

POLISH ANALYTICAL CHEMISTRY IN THE PERIOD 1944-1974*

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ABSTRACT

Some general comments concerning analytical chemistry and its role in modern science, industry and life are made. The activities of the Commission of Analytical Chemistry of the Polish Academy of Sciences are then briefly described. Finally the main research directions of analytical chemistry in Poland in the last 30 years are outlined.

Analytical chemistry according to the modern meaning is an applied science dealing with the elaboration and formulation of laws, criteria and methods which allow one to determine, with estimated precision and accuracy, the qualitative and quantitative composition of material subjects.

The work being done in analytical chemistry could be divided into three main groups.

The first group contains the basic research done in each area of analytical chemistry. The main task of this research is to check to what extent the laws formulated by physics and chemistry, usually for simplified model systems, describe the complicated, real systems existing in analytical samples. This enables us to introduce corrections if possible and necessary. There are also being investigated and formulated new general dependencies, which make it possible to foresee the course of analytical processes.

The second group contains general analytical problems such as investigation and formulation of criteria for analytical methods characterization, the problems of optimization of methods and of analytical control systems, statistical evaluation of analytical results, the problems of production and testing of standards, etc. This group could be called the metrology of chemical composition of matter.

The third group deals with the elaboration of analytical methods for the determination of the chemical composition of particular material samples, and elaboration of complex systems of analytical 'on-line' control of particular technological processes.

The IV Polish Conference of Analytical Chemistry is being convened in the year of the XXX Anniversary of the Polish People's Republic. This leads

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to some thoughts concerning the output of Polish analytical chemistry during the period. These considerations are the more reasonable because this period of 30 years is also very important in the historical development of analytical chemistry as a branch of science. During this period a magnificent development has taken place. Chemist-analysts have developed many new chemical methods. They have used many physical methods, and exploited the progress made in electronics, automation and computers. They have taken analytical chemistry out of the epoch of chemical analytical laboratories in which chemical-only methods of qualitative and quantitative analysis, gravimetric, volumetric and elementary methods, have prevailed. The limits of detection and determination have been very much enlarged in the direction of trace amounts of components. New possibilities of identification and determination of components in complex natural and artificial mixtures have been discovered. Chemical analysis entered the epoch of automation as well in an analytical laboratory as directly on technological lines. Chemical analysis is now the basis of quality control in industry; of modern medical diagnostics; of the health safety of mankind; and also of all investigations connected with the environment.

The comprehensive application of analytical chemistry methods, mentioned above, leads to the conclusion that the state of the art of analytical chemistry in the country is one of the important parameters which describe the level of the general development of this country.

The beginning of the formation of modern analytical chemistry in Poland was disrupted by the 1939-45 war. All the existing analytical laboratories were destroyed. This caused at least 15 years' delay in further development.

It is, however, to be stated that modernization had begun before the war in the 1930s, and many works done at that time were of modern scientific level. One can mention, for example, the work done on chemical analysis by Miłobędzki, on technical analysis by Struszyński, on organic elementary analysis by Bobrański and Sucharda, on spot-test inorganic analysis by Stalony-Dobrzański and on potentiometric titration by Drewski. Investigations in polarography and emission spectroscopy were started then by Kemula and Michalski. In spite of the limited funds which were at the disposal of these people, they have educated new chemist-analysts, who after the war began to create anew Polish analytical chemistry.

One can divide the 30 years of Polish analytical chemistry in the Polish People's Republic into two periods. The border between them is the year 1955—the year of the creation of the Commission of Analytical Chemistry of the Polish Academy of Sciences, headed from the very beginning by Professor Wiktor Kemula.

The first period—before 1955—was devoted to the reconstruction of industrial analytical laboratories and of analytical laboratories in universities and polytechnic institutes. Many new analytical centres were created in research institutes of the Polish Academy of Sciences and of industry. It was also a period during which there grew among Polish chemist-analysts an awareness of the significance of analytical chemistry as an instrument for controlling the quality of the production of Polish industry, until that time in a state of reconstruction and development.

