction of dialkyldisulfides with chlorine in presence of excess amounts of fine-dispersed potassium fluoride. The discovery of this method gave an opportunity to obtain trifluorosulfurane derivatives of various useful types, such as DAST, aryltrifluorosulfuranes and the first examples of heteryltrifluorosulfuranes, in which the trifluorosulfurane group is adjacent to the heterocyclic carbon atom [21].

The achievements in investigation of 1,1-dihydropolyfluoroalkylsulfides allowed to elaborate a general strategy of their synthetic application in a form of successive processes via dehydrofluorination–nucleophilic substitution, involving substitution of the hydrogen atoms in the α-methylene group by chlorine. This approach allowed to develop convenient methods for preparation of the reactive fluorine and sulfur containing acyclic compounds, such as olefins, acetylenes, amines, imines, enamines, vinylazides, vinylizothiocyanates, thiocarbonic acid derivatives and heterocyclic derivatives, for example pyroles, pyrazoles, triazoles, uraciles, and pyrimidines [22]. In another research direction of this group, chemical properties of polyfluoroalkyldithiocarboxilates received a particular attention. A critical influence of fluorine-containing alkyl substituent on the [3 + 2]- and [2 + 4]-cycloaddition reactions of these compounds was demonstrated [23].

The new types of fluorine-containing thiocarbonyl compounds containing in α-position to the thiocarbonyl function a carbonyl group were obtained, they included: dithioesters of difluoropyrograpе acid and polyfluoroalkyl-1-thioxo-2-oxosulfones. Application of these compounds in cycloaddition reactions allowed preparation for the first time fluorine-containing ketones with oxathiines substituents [24].

The synthetic methods for acyclic pentapolyfluoroalkoxyphosphoranes were elaborated on the basis of 1,1-dihydropolyfluoroalkylphosphites adducts with chlorine and bromine. These compounds appeared to be convenient reagents for the alcohols, mercaptanes, amines, aldehydes and ketones, and also for carboxylic and phosphorus acids polyfluoroalkylation.

Since 1986, when the Institute of Bioorganic Chemistry and Petrochemistry (IBCP) was established, academician Valery Kukhar initiated the research in fluorine chemistry which focused on development of asymmetric methods for preparation of biologically relevant, fluorine-containing analogs of α-, β- and γ-amino acids, α-hydroxy acids, amines, as well as their phosphorus and sulfur derived compounds [25]. These novel synthetic methods are represented by asymmetric homologation of nucleophilic glycine equivalents using fluorinated substrates *via* alkyl halide alkylation [26], aldol [27] and Michael addition reactions [28], a biomimetic reductive amination of fluorinated carbonyl compounds to the corresponding amines and amino acids [29] as well as using sulfinyl auxiliaries for the preparation of fluorine-containing amino compounds [30].

Research efforts in the IBCP are also directed at synthetic strategies based on an application of readily available β-alkoxyvinyl polyfluoroalkyl ketones (synthetic equivalents of 1,3-ketoaldehydes) for practical preparation of β-aminovinyl polyfluoroalkyl ketones (useful as *N*-protected components in peptide synthesis), fluorinated heterocycles (pyrones, pyrazoles, isoxazoles pyrroles pyrimidines, etc.), γ-aminobutyric and mevalonic acids [31].

In another research project conducted in IBCP the preparation of various types of enantiomerically pure fluorinated amino acids was realized by resolution of racemic mixtures with application of hydrolytic enzymes [32]. New regioselective deoxyfluorination of propargylic ketones and alcohols is studied and applied to synthesis of fluoro-containing unsaturated fatty acids and corresponding metabolites.

In the Institute of Physical Chemistry (IPC) the investigation of electrochemical aspects of Freons' reactivity is currently in progress. This study includes utilization of various electron transfer mediators, nontraditional mediums (low temperature ionic liquids) and special electrodes, processes of homogeneous catalytic free radical fluoroalkylation of Freons with organic substrates (thiophenols, phenols, heterocyclic compounds) and carboxylation of fluorine containing imines as a general approach to fluorinated amino acids [33].

Organofluorine chemistry in Ukraine is represented well not only in diverse academic organizations, but also in a commercial company Enamine Ltd. Research and Development department of Enamine has tight collaborations with Kiev National Taras Shevchenko University and IOC on the development of new methods for the synthesis of biologically relevant fluoroorganic compounds. In particular, the chemistry of fluorinated amino acids [34], nucleosides [35], and heterocycles [36] is being developed at Enamine. Such reagents as SF_4, CF_3I, SbF_3, MeTFP, CF_3CHN_2 are routinely used by the practitioners at Enamine as fluorinating tools to prepare various fluorinated building blocks.

The readers can find more detailed information about current research projects conducted by Ukrainian fluoroorganic chemists in the collection of manuscripts included in this Special Issues.

We would like to express our gratitude to the editorial board of the Journal of Fluorine Chemistry, editorial staff, all authors and referees for making this Issue possible, and hope it will be of interest to the readership of this Journal.

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Available online 21 December 2009