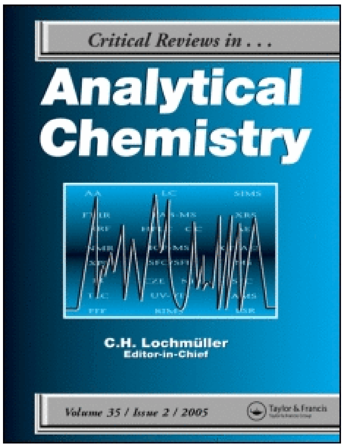
This article was downloaded by:
On: *17 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Critical Reviews in Analytical Chemistry

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713400837

Education in Analytical Chemistry

George-Emil Baiulescu; George G. Guilbault

To cite this Article Baiulescu, George-Emil and Guilbault, George G.(1987) 'Education in Analytical Chemistry', Critical Reviews in Analytical Chemistry, 17: 4, 317 — 356

To link to this Article: DOI: 10.1080/10408348708542798

URL: http://dx.doi.org/10.1080/10408348708542798

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

EDUCATION IN ANALYTICAL CHEMISTRY

Author: **George-Emil Baiulescu**
Department of Analytical Chemistry
National Institute of Chemistry
Bucharest, Romania

Referee: George G. Guilbault
Department of Chemistry
University of New Orleans
New Orleans, Louisiana

Downloaded At: 15:18 17 January 2011

I. INTRODUCTION

It was 4 years ago that this author presented a book, *Education and Teaching in Analytical Chemistry*,[1] intended for students and specialists trying to develop an opinion regarding the role of analytical chemistry as a science and of chemical analysis as an "art" in the progress of modern society. The idea of writing this book was conceived during the International Conference of Analytical Chemistry (Lancaster, July 1980), where this author presented a plenary lecture.[2] At that time the present author was preoccupied with the explosive evolution of analytical methods and, hence, issued a study entitled Moral Ageing of Analytical Methods. Both the book and this study were well received by specialists. Therefore, in 1983, as a guest of the University of Zaragoza, Spain, this author lectured on Analytical Chemistry: Science or Art?[3] to emphasize its role in the development of modern technologies and contemporary society.

The present author's interest in the teaching and education of analytical chemistry has focused on the stages of the analytical process, especially on sampling. Because this author considers that sampling plays a key role in the general analytical process, it has become an important part of this author's efforts.[4,5]

Among other aspects related to the history and philosophy of analytical chemistry, the present author has been particularly interested in the concepts of analytical chemistry. Thus, at the first International Symposium on the History and Philosophy of Analytical Chemistry held in Vienna in 1985, Ciucu and Baiulescu presented a communication on this topic.[6]

The author hopes it is obvious that he considers these problems of analytical chemistry of equal importance with the progress of scientific research in the same field. Perhaps this is why this author was pleased to accept the invitation extended by *CRC Critical Reviews in Analytical Chemistry* to write this paper.

The problems of education in chemistry in general, and in analytical chemistry in particular, gained the attention of higher scientific organizations, especially the Working Party on Analytical Chemistry of the Federation of European Chemical Societies, whose outstanding contribution to the field was recently enriched with a statistical survey on the education of analytical chemistry in Europe, 1983/1984.[7] In the present paper, this author will try to be as original as possible and present a personal point of view.

Starting with this author's 30-year experience in teaching analytical chemistry and chemical analysis, several concepts about education in analytical chemistry will be presented.

Since this author will concentrate on this narrow subject and try to stay free of any "outside" influence, references will supply examples only of some personal views.

To make this contribution original, the author will not follow the structure of the recently published book,[1] but will make a logical and ordered presentation in six sections, whose lengths will depend on their importance. In the above-mentioned book, Chapter 3 was divided into three sections: input, black-box, output. In this publication, the discussion will unfold more philosophically; input is replaced by idea, concepts, and connections, each developed through a separate section. Black-box is replaced by development and applications, and output by aims, i.e., the correlation between teaching, research, and industrial production.

It is hoped that the obligation to supply *CRC Critical Reviews in Analytical Chemistry* with an original contribution on education in analytical chemistry will be fulfilled.

II. THE IDEA

This section will attempt to prove that to carry out successful scientific research in any field one should be educated in that particular field as well as in neighboring fields.

To be educated in a field means to acquire intuition, imagination, and intelligence. It is the duty of a professor to impress upon students through daily contact the primary importance of general education for a future educator or scientist.

Thus, to have original ideas, one should necessarily act with intelligence, imagination, and intuition; the order has been changed on purpose in order to emphasize that only an intelligent man may act with imagination and intuition.

