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Miscellaneous

About Ivan Ludvigovich Knunyants

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Abstract

My memories of Academician I.L. Knunyants—the founder of Fluorine Chemistry in Russia.

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1. Introduction

The first time I met I.L. Knunyants over a half century ago, in 1954 when my classmates Lev German, Boris Dyatkin and I have just graduated from the Chemistry Department of the Lomonosov's Moscow State University and were assigned to the laboratory headed by I.L. Knunyants at the Institute of Organoelement Compounds (known also as INEOS by its Russian acronyms) of the USSR Academy of Sciences. We were aware that Knunyants had synthesized the anti-malarial drug acrichin and anticipated to work under Dr. O.V. Kildisheva, who was in charge of the medicinal chemistry group developing new drugs in the Knunyants's lab. We were surprised and disappointed when I.L. told us that we would be doing research in fluorine organic chemistry. Seeing our downcast faces, I.L. firmly repeated ''Only fluorine and nothing else. If you don't like it look for a job elsewhere.'' Since we were only senior lab technicians, lowly lieutenants in the army reserve, we had no choice but to meekly agree with Knunyants, a full member of the Academy of Sciences and an active Mayor General of the Soviet Army. Now, many years later, I look upon that four-person meeting as the birth of Fluorine Chemistry in the USSR Academy of Sciences.

In retrospect I realize that it was not only a good decision, but also a decision that impacted my entire life. In those days not many scientists were working in fluorine chemistry. Many novel research results that we achieved under IL's leadership were pioneering achievements and were published in chemical periodicals abroad. Fortunately, my name became quite well known in the scientific literature and, because of that, 3M offered me a position at the age of 60, when my wife and I moved to the USA in 1991. I worked at 3M for 11 years, doing research and developing new products based on fluorine.

2. I.L. Knunyants and fluorine chemistry

Our first workplace back in 1954 was the old chemical lab of Professor A.E. Chichibabin, IL's former teacher. It was a two-story building located in a row of old residential houses. The building belonged to INEOC AN USSR, but was on Bauman Street, remote from the main buildings of the institute. It was a 10-min walk from the Military Academy of Chemical Defense (MACD), where IL was in charge of a large department. It was convenient for him to drop in at our building two or three times a week to see how things were going and to direct our research.

Our first assignment from IL was the synthesis of perfluoroisobutylene (PFIB), a colorless, odorless gas. He thought that PFIB might become an effective War Gas because inhalation of only 4–5 l of PFIB gas or 6-h exposure to it in concentration as low as 0.5 ppm could cause fatal pulmonary edema. We have been making PFIB by polytetrafluoroethylene pyrolysis, using primitive homemade tools (steel pipe, heating oven, etc.). The mixture containing PFIB was distilled in a low-temperature Dewar column with rubber joints. The column was too large to fit into the hood so it was set up on the floor nearby. The most dangerous part of the operation was draining the cold (−78 °C) gas mixture from the glass trap into the column

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distillation flask through a piece of rubber hose. Rubber is brittle at such low temperatures and the hose would fracture now and then. Whenever this happened the mixture containing the lethal PFIB would spill onto the floor. We bolted to the street outside while one of us would hold his breath and pour ammonia solution onto the floor. A container of ammonia solution was always kept handy near the lab entrance. It is remarkable that in all the years that we worked with PFIB, there was not a single injury. Only later it was found that PFIB could not be used as a chemical weapon anyway, because it reacts with water and therefore quickly decomposes when released into the humid atmosphere.

IL assigned research subjects to each of us. Lev and Boris started to study reactions of PFIB and hexafluoropropene, which involved rather ordinary organic synthesis, the subject of our diplomas at Moscow State University. (My thesis, published in the Zurnal Obshchey Khimii [1] was my first publication.) As to myself, IL assigned me to study unfamiliar telomerization of trifluorochlorethylene and the individual telomerhomologs reactivity. Lev and Boris quickly made good progress and even published several articles, but I bogged down in my subject for 2 years with no results (you can imagine the equipment we used at that time for telomerhomologs separation). I was young, inexperienced, could not disobey the Academician, and had no idea how to get my assignment changed. However, everything changed when I realized a quirk in IL's personality. He could not resist a show of initiative. So I would bootleg experiments without saying a word to him. Then when one of my surreptitious experiments was successful I would say, ''IL, look at the interesting result I got in this reaction.'' The reaction of PFIB with water just happened to be such an experiment [2]. The subject of my research was immediately changed and IL never again mentioned telomerization or polymerization to me. Incidentally, I have avoided polymers ever since. I learned that I can, and should, choose my own research subjects. Since then, no one has been able to channel my work in one direction for too long, because any experiment answering the initial question always brings up new questions requiring additional experiments.

