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Review

Chromatography in Ukraine: Development and Achievements

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Abstract: The development of chromatographic science in Ukraine is due to the widespread use of chromatography to solve practical problems. The centers of chromatographic research in Ukraine are in large cities: Kyiv, Kharkiv, Lviv, Odessa, and others. Along with the development of chromatographic research methods, analysis methods for control of food and agricultural raw materials, medicinal products, petroleum products, and determination of the environmental pollution state are created. Chromatography is used in medicine, pharmacology, biology, and other sciences.

Keywords: history of chromatography; chromatographic method development; methods of analysis

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Chromatographic research in Ukraine is developing quite rapidly due to the wide range of its practical use. The history of chromatography in Ukraine is closely related to Mykhailo Tsvet, whose father was Ukrainian (born in Chernihiv), and his mother Italian [1]. Tsvet performed his chromatographic research at the University of Warsaw from 1902 to 1903. He first reported the method of chromatography in March 1903 at a meeting of the botanical branch of the Warsaw Society of Naturalists in a report on "On a new category of adsorption phenomena and their application to biochemical analysis".

In 1938, Kharkov Izmailov N.A. and Schreiber M.S., for the first time, published work on circular thin layer chromatography and subsequently developed other variants of the method of thin-layer chromatography [2,3].

Research centers in the field of chromatography in Ukraine are located in Kyiv, Kharkiv, Lviv, Odessa, Dnipro, and other cities. At the Institute of Colloid Chemistry and Water Chemistry of the National Academy of Sciences of Ukraine in Kyiv, research and development of the method of ion chromatography are carried out. This method effectively determines ionic compounds and, in many cases, is normative for ecological research of natural waters and water quality control (particularly in energy) [4–10].

The works were carried out under the guidance of professors L. Loginova and A. Kulikov at the V. N. Karazin Kharkiv National University, the "Ukrainian Scientific Pharmacopoeial Center for Drug Quality" which is dedicated to studying high-performance liquid micellar chromatography [11]. L. Loginova and A. Kulikov proposed models for determining the retention time of compounds in micellar liquid chromatography, which allow optimization of the composition of the micellar mobile phase for better separation of anions [12–14]. The influence of reversed-phase sorbents on the separation efficiency [15–17] and organic phase modifiers—organic alcohols, acetonitrile, tetrahydrofuran, organic acids—were studied for separation efficiency [18]. Methods of quality control for medicines and substances have also been developed. Studies using micellar thin layer chromatography to analyze biologically active compounds, in particular, coumarin, licurazil, and glycyrrhizinic acid in plant medicinal raw materials, were carried out [19,20].

Methods of pharmaceutical analysis of drugs, medicinal raw materials, and food products are being developed in the O.B. Bogatsky Physical-Chemical Institute of National Academy of Sciences of Ukraine in Odessa. The studies use high-performance liquid and high-performance thin-layer chromatography [21,22].

Investigations of the content of biologically active substances and impurities in drugs and medicinal plant raw materials are carried out in the laboratories of liquid

chromatography-mass spectrometry of Zaporizhia State Medical University [23]. This laboratory develops methods for determining compounds that are markers of various diseases [24]. Studies of drug metabolism in living organisms are also conducted [25].

Chromatographic studies at the Department of Toxicological and Analytical Chemistry of Danylo Halytskyi Lviv National Medical University concerning the determination of toxins and residual amounts of drug substances and their metabolites in biological samples were performed by thin-layer chromatography (TLC) and gas chromatography (GC). In particular, Ph.D. Docent Bidnichenko Yuriy Ivanovych deals with the problem of toxins analysis from poisonous fungi [26]. The determination of fungal toxins was performed by TLC [27], disk electrophoresis [28], capillary electrophoresis [29], gel chromatography [30], and high-performance liquid chromatography (HPLC) [31].