It is certain that the three act together in the development of new ideas. This will also be the outcome of continuous information and, especially, of the daily work of the researcher.

It is worth noting that the educational process that takes place in specialized institutions teaches the future specialist how to think in the field in question. Once the educational term is over, it is absolutely necessary that the truly gifted individuals are hired either in higher education or research institutions.

Downloaded At: 15:18 17 January 2011

In order that the beginners develop their potential, they must be included in a team and work under the supervision of specialists, who will instill in them a passion for scientific research.

With only a few exceptions, found mostly in the past, the outstanding achievements in any field of research have emerged from teams and, today, from multidisciplinary teams.

One may ask whether an idea originates from one individual or from the team. The truth is that the ideas come from individuals, but their embodiment and execution occur most efficiently from discussions among a team of specialists. Exposed to discussion, the ideas may be enriched or even generate new improved ones. This is the element of progress of the collective research activity. Each researcher contributes with intuition, imagination, and intelligence to the achievement of some research project.

The leader of the team should be an experienced scientist, who is able to evaluate and correlate the results and coordinate the work towards the final aim.

A parallel can be established between the high-grade research team and professional Alpinism. The leader of a research team assumes the responsibilities of the head climber, who has to gather competent co-workers, able to take over leadership and continue the work.

This is the role of a continuous leadership through the individual and for the individual.

The university professor should be a person of vast general scientific culture; in addition to being a good specialist only such an individual can train future teachers and professionals.

One may encounter in some countries of Europe as well as in the U.S. a tendency to ultraspecialization in important, yet restricted, fields. This trend may be useful because those schools which adopt it produce graduates of high specialization in such fields as IR-spectrometry, gas chromatography (GC), high-pressure liquid chromatography (HPLC), etc., exceedingly useful areas in applied analytical chemistry.

To do research in analytical chemistry, however, one needs "knowledge extended on the horizontal", i.e., knowledge that one needs in order to develop "analytical thinking".

The role of the leader is to teach the beginner how to think rather than how to reproduce.

This section shall be divided into three topic areas, intuition, imagination, and intelligence, the meaning of which should be elucidated next on the basis of examples.

This author will not refer to the philosophic meaning of these terms, assuming that these are known, but will discuss their meaning in relation to the formation of the field in question.

A. Intuition

Intuition is a feature of an individual of normal physical and intellectual evolution. It assumes thinking without excessive utilization of extraneous sources of information. Watching nature, man learns by intuition how to use to his advantage various natural products and how to improve their quality. To achieve the latter, one should resort to imagination and intelligence. A study of the history of analytical chemistry will show that, in its early stages, such observations were intuitive.

Because the extent of this paper allows only a discussion of general problems, this author gives here only one example which he thinks is conclusive evidence of the role of intuition in acquiring some results or some information.

Whether we like it or not, chemical analysis in ancient times may be traced to sampling. At first, man picked materials from nature to process them into useful products.

The age of metals evidences this. Early man tried to obtain metals from rocks and minerals; metals were used as such or in alloys; this was the Bronze Age.

In the case of some metals existing in nature, e.g., gold and the platinum metals, attempts were made in old times to find new sources. This required new methods of separation and, of course, analysis.

Thus, according to Szabadváry (see his excellent book *History of Analytical Chemistry*) "the cupellation procedure was already in general use in the 14th century, and many records show that at this time it was in statutory use for the examination of gold."[8] It was also Szabadváry who assessed the antiquity of the fire-assay process: "The fire-assay has been used since the beginning of the recorded history of analysis. In the Old Testament there are several references to its use."[9]

Due to the special properties of gold, such as malleability and ductility, its sampling imposed special requirements: to obtain correct analytical information on the gold content of a sample, large amounts of it were needed (tens of grams). Wet treatment of large samples created some difficulties and required utilization of the fire-assay method.

In a relatively recent paper, Kallman shows: "Perhaps the most important single analytical method in precious metals analysis is the fire-assay, considered by many to involve as much art as science."[10] This remark contains a lot of truth. In Baiulescu's lecture at Zaragoza,[3] it was remarked that, while analytical chemistry is a well-defined independent science, chemical analysis is, in many instances, closer to an art. This "definition" may include the fire-assay as well. It is interesting that this assay has retained its validity through the ages. Several modern physical methods of analysis may be employed after separation by fire-assay to analyze impurities in gold, and even gold itself. Thus, one can successfully employ atomic absorption spectrometry (AAS) or atomic emission spectrometry (AES) with inductively coupled plasma.