In our conversations IL emphasized chemical reactions or processes that could have practical or theoretical value, commented on interesting behaviour of some materials, and advised us how to make them useful. IL was an erudite person, and he clearly saw the future of fluorine chemistry. When we started, very little was known about fluorolefins, perfluoroketones, polyfluorinated acids and other fluorinated compounds. Knunyants led us into this new field of chemistry, plotting the path to continue our research. With the passing years new scientists joined our laboratory and it expanded. In 1993 we relocated to the main institute building on Vavilova Street and started to study reactions of other fluorinated olefins. Along with Knunyants, Lev German and Boris Dyatkin were pioneers of this field. They have contributed greatly by their wide-ranging research into the reactions of fluorinated olefins with nucleophiles, and toward understanding the difference between fluorolefins and the hydrocarbon analogs [3] (see also review [4]). Two significant results of their research were the synthesis of the anticancer drug 5-fluorouracil [5] and alfa-fluoroacrylates, used in the production of temperature-resistant organic glasses. The windshields for the first ultrasonic airplanes was made of these special polymeric materials.

Knunyants pioneered the research of electrophilic reactions of fluorolefins [6,7] and also the chemistry of mesomeric carbanions [8]. Another IL achievement is the discovery and the development of chemistry fluorinated sultones [9], important starting materials for producing fluorinated membranes. The synthesis of fluorosultones was achieved under IL leadership by G.A. Sokolski in the Academy of Chemical Defense. At that time it was close cooperation between the chemists of the Military Academy and our lab in INEOS. We were often invited to (MASD) for joint seminars. In fact, I defended my PhD dissertation on fluorinated beta-lactones [10] at the Military Academy.

Another practical accomplishment was the creation of an oxygen-carrying blood substitutes. It was called ''Blue Blood'' [11,12]. At the beginning of 1970 professor Grigory Yakovlevich Rozenberg from the Central Institute of Blood Transfusion came to our lab for consultation about inert perfluorinated liquids, which were used in America as blood substitutes during experiments with animals. Professor Rozenberg sponsored the work on artificial blood substitutes in our group in 1970. The group consisted of four people: Lev Gervits, Kirill Makarov, Tatjana Abroskina and myself, as leader of the group and Knunyants' deputy for this problem in the AN USSR. After I left INEOS in 1975, this work was successfully accomplished by Gervits and Makarov, in cooperation with scientists from the Institute of Theoretical and Experimental Biophysics, Russian Academy of Sciences in Pushchino City (Moscow Region) E.I. Maevsky and G.R. Ivanitsky and also people from other organizations. They were awarded the prize of the Russian Counsel of Ministers for development of inert oxygen-carrying blood substitute called ''Perftoran''.

After 5 years of work on blood substitutes I left the INEOS Knunyants's laboratory for a completely personal reason, which had to do with the security clearance required on this job. Every person involved in this research had to have the so-called second degree of access. In USSR, having such security clearance could result in extra restrictions of international travel (even without such complications international travel was severely restricted for citizens of USSR). By the time, my family and I started already to contemplate emigration, to follow a few our close relatives who managed to make it and started successful careers in the United States. In addition, I considered security requirements surrounding access to the so-called classified materials to be ultimately idiotic. For example, articles written by American pioneers in the field of fluorinated blood substitutes, such as Leland Clark, Henry Sloviter, Robert Geyer and others, were published in ''Federation Proceedings'', a periodical freely available at many university libraries in the U.S. We, however, received copies of these articles secretly from the Presidium of Academy of Sciences, and had to read them in a special room in INEOS controlled by the so-called First Department (which was really a branch office of KGB; in

the USSR, such departments existed in every organization of slightest significance). We had to sign in and out of the room indicating what we have been reading and how much time we have spent there, and to fill all kind of paperwork that afterwards could be used against us later when our request to let us emigrate would be considered.