Attention in scientific research of the Department of Analytical Chemistry of Kyiv National University is paid to studying sample preparation methods by solid-phase extraction. These studies were developed under the guidance of Prof. V.M. Zaitsev. The method of solid-phase extraction concentration of 2,4-dichlorophenoxyacetic acid, phenol, 1-naphthol, 2,4,6-trinitrophenol using silicon oxide from water and biological fluids is described in [32–34]. The topic of associate professor M.G. Zui was related to the determination of oxygen-containing organic compounds: phthalates, parabens, benzophenols, and oxygen-containing compounds [35,36].

Chromatographic studies of the purification of polyacrylamide gels for medical purposes, which were synthesized at the F.D, Ovcharenko Institute of Biocolloid Chemistry of the National Academy of Sciences of Ukraine, were conducted at the Institute of Environmental Geochemistry [37].

Gas and liquid chromatography were used at Lviv Polytechnic National University to study the kinetics of reactions at the departments of basic organic and petrochemical synthesis technology (now the Department of Organic Products), chemical technology of oil and gas refining, at the Department of Analytical Chemistry (analytical and general chemistry, organic chemistry) [38,39].

The scientific work of the zeolite-chromatographic group headed by Professor Onufriy Banach of the Danylo Halytskyi Lviv National Medical University was devoted to the study of using zeolites in gas chromatography [1,40]. For stationary zeolite phases for gas chromatography, the "low substitution effect" was found, which consisted of the fact that in zeolites, when sodium cations were replaced by other alkali metals, the parameters of gas retention in gas chromatography conditions changed sharply. In particular, for ethane, methane, and oxygen with a low degree of substitution (9.8%) of Na + cations on K + in zeolites, the retention time of these gases increased rapidly, which was also observed in cases of substitution of 6% Na + on Rb + and 5% Na + on Cs + [40,41]. The phenomenon of inversion of the chromatographic retention was also observed, which consisted in changing the sequence of the components of the mixtures from straight to inverted. For the phenomenon of chromatographic inversion, two types were identified: (1) inversion caused by exposure to one of the components in the studied gas sample; (2) inversion depending on the degree of substitution of cations in the zeolite [40,42]. Studies of the chromatographic properties of zeolites revealed the specific selectivity of silver-containing zeolites of type Y to carbon monoxide [40]. The study of the properties of modified zeolites made it possible to create effective methods for analyzing the content of oxides of carbon, nitrogen, and sulfur and hydrocarbon gases C1–C4 in atmospheric air. Zeolite stationary phases were used for gas adsorption (solid-phase) chromatography and extraction, concentration, and desorption for the quantitative chromatographic determination of these air pollutants [43–49].

Considerable attention in chromatographic research in Ukraine is paid to the state of environmental pollution by toxic substances. To this end, methods are being developed for environmental monitoring and detecting toxic substances, such as pesticides. This activity is the subject of attention of state bodies and is defined by law.

Chromatography plays a notable role in the chemical and analytical support of toxicology and hygiene of pesticides, occupational health, and the environment. In Ukraine

and the former Soviet Union, it was introduced in the early 1960s in the physicochemical laboratory of Kyiv Research Institute of Hygiene Labor and Occupational Diseases. It was headed in 1960–1964 by Marta Arkhipovna Klysenko, who, with the participation of specialists from the same laboratory, had previously developed the country's first methods for determining pesticides in the air of the working area (arsenic-containing insecticides—1954; phosphorus and organochlorine insecticides—1955–1956); in soil, food and biological environments (1962) [50]. The implementation of such developments was of great importance for the hygienic regulation of pesticides, addressing their use in agriculture, safety for the health of workers and the general population, and the environment. This laid the foundations of a new scientific field—analytical chemistry, namely "analytical chemistry of pesticides".

In the All-Union Research Institute of Hygiene and Toxicology of Pesticides, Polymers, and Plastics of the Ministry of Health of the USSR 1965–1989, under the guidance of Doctor of Biological Sciences, Professor M.A. Klysenko, special attention was paid to theoretical issues (study of the relationship between chromatographic behavior of pesticides of different chemical nature and their molecular structure, behavior in the environment and the body), application of chromatographic methods in basic research in toxicology, and biological exposure monitoring [51–55].