B. Imagination

Imagination is a feature of any man with some education in a certain domain; it is developed by sustained accumulation of information from specialized literature or, to a greater extent, by activity in the laboratory.

One can prove that in analytical chemistry imagination has facilitated major discoveries. Although the present author shall return to this point, it is important to mention that the study of reactions with organic reagents has benefited substantially from imagination.

Dimethylglyoxime is regarded as one of the first analytically useful organic reagents; however, these reagents have been used as natural products for quite a long time.

Thus, Szabadváry writes: "In fact the first analytical reagent of which there is record,

Downloaded At: 15:18 17 January 2011

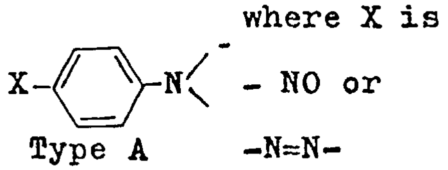
Downloaded At: 15:18 17 January 2011

the gallnut liquid, recommended by Pliny for identification of iron, is an organic reagent. Several other organic reagents were in common use long before dimethylglyoxime was discovered, for example, oxalic acid, tartaric acid, succinic acid, and starch.''[11]

A systematic study of various natural organic reagents has revealed that they possess relatively limited possibilities. Many organic reagents with dedicated groups have resulted from the field of organic synthesis. The controlled synthesis of organic reagents has demonstrated the existence of some groups which ensure that the reaction occurs and, perhaps, even the selectivity. These groups were termed functional or active analytical groups.

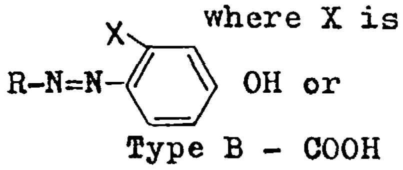
With their creative imagination, analytical chemists introduced into practice a large number of high-performance organic reagents, such as oxine, cupferron, dithizone, thenoyltrifluoroacetone, Arsenazo III, etc.

In past and present research carried out in the Department of Analytical Chemistry at the University of Bucharest on the analytical chemistry of palladium, it has been shown that this element may react in solution with organic reagents which contain two types of analytical functional groups (A or B) as shown below:



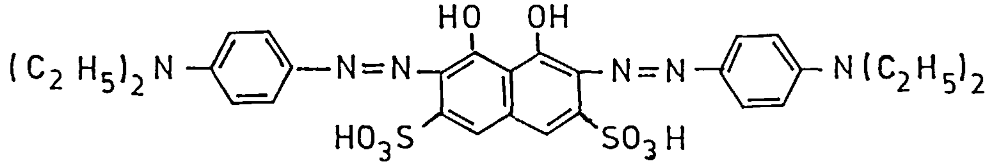
Type A

and

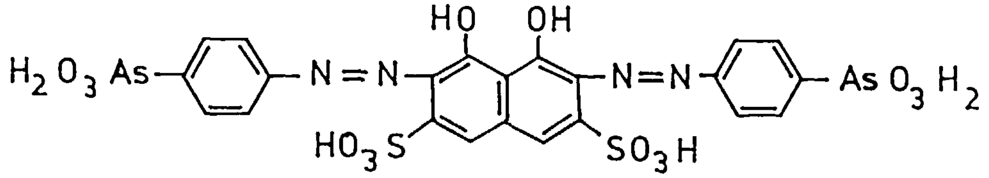


Type B

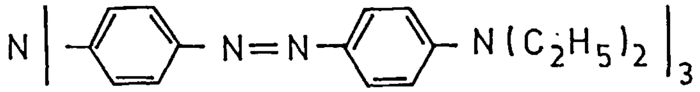
Systematic studies on a large number of organic reagents have shown that Methyl Red gives a very sensitive reaction with palladium (II).[12] Examination of this reagent reveals that in the molecule there exist both types of groups A and B. Therefore, a highly sensitive reagent for the spectrometric determination of Pd (II) was suggested,[13]



which gives a reaction whose sensitivity is similar to that of Palladiazo reagent.[14]



Recently, a new reagent was synthesized that outclasses all these reagents and is very sensitive, a trisaze derivative of triphenylamine,[15]



which, due to its very high sensitivity in reaction with Pd (II), was called Pallatriazo.

These examples demonstrate the role of imagination in the programing of some experiments designed to obtain competitive organic reagents.

Unfortunately, efforts have so far resulted in reagents of either very good selectivity or high sensitivity. However, the two operational parameters of the analytical process are interdependent and, therefore, the intelligence of the analytical researchers will lead to reagents with both good selectivity and high sensitivity.