Sometimes security clearance was to be used for less obvious reasons. INEOS did not stock an adequate supply of chemicals immediately required for our work. For that reason IL one day sent me to the chemical plant in the Kirov's region to obtain some hexafluoropropene needed for our blood substitute work. This plant, in a small city, was considered a government secret. I spent 2 days and one night there. I was told later, that I was being spied upon and checked out every 15 min during my presence there by people who were in charge of the security at the plant. I believe that my participation in the blood substitute program, and that visit to the secret plant were the main reasons that my family was repeatedly denied permission to emigrate from the Soviet Union for 12 long years after we applied for emigration, before finally, on the wave of perestroika, we were granted permission to leave the country.

3. I.L. Knunyants as the lab leader

IL was a democratic and accessible boss, a rarity at that time in the USSR. He was a naturally kind person, who loved his associates. His favorite was N.P. (Tanya) Gambaryan, a brilliant research chemist [13]. By the way she was a supervisor PhD work of Quing-Yun Chen, who now is one of the leading fluorine chemists in China. Tanya Gambaryan had a somewhat unique personality. She was often stopped at subway station because she walked all over town barefoot. Once, cleaning the lab, she collected remnants of sodium metal and discarded them by tossed into the Yausa River two or three miles away from downtown. The resulting bubbles of exploding hydrogen made loud popping noises that sounded like gunshots, which continued for a long time around there. That really unnerved the authorities, since it was the eve of the anniversary of the October Revolution, when security was on high alert for possible terrorist activity by enemies of the Soviet Union.

I earlier mentioned the joined seminars involving our lab and the chemists of the Military Academy. The Academy had a large campus in Moscow surrounded by not so high stonewalls with security towers and armed guards. To enter the campus one had to go through the armed gates, and then sign in and obtain a pass to proceed to the laboratory buildings of General Knunyants. Once in a while, to save time, Tanya Gambaryan would climb over the wall instead of going through the gate. When IL found out about this, he was furious, ''Tanya the guard could have shot you dead and not have been punished—might even have been promoted!'' He liked Tanya and worried about her. In working with IL more than 20 years, I do not recall a single incident where he punished anyone by demotion, suspension, or salary reduction. He would become angry and shout at us, but that was it, although there were many occasions (see below) that other leaders would mete out severe punishment in similar cases.

Knunyants always considered his coworkers as friends; he really enjoyed debating and wagering with his friends. We often placed bets on the outcome of a chemical reaction. The loser had to pay off with a bottle of (no, not vodka), but Armenian cognac. I recall a debate he once had with Tanya Gambaryan. He insisted that metallic sodium would not react with 100% sulfuric acid (concentrated H_2SO_4 + 4% SO_3) because acid was not dissociated. Apparently he was wrong, because there was a violent explosion, followed by tedious time of cleanup work to neutralize the spill, and to wash off all the sulfuric acid and its salts from the hood. Sometimes IL stayed after work to play chess with someone or other, often with Igor Rozhkov. Although IL was very busy person, dividing his time between the Academy of Sciences and the Military Academy, he loved to participate in the celebration of birthdays, anniversaries, lab picnics in the forest near small village ''Sosenky'' 17 miles West of Moscow, dissertation presentations, etc. I will never forget his kindly arrival at the ''Budapest'' restaurant for the celebration of my wife's successful defense of her dissertation. He strode into the hall with a big smile, dressed in his full general's military uniform. We had not expected him to be there, and were very surprised and pleased that he was with us! IL made us all feels right at home in the lab. Due to our intense interest in the work, our passion in search of new reactions, and the interesting discussions in the colloquiums, we put in very long hours in the lab, often late into the night. In fact, the middle of the night was an ideal time to ascuire NMR spectra, because the traffic of electric trams stopped on Vavilova Street at 1 a.m. and NMR spectra of better quality could be recorded. In view of this, Erlen Fedin the manager of INEOS NMR spectroscopy group granted me a permission to use NMR spectrometer at night. So I often worked until morning and to the consternation of my family, absent-mindedly neglecting to alert them of the fact now and then.