At this same time the necessity of close collaboration among analytical laboratories of industry, universities and research institutes was recognized. The I Polish Conference on Analytical Chemistry was organized in 1951, and people began to organize seminars on certain analytical methods.

The creation in 1955 of the Commission of Analytical Chemistry, which began to publish in 1956 the Polish scientific journal *Chemia Analityczna* (*Analytical Chemistry*), is the starting point of organized activity in the field of analytical chemistry in this country.

The main tasks formulated by the Commission are the following:

Organization and activation of the collaboration of analytical chemists by the creation of proper Subcommissions dealing with particular methods or other problems.

Arranging exchanges of experience in seminars and symposia.

Influencing the scientific and industrial community of the country at large, to reach a better understanding of the role of analytical chemistry in science, the national economy and other fields.

Permanent training of analytical chemist in modern analytical methods by training courses at various levels.

Representing Polish analytical chemistry in scientific world organizations connected with analytical chemistry, organizing collaboration with foreign organizations of analytical chemists, and organizing Polish Conferences on Analytical Chemistry with participation of foreign scientists.

During its almost 20 years of existence the Commission of Analytical Chemistry has grown considerably. It is now acting through six methodical Subcommissions (Subcommissions of: Analytical Atomic Spectroscopy, Analytical Molecular Spectroscopy, Polarographic Analysis, Chromatographic Analysis, Application of Mathematical Methods in Chemical Analysis, of Analytical Reactions and Reagents), and through nine Subcommissions devoted to various branches of industry and other analytical problems (Subcommissions of: Teaching in Analytical Chemistry, Analysis of Metals, Analysis of Gases, Analysis of Standards, Analysis of Fats, Analysis of Polymers, Analysis of Petrochemical Products, Analysis of Pharmaceuticals and Analysis of High Purity Materials). The Commission has organized during this period of 30 years three Polish Conferences on Analytical Chemistry and 40 Symposia on various methods with the participation of over 8000 analytical chemists. Two hundred and forty-seven seminars on various analytical problems were organized with the participation of over 15000 people. Finally, 63 training courses were organized with over 2600 participants. In the journal *Chemia Analityczna* 2247 scientific papers, 179 notes for analysts and 77 review papers have been published. The size of *Chemia Analityczna* grew from 21 printed sheets in 1956 to 80 printed sheets in 1973.

It is quite certain that the Commission, using the willing collaboration of a large community of very active and devoted analytical chemists, closes the period with a favourable balance. It begins the twentieth year of its activity as an organization with good traditions, and aware of its main goal, which is the further, fast development of analytical chemistry in this country.

Considering the publications in *Chemia Analityczna* in the last 10 years

and comparing them with papers published in the preceding 20 years, one notes the drop in number of papers concerning purely chemical methods and the distinct increase in number of publications devoted to instrumental methods of analysis, mainly electrochemical, spectral and chromatographic. This corresponds with the general trend all over the world towards the instrumentation of chemical analysis.

The development of analytical methods, including instrumentation, is frequently based on the development of chemical research. Quite a lot of research has been carried out in this area in Poland, primarily concerning complex compounds, mainly with organic reagents which are useful in analytical chemistry¹⁻¹⁶. There are being investigated and elaborated new colour reactions for inorganic^{11, 12, 14, 16, 17} and organic^{3, 14, 17-24} analysis.

Among chemical analytical methods most work has been done on complexometric titrations mainly using complexones^{25, 26}. The other titration methods, in aqueous solution²⁷ as well as in non-aqueous²⁸⁻³⁰ media, have been investigated. New indicators are being investigated³¹⁻³². A new volumetric method called thiomercurimetry³³ has been developed.