All the foregoing examples prove that imagination and intuition are necessary but not sufficient by themselves for a researcher to contribute substantially to the research and implicitly to the teaching and education of analytical chemistry. There is an additional condition: intelligence.

C. Intelligence

Only an intelligent individual can conduct meaningful research. The teacher should develop the intellectual capacity of the student, influencing his passion for the profession chosen. A dedicated individual succeeds in selecting from the enormous amount of information available that which ensures the development of thinking towards a given aim. Only an intelligent individual can read "between the lines". The teacher should show his students how to read and how to think while reading.

All major discoveries in the history of science stemmed from simple facts that at first sight may have seemed obvious and ordinary. At certain times in the development of science, gifted researchers appear who can draw the essence from the information accumulated. They are the researchers who "push" science forward, introducing new fields of research. Examples of this are polarography and atomic absorption. Consider the evolution of a technique which has already become "classical" for chemical analysis: AAS. This technique, in its initial form, employed a flame as the "reagent", extracting information from the sample. This use of flame as the "reagent" has been studied for a long time by famous researchers such as Bunsen and Kirchhoff. Unfortunately, few analytical chemists are aware of the basic contribution of a Romanian scientist, Nicolae Teclu, who thoroughly studied the behavior of various flames and developed the Teclu burner. This discovery made him known as a remarkable researcher.[16]

Downloaded At: 15:18 17 January 2011

The experience gained with the utilization of flame in qualitative analysis, together with the finding regarding the characteristic coloring of flame by ionic compounds (sodium, potassium, calcium, barium, etc.) have set the foundation of emission spectrometry. Unfortunately, this technique seemed obsolete from the beginning because only a limited number of elements could give out some information with the flame as reagent; the temperature of the flame was not sufficient to produce ionization. Using some hot flames produced by special gases, e.g., $(CN)_2 + O_3$ etc., has produced only limited effects.

When everybody considered that the usefulness of the flame as reagent was ended, Walsh, Alkemade, and Milatz launched their idea that information should be obtained from the atoms in the ground state. This may seem obvious to everyone now, but it came as a flash of intelligence.

Thus, AAS was born, a technique with high sensitivity and fairly good selectivity for a large number of elements.

This technique has not eliminated flame spectrometry because the two are complementary. Although atomic absorption, especially in its flameless variant, offers obvious advantages, it is a sequential technique, with some disadvantages. Therefore, to supplement it, the intelligent research of Greenfield and Fassel resulted in ICP-AES.

The flame spectrometry — AAS-(ICP-AES) triad — supplies an eloquent proof for the statement that intuition, imagination, and intelligence contribute to the development of science, to the birth of new fields of science, and to the improvement of old ones.

III. CONCEPTS

This section has a pronounced content of the philosophy of analytical chemistry. This is so because the present author intends to discuss here the fundamental concepts of analytical chemistry: quality, quantity, and structure. These concepts are the basis of education in analytical chemistry and of research in chemical analysis.

To obtain reliable analytical information, one should, at first, act intelligently upon the sample. This author introduced the notion of reliability of analytical information during a symposium in Helsinki,[4] trying to prove that, as in electronics, one should be certain of the process of obtaining analytical information. It was shown that all three concepts of analytical chemistry are in direct interdependence upon the sample to be analyzed.

Therefore, before any discussion about these concepts, some references to the sample to be analyzed will be made. As has been shown,[5] the sample acts as a connection between the method and the instrument. Only an exact knowledge about the history of a sample ensures the obtaining of reliable analytical information. To act efficiently upon the sample, one has to know its history, that is, where it comes from, and one should have knowledge of basic chemistry, geochemistry, physiology, the environment, etc. Only by employing adequate chemical or "physical" reagents can one get the needed information.

Unfortunately, while many books and reviews about the methods of analysis have been and are being written, only recently have some works been dedicated to the sample, the most complete and interesting of which was published by the Royal Society of Chemistry,[17] refering only to some aspects of sampling, especially, the preparation of the sample.

If we deal now with the stages of analytical process:

Sampling → Measurement → Data processing

we notice that due to automation and robotization the measurement itself takes ever shorter times. Most of the attention is devoted to sampling and data processing. A chemometric estimation of the three steps of the analytical process yields about a 40:20:40% ratio.

Sampling claims a predominant part of the general analytical process and is often the main source of errors of analysis. The sampling of solids is not yet automated.