The organization and coordination of development, testing, and implementation of methods for chromatographic determination of pesticides in all union republics was carried out by the Group of Experts on Review and Recommendation of Methods for Determination of Pesticides and Biologicals in Food, Feed, and Environment at the State Chemical Commission for almost 30 years. During this time, the Ministry of Health of the USSR developed and approved more than 6000 guidelines for the determination of micro quantities of pesticides, which were issued as official guidelines in more than 20 collections of guidelines and 8 reference publications on the determination of pesticides' micro quantities in food, feed, and environment. Today, these recommendations are relevant in the field of pesticide safety control not only in Ukraine [56–63].

During the time of independent Ukraine, the analytical chemistry of pesticides has been developed intensively in connection with the introduction of the latest agricultural technologies. This involved using a wide range of chemical plant protection products, including "new molecules". To register pesticides in Ukraine, it is mandatory to develop methods for controlling air pollution in the working area and air, soil, and water as well target crops and the raw materials obtained from them (seeds, grains, fruits, roots, etc.). The methods are validated according to the requirements of international standards, issued in the form of guidelines. The issues are considered in the Ministry of Environment and Natural Resources of Ukraine by the Group of Experts on Methods for Determining Pesticides and Agrochemicals in Food, Agricultural Raw Materials, Feed and Environmental Facilities and in the Committee on Hygienic Regulation of the Ministry of Health of Ukraine by the Commission on methods of control of dangerous factors. The State Service of Ukraine for Food Safety and Consumer Protection approves standards as official by the Ministry of Environmental Protection and Natural Resources of Ukraine. Over the last 30 years, almost 1800 guidelines have been developed and published in the official editions of the collections mentioned above, which reach almost 110.

Particular attention is paid to the development of selective methods for analyzing pesticides in the presence of their toxicologically significant metabolites and transformation products in the environment and other chemical pollutants [64–66].

To develop methods for determining pesticide residues, analytical laboratories must be accredited by the National Accreditation Agency of Ukraine according to ISO/IEC 17025, confirming competence in interlaboratory tests at the national and international levels. The main institutions/developers accredited for this type of activity in the Committee on Hygienic Regulation of the Ministry of Health of Ukraine are:

- L.I. Medved's Research Center of Preventive Toxicology, Food and Chemical Safety (Ukraine, Kyiv);

- Hygiene and ecology institute of Bogomolets National Medical University (Ukraine, Kyiv);
- Y.I. Kundievs Institute of Occupational Medicine of the National Academy of Medical Sciences of Ukraine (Ukraine, Kyiv).

Chromatographic laboratories of the State Research Control Institute of Veterinary Drugs and Feed Additives, Lviv, are developing and implementing methods of control of veterinary drugs and feed additives. The results of their research were printed in the professional publication "Scientific and Technical Bulletin of the State Research Control Institute of Veterinary Drugs and Feed Additives and the Institute of Animal Biology" (https://scivp-journal.com.ua/index.php/journal, accessed on 2 May 2022).

The institutes of the National Academy of Sciences are engaged in developing chromatographic methods of analysis and monitoring the state of environmental pollution. The A.V. Dumansky Institute of Colloid Chemistry and Water Chemistry developed a methodology for the study of organochlorine pesticides (OCP), polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs) in surface and drinking waters, bottom sediments, and aquatic species of living organisms of the bottom layer from different water systems of Ukraine. The study of persistent organic contaminants (POPs) includes the following steps: extraction, concentration, identification, and determination of these compounds using adequate methods of sample preparation, and modern chromatographic and gas chromatography/mass spectrometric methods. These methods include gas chromatography/mass spectrometry with mass detection selectivity by total ion current and selective ion control (SCAN, SIM modes), gas chromatography with detection of electron capture, and high-performance liquid chromatography with detection of fluorescence.