Kinetic and catalytic methods are not very widely used. The method based on the reaction of sodium azide with iodine for the determination of sulphides and many sulphur-containing organic compounds³⁴ has received attention. Methods for determining trace amounts of some elements, based on their ability to catalyse the selected reactions^{35, 36}, have also been developed.

The investigations in organic elementary analysis deal with new fast combustion procedures and with the construction of proper apparatus^{37, 38}.

With regard to the group of atomic spectroscopy methods, difficulties in supply of proper apparatus caused some delay in analytical research, especially in x-ray fluorescence, atomic absorption and fluorescence and the microprobe method. Nevertheless these methods are already used in industry for current analytical control. The research was done mainly in emission spectrography and concerned, e.g. the mechanism of action of spectrographic carriers^{39, 40}. New methods for trace analysis⁴¹ and new spectrographic techniques⁴² were elaborated. One works with hollow cathode⁴³, induction plasma⁴⁴ as excitation sources.

There are also papers dealing with x-ray fluorescence⁴⁵, flame spectrophotometry⁴⁶ and atomic absorption⁴⁷.

Methods of absorption spectrophotometry also reached a high level during the last 15 years as a result of the analytical research done in u.v., v.i.s.⁴⁸⁻⁵³ and i.r.^{54, 55}, with reference to the analytical applications of absorption spectrophotometry in u.v., v.i.s.^{3-5, 9, 10, 13, 14, 16, 17, 19, 20, 22-24, 56-63} and i.r.⁶⁴⁻⁶⁷. An interesting apparatus called spectromonitor was constructed⁶⁸. It is a fast scanning absorption spectrophotometer, working in a slightly limited u.v. and v.i.s. region, with kinescope registration. Research in spectrofluorimetry^{15, 69} is also proceeding.

Raman spectrometry⁷⁰, as well as mass spectrometry of organic components⁷¹, are also the subject of research. The double focused spark mass spectrometer is being used for industrial purposes only. Occasionally e.p.r.⁷² and n.m.r.⁶⁷ methods are used for analytical purposes.

The main achievements of Polish analytical chemistry during the 30 years period under discussion are in the area of electroanalytical methods. One

has in mind the work of Professor W. Kemula and his research teams on chromatopolarography⁷³ and chronovoltammetry with a hanging mercury drop electrode^{74,75}. Chromatopolarography is an excellent method for the separation and determination of many mixtures of organic compounds. Chronovoltammetry is widely used for trace analysis. The polarographic methods—classical and modified—are being investigated from the point of view of basic research⁷⁶⁻⁷⁸ as well as their application in inorganic^{76,77,79-85} and organic⁸⁶⁻⁹¹ analysis. With regard to other electroanalytical methods, one has to mention the works done in potentiometric titration in aqueous^{21,92} and non-aqueous^{28,93} media, amperometric titrations⁹⁴, coulometry^{59,95} conductivity⁹⁶ and, last but not least, potentiometry using ion-selective electrodes^{97,98}. Electrochemical methods are also being used for gas analysis⁹⁹.

Among radiometric methods neutron activation analysis using thermal neutrons^{100,101} or neutron generators¹⁰²⁻¹⁰⁴, non-dispersive x-ray fluorescence methods^{105,106}, and others¹⁰⁷ are being developed. An achievement on a world scale is the introduction of a photoactivation method based on the betatron in the copper mining industry¹⁰⁸.

Chromatographic methods, namely ion exchange chromatography^{109,110}, extraction chromatography¹¹¹⁻¹¹³, paper^{61,114-116} and thin layer^{61,89,117-122} chromatography and gas chromatography¹²³⁻¹²⁸, are being very widely investigated in this country. Basic research is being carried out in chromatographic processes^{129,130}, the properties of sorbents¹³¹, the preparation of new sorbents¹³², also development of specific ones¹³³⁻¹³⁶. Interesting results have been achieved using clathrate compounds as sorbents^{117,122,137}. We have investigated liquid chromatography¹³⁸ and the combination of gas chromatography with mass spectrometry¹³⁹.