English is, according to my knowledge, the only language that includes in one word — sampling — all the operations regarding the sample, beginning with collection and ending with passing it to the measuring equipment.

The first condition imposed on a sample is perfect homogenization, and, in the case of surface analysis (beam analysis), the reproducibility of the surface to be analyzed from which the beam (reagent) picks up the analytical information.

In the present author's opinion, sampling includes the wide variety of separation methods because they must solve intricate problems. This author considers chromatographic analysis an improper term because it is really a chromatographic separation, after which the components are analyzed step-by-step by a more or less sophisticated detector.

The separation techniques are the heart of any analytical process. As far as efficiency is concerned, these techniques have contributed, through automation and computerization, to the control and optimization of the technological process. On-line chromatography has become commonplace in inspections of product manufacturing.

Separation methods are no longer in the laboratory stage; they are used in the reprocessing of nuclear materials and in HPLC-type industrial equipment, which yields chromatography-grade chemicals.

Downloaded At: 15:18 17 January 2011

A comprehensive study on the beginning and development of nuclear energy was recently issued by the remarkable French specialist Goldschmidt.[18] When one reads this book (between the lines as well), one realizes that in the nuclear programs of various nations an important part was played by analytical chemistry, both through its separation and analysis techniques.

All these facts argue for the need of giving more importance to the sample in teaching and education in analytical chemistry.

Examination of the correlation of sample-method-instrument or, to be more equitable, method-sample-instrument shows that no matter how improved the methods of analysis and the equipment the analytical result will never supply the real value of sample composition because of the inherent errors of sampling. As was already stated: "No analysis is better than the sample itself."[4,5]

After these brief considerations regarding the sample, let us now discuss the concepts of quality, quantity, and structure.

A. Quality

Analytical chemistry as an independent scientific undertaking operates with three well-defined notions: quality, quantity, and structure. Teaching and education in analytical chemistry should give corresponding importance to these concepts because they practically define the domains of chemical analysis — applied analytical chemistry.

Unfortunately, the importance (hours of class and laboratory) and affiliation (i.e., teaching by analytical chemistry staffs or by others) of these domains differ from one nation to another. The statistical surveys which are made from time to time are, in this respect, useful; one of the successful surveys for Europe was conducted by the Working Party on Analytical Chemistry.[7]

In the following, this author gives his personal views on these concepts. First of all, let us see which is the logical order of teaching knowledge of analytical chemistry and chemical analysis.

To characterize a sample, after the preliminary processing of the material, one has to obtain its fingerprint. This may be more or less complete and certain or can establish its quantitative approximate composition. The qualitative analysis tells us about the nature of components and, if correct, about the ratio between the components.

While early chemical analysis required quantitative characterization of some natural products, such as rocks, ores, waters, and gas emanations, the development of organic synthesis and of organometallic compounds has enlarged the spectrum of qualitative analysis. The qualitative characterization of synthetic products has imposed the utilization of improved methods of separation and analysis (chromatographic and spectrometric, respectively). Thus, the chromatogram or spectrogram of a well-defined product represents its fingerprint. These fingerprints are listed in catalogues and exist in the memory of intricate equipment (e.g., the GC-MS-computer).

It is erroneous to believe that qualitative analysis is restricted to reactions in a flame or a test tube, although it may start like that.

It will be shown in another section that the study of chemical reactions should not be neglected. The study of these reactions has facilitated the devising of new techniques of analysis (e.g., utilization of ion-selective electrodes).

Ion-selective electrodes have emerged from the intelligence of some researchers who employed well-defined reaction products as other solid or liquid membranes.

Due to the large number of possible reactions in solution, the corresponding number of such electrodes is unlimited. The problem is to prepare electrodes with both adequate sensitivity and selectivity.

The concept of quality is correlated with the two operational parameters of the analytical process, i.e., sensitivity and selectivity.

Those who study reactions have to observe both requirements of the analytical process.

Downloaded At: 15:18 17 January 2011

Thus, to do qualitative analysis, one should find sensitive and selective reactions. In case the selectivity is not adequate, one may resort to masking agents or the techniques of separation.

Laboratory work should teach the student the "alphabet", i.e., the basic notions of analytical chemistry and chemical analysis. Simple reactions conducted correctly in the laboratory will get the student accustomed to accuracy and will develop scientific thinking.

When this author was a student 35 years ago, the art of a chemist, Lecturer Teodosiu of the University of Bucharest, made an impression. He taught the reactions of qualitative analysis and impressed on his students the opinion that it is more difficult to do qualitative analysis than quantitative analysis.

Modern automated, robotized, and computerized