The content of OCP, PCBs, and PAHs was monitored in surface and drinking waters, bottom sediments, and aquatic species of living organisms of the bottom layer from different water systems of Ukraine. The concentrations of these compounds at monitoring points were determined, and their bioavailability and the degree of bioaccumulation were determined. Systematic data on identifying OCPs and PCBs and their levels in various tissues of aquatic organisms are very limited. To assess the threat to aquatic ecosystems and humans posed by OCPs and PCBs, they were extracted from muscle and other fish tissues by liquid extraction, and reliable results were obtained on the content of these compounds. To assess the state of the environment, the results of monitoring the OCP, PCBs, and PAHs in these areas were summarized [67–70].

The laboratory of complex geochemical research of the Institute of Environmental Geochemistry of the National Academy of Sciences of Ukraine is engaged in monitoring objects of the environment by chromatographic methods and determination of the level of ecological safety. Extensive research has been conducted near the city of Kalush. Large enterprises in the petrochemical industry were located in this city. One such enterprise was the production association "Chlorvinyl", whereby direct chlorination of industrial hydrocarbons produced perchlorethylene, carbon tetrachloride. One of the by-products formed was hexachlorobenzene. Its content in waste reached over 90%. As there were no technologies and facilities for high-temperature incineration of this waste in the Soviet Union at that time, it was decided to bury the waste containing hexachlorobenol in iron barrels at a landfill near the city. Large quantities of this product accumulated. Over time, the barrels corroded, and hexachlorobenzene began to enter the environment, contaminating soils, groundwater, and surface water. Concentrations exceeded permissible levels by thousands of times. In this regard, the area has declared a state of emergency, and the question arose about the removal of hexachlorobenzene for its disposal by high-temperature incineration. For hexachlorobenzene, the laboratory of complex geochemical research determined the disposal of waste, monitored landfills, and drew up appropriate maps, according to which hexachlorobenzene was exported from these areas [71,72].

Together with scientists from Romania, led by Dr. Virginia Coman and Dr. Simion Beldean, the Laboratory of Complex Geochemical Research, under the NATO Science for Peace Program Project, monitored the pollution of the Tisza River [73]. The study of the content of toxic substances and the identification of sources of pollution is essential given

that the river flows through the territory of European countries, and pollution by toxic substances in one of them may pose an environmental hazard to another country. Software has been developed to predict the time of pollutant propagation along the riverbed and its concentration along the river in case of emergencies [74].

Chromatographic studies of subsoil air for the content of light fractions of hydrocarbons and helium are used to search for minerals, oil, and gas by scientists of the Institute of Geological Sciences of the National Academy of Sciences Ukraine [75].

The Chromatographic Society is engaged in the development of chromatographic research in Ukraine, establishing contacts and cooperation between scientists and specialists working in chromatography. The origins of the Chromatographic Society of Ukraine can be attributed to the All-Union Association of Chromatographers named after M.S. Tsvet, organized by Karl Sakodinsky. Zinaida Klymak coordinated the activities of the Association in Ukraine. With the collapse of the Soviet Union and the formation of Ukraine as an independent state, the question arose about the organization of the Chromatographic Society of Ukraine. Modest Gertsiuk was elected president of the Chromatographic Society at the founding congresses (Figure 1).

Figure 1. Modest Gertsiuk.

During its activity, the Society organized international conferences "Methods of chemical analysis" and "Chemical safety: problems and solutions," which discussed all aspects of the development and practical application of Separation Sciences. The scientific journal "Zurnal Hromatograficnogo tovaristva" (Journal of Chromatographic Society) was founded, publishing articles by domestic and foreign authors on the development of chromatography and separation sciences (Figure 2).

The journal publishes articles on the activities of research centers, laboratories, and groups working in this field, and scientists who contribute to the development of chromatographic research. The Chromatographic Society is a member of the Central European Group for Separation Sciences (CEGSS) and the European Society for Separation Sciences (EuSSS). The participation of the Chromatographic Society in the activities of these organizations contributes to the expansion of contacts between Ukrainian scientists/chromatographers and their foreign colleagues and the development of chromatography in Ukraine.

Figure 2. Modest Gertsiuk during chromatographic conference in Kyiv (2015).

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