In the second half of the period under discussion the analytical chemistry of traces^{3,5,6,9,12,16,17,41,46,47,58-60,63,74,75,100,101,104,110,140,141} and the directly connected separation and enrichment methods¹⁴²⁻¹⁴⁶ have been developed very widely in Poland. Emission spectrography⁴¹, absorption spectrophotometry v.i.s.^{3,5,6,10,13,16,22,58,60}, spectrofluorimetry^{15,69}, chronovoltammetry⁷⁵, polarography^{80,82}, coulometry⁵⁹, neutron activation analysis¹⁰⁰ and kinetic methods³⁵ have been used for determination of trace contaminations in high-purity materials. Separation and enrichment methods are being investigated—in particular, ion-exchange chromatography^{109,110}, extraction chromatography^{111,113}, extraction^{12,139,147,148}, precipitation, coprecipitation and volatilization methods¹³⁹⁻¹⁴¹, and electrophoresis^{149,150}.

A relatively large amount of theoretical and experimental work has been done in this country on basic problems of analytical chemistry. One has to mention several papers concerning the limits of detectability, precision and accuracy of analytical methods^{49,138,151-153}, and studies on planning of experiments in analytical chemistry and on optimization of analytical methods^{52,53,84,154,155}. Various methods are being compared from the point of view of precision^{5,156,157}, the works concerning analytical standards^{54,158-161}, the investigations of the homogeneity of analytical samples¹⁶².

In recent years computers have been introduced into analytical research

for the elaboration of some analytical problems, as well as for collection and processing of analytical data^{52, 53, 55, 149}.

Polish analytical chemistry in the 30 years of the Polish People's Republic has without doubt produced a considerable output. First of all, many chemist-analysts have been educated who are now working in basic and applied analytical chemistry. In the first Polish larger analytical centres organized by Professor W. Kemula (for electroanalytical and trace methods), by Professor A. Waksmundzki (for chromatography), by Professor J. Świątosławska (for spectral methods and problems of precision and accuracy) and by the late Professor M. Struszyński and the author of this paper (for trace analysis and separation and enrichment methods), the new workers became assistant professors and professors, which created new analytical teams embracing new areas of analytical chemistry. In this same period new analytical laboratories in research institutes, universities and polytechnic institutes have been created in which the young chemist-analysts are being educated to work in industry, in medical centres, in agriculture and in protection of the environment. These centres are developing further analytical chemistry and fighting for the still better understanding of its importance. Polish industry, which is being more and more modernized, which has already entered upon the new phase of automation and which undertakes the production of more and more sophisticated materials and devices, understands better and better the role of analytical chemistry and contributes to its general development by introducing modern analytical methods.

One has to expect that the universities and polytechnic institutes in this country, which are also developing rapidly, will have in the near future the possibility of providing analytical laboratories with modern apparatus and become fully modern education centres in analytical chemistry.

The main directions of development of analytical chemistry for the next 15 years, given by the Subsection of Analytical Chemistry of the II Congress of Polish Science are the following: the further development of chemical basis of analytical methods; the further development of analytical chemistry of traces, inorganic as well as organic; instrumentation and automation of analytical chemistry with particular consideration to 'on-line' control; wide introduction of computers into analytical chemistry, which is the first step to full automation of technological processes.

We have the well educated and organized staff of chemist-analysts. The permanent increase in the expenditure of money for research and development in this country confirms that the financial conditions of the development of analytical chemistry will also be fulfilled.

I am sure that Polish analytical chemistry, which has well accomplished its tasks in the first 30 years of the Polish People's Republic, entering the next period will fulfil its duties in the programme of social and economic development of this country.

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* The aim of this set of references is to enable readers to find Polish analytical chemists engaged personally or in teams in research in various fields of analytical chemistry. No other criteria of selection have been used.

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