4. Contacts with foreign scientists

With the Iron Curtain firmly in place at that time, few Soviet citizens could travel abroad, for business or pleasure. Communication with the outside world and contact with foreign scientists was tightly controlled. In 1965, by pulling rank, Knunyants obtained authorization for his group to attend the Third International Fluorine Symposium in Munich. Then, 2 years later, in 1967, Knunyants, Alexander Vasilievich Fokin, and myself presented papers at the Fourth Fluorine Symposium in Estes Park, Colorado, USA. It was a miracle that we arrived in Colorado at all, since we knew basically nothing about the American transportation system, and spoke practically no English. We were handed our plane tickets to New York at the Presidium of the Academy of Sciences only a few hours before departure. In New York City we spent the night in a hotel, and then flew to Denver in the morning. In Denver we boarded a Greyhound bus to Estes Park. There was a considerable interest in our presentations at the Symposium. Our American colleagues graciously spent their free time showing us around. My chaperone to Rocky Mountain National Park was Alan Lovelace and other American scientists were also accompanied

Knunyants and Fokin. In 1969 the car with power steering was a novelty and I do recall Alan proudly demonstrated me how he could easily turn the steering wheel while the car was standing still. However, our American trip was not without incident. One morning IL was in panic; he said he had lost his passport. Considering the times, for a Russian traveling in the USA, his fear was understandable, even for a man of his high rank. Alexander Fokin tried to calm him down, and finally we did find the passport in his room.[1]

IL encouraged cooperation with the outside world, facilitating an exchange of scientists between our lab and the famous scientific centers abroad. In 1968 I was sent on sabbatical to work with Professor W.K.R. Musgrave at Durham University. At the same time, Igor Rozhkov went to Manchester University. During those important 6-months at Durham, I acquired friends abroad, such as Doctor R.D. Chambers, who was then a senior scientist like me (Dick Chambers now is a Fellow Royal Society). In that half-year at Durham we published three articles [14]. My friendship and cooperation with Dr. Chambers endured, even after I started my work at 3M Co. Also it was in England that I first realized what it meant to be out from behind the Iron Curtain, and how isolated Soviet scientists were from the rest of the world. In Great Britain, neither the government, nor the police could accuse a person without reason. It gave me a new and previously unknown feeling of security. Just before leaving for England I had been instructed at the Presidium of the Academy of Sciences on how I'm supposed to behave abroad and at the end of the instruction session I was told to sign a blank piece of paper at the bottom line. Can you imagine that? Certainly life in England was indeed very much different from life in Moscow!

In 1969 5th Fluorine Chemistry Symposium took place in Moscow by initiative of Knunyants, who appointed Academician Nikolai Nikolaevich Vorozhtsov as his deputy and me as Scientific Secretary of the Symposium. Many American scientists from industry, academia, and even military, participated in this quite successful Symposium. There were a lot of foreign scientists attending (for instance, Aerojet-General Corp, Azusa, CA; Air Force, New York, NJ; Argonne National Lab, Argonne, IL and others) it attracted considerable attention from the KGB and GRU (Military Intelligence). Agents of these organizations flooded the site of the Symposium held in the Moscow University. These people could be easily recognized by their clothes: black jackets and neckties. They looked exactly like the members of the Dutch secret police who retrieved the Princess in the romantic comedy film ''Roman Holiday'' with Gregory Peck and Audrey Hepburn.

Early in 1966, Professor Paul Tarrant (University of Florida) wrote to IL, soliciting articles for the first volume of a new series, ''Fluorine Chemistry Reviews''. This publication was to devote to the latest achievements in fluorine chemistry, authored by significant internationally known contributors to their special field of the chemistry. Knunyants volunteered me to write an article about fluorinated ketenes, which was published in the first volume [15]. Actually, the review was based on my Doctor of Science degree dissertation. Professor Tarrant sent us personal copies of Fluorine Chemistry Reviews (volume 1, issue No 1) signed by the editors and members of the editorial board. I treasure this copy as a memory of Knunyants, Voroshzov, Richardson, Glemser, Park and others.

5. I.L. Knunyants as a "dissident"

Knunyants was a high ranking officer in the USSR Ministry of Defense, a three-times Stalin prizewinner, a Lenin prizewinner, a Hero of Social Labor, a general, and a full member of the Academy of Sciences. A person of such rank certainly belonged to the Soviet Elite and enjoyed many benefits: special food packages, a chauffeured car, a large high-rise apartment on the Kotelnicheskaya embankment, overlooking the Moscow River and the Kremlin.[2] He used to invite us to his apartment. IL was very proud of his collection of paintings by the old masters. Some of the paintings he had restored himself—it was his hobby!

While giving the Soviet System its due, in many cases IL had his own opinion. But he was quite a shrewd person, and never gave anyone grounds to prosecute him. Only within the small circle of his trusted colleagues did he speak openly. And none of us ever betrayed his trust. I would like to remind that in the 1960s and 1970s, Soviet citizens and science in the USSR were under tight government control. Any attempt to oppose the Communist Party establishment was unacceptable and even dangerous.

Here is an example to illustrate how Knunyants operated independently. One day he called me into his office, locked the door, and said ''Yuri, I hear that you have expressed some negative opinions about the Communist Party. My sources are unimportant, and I will not reveal them to you. In your

[1] The other and a funny incident happened in New York on the way home. We were going to spend a little money, which we had obtained in the Academy of Sciences, and stopped into a huge department store. Alexander Vasilevich picked out a coat for his wife, but was not sure of the size. He spotted a woman who looked to be about his wife's size, and in broken English he asked if she would help him by trying on the coat so he could see if it fit her. To our surprise she replied in Russian ''Yes, I'd be glad to help you.'' She turned out to be a childhood friend of mine, Natasha Ogorodnikova who was working in New York at the UN.

[2] And speaking of elite people getting special treatment by the authorities and even law enforcement officials, I will conclude by describing an incident that happened with IL. He was driving his car to take a small refrigerator to a repair shop. The handyman of our lab, Yasha Neroslavsky, who helped him, told us this story. ''Driving his ''Pobeda'' automobile along Lenin Prospect, IL ran a red light at the Gagarin Square. As the policeman was approaching IL stopped the car. The car ''Moskvich'' behind us also stopped. In Moscow, the traffic violation protocol was well known. Either you pay the fine on the spot and do not ask for a receipt, or you get hauled in to the police station, detained for several hours, pay the fine anyway, and probably lose your license. We could see the menacing expression on the face of the approaching policeman. 'Yasha, give me my service cap', IL said. (He always carried his military service cap on the back window shelf). IL put on his General's cap with the scrambled eggs on the visor and we watched the policeman's facial expression change from angry to friendly. He smilingly leaned into the car opened window and said 'Please drive carefully, comrade general. You could kill yourself'. Then he stalked back to the ''Moskvich'' parked behind us. I am sure – added Yasha – the unlucky driver of that car got slapped with a double fine for this traffic violation''.

conversations, you can criticize the Party and the Government, but always finish on a positive note, saying something like: 'It is just a small problem, but in general we live in a great country' or 'We have a lot to be proud of.' If you do that, I can defend you; otherwise I won't be able to protect you.'' This was a very useful suggestion, or perhaps a caution, that I have remembered ever since.

In the pervasive Communistic atmosphere of the Soviet Union of the 1940s through the 1960s some absolutely ridiculous theories of Biology, proposed by an Academician Lysenko, were very popular. The Party enthusiastically supported these pseudo-theories because they were in close agreement with the basic Communist philosophy. Several scientists were actually prosecuted for disagreeing. In 1963 Zhores Medvedev sent his famous manuscript ''Biological Science and the Personality Cult'' to some Academicians, including Knunyants.

This controversial manuscript eventually was illegally published in Samizdat in 1966. I.L. Knunyants was not afraid to share the manuscript with us. Medvedev discussed the destruction of Biological science in 1948–1950 in the Soviet Union and its consequences for the country. This article was landed like a bombshell, because prior to its publication, no one dared to criticize the Party Line, as approved personally by Josef Stalin. While Zhores was put into ''psikhushka'' (a special hospital-prison for dissidents as if they are nuts) IL was among those, who got Zh. Medvedev out of trouble and helped him to leave USSR for Great Britain. IL himself was very critical of Lysenko's ridiculous theories and the presentation he gave at an Agricultural Academy Session in 1948 ''About the Situation in Biological Science''. Another example of IL's independency was his outspoken opinion of a well-known scientist and human rights fighter, Andrey Sakharov and his essay ''Progress, Coexistence and Intellectual Freedom'', which was published abroad. Knunyants said that all governments should genuflect to Sakharov. He was not afraid to say so, even at a time when the Party was organizing campaigns to denounce Sakharov. One should note that even the Director of the Institute, Academician A.N. Nesmeyanov, held Sakharov in disdain. I would like to describe these events in more detail, since now, in 2007, when Russia has changed it is hard to imagine that such incidents actually took place.

In summer of 1974 all INEOS managers, starting from Doctors of Science and higher, were called into the director's conference room. When Lev German, Boris Dyatkin, and I arrived, we learned the subject of the meeting. Nesmeyanov wanted all of us to sign a letter denouncing Sakharov. I was sitting at a small separate table with Boris Englin, and we did not want to sign. Boris said he would just walk out. That was easy for him; he already had a permission to immigrate to Australia. As to me, such a demonstrative action means being immediate fired. Nesmeyanov slowly started reading his letter to ''Pravda''—the main Communist Party newspaper. Fortunately, we were rescued from our predicament in the knick of time. Academician Martin Kabachnick suggested some changes, and Boris Dyatkin and Leonid Zakharkin supported him. Nesmeyanov agreed, and adjourned the meeting for 30 min to make the corrections. Everyone returned, except Englin and myself. For the record, I unexpectedly was called away on an urgent matter concerning the blood substitute program. The letter never appeared in ''Pravda'' newspaper; therefore, our disappearing act had no consequences. Mark Efimovich Volpin was Nesmeyanov's deputy at the time. In 1993 he was visiting 3M Co. and made a presentation about Chemistry at INEOS. He was our family guest. Time and democratic America made us closer to each other. We viewed the past through rose-colored glasses. We felt like old friends and shared endless memories. He was surprised to find out how Englin and I had managed to sneak out of that nasty meeting.

In summer of 1968 the Soviet Army intervened in Czechoslovakia and shut down Prague's revolution. In INEOS as in other organizations, a meeting was convened to support this action. I did not attend as I was on vacation. Upon returning, I was told that at the end of the meeting Tanya Gambaryan required a voting. The meeting chairman—INEOS Communist Party Lieder Savely Ioffe agreed and asked for a show of hands as to which supports this action, who is against it, and who abstains. To the great surprise and indignation of the organizers, six members of Knunyants' lab, T. Gambaryan, N. Kasmina, E. Rohlin, Yu. Zeifman, Yu. Aronov and G. Krasnikova (Knunyants's stepdaughter) raised their hands to abstain. IL defended his employees to the hilt, but Yu. Aronov was fired anyway, despite IL's valiant efforts.

6. Conclusion

Ivan Ludvigovich Knunyants was ravaged by Alzheimer's disease in his final years, and passed away in 1990. He is interred in Moscow's Novodevichje cemetery. As a General of Soviet Army, he was buried with full military honors, with an elaborate funeral conducted by the staff of the Military Academy. A Guard of Honor was posted at attention on a pedestal in front of the casket. Several of his former students, including General Alexander Vasilevich Fokin, gave eulogies. The event concluded with a rifle salute. While leaving the cemetery I reflected on how lucky I was to have met such a great scientist, wonderful leader, and kind human being in my lifetime. I thought about how I had worked in the best chemical Institute of the Academy of Sciences. In my heart I consider it my ''alma mater,'' where I spent the best years of my research life. Just 2 weeks after the funeral, my wife and I made our permanent move to the United States of America to start our second, completely different, life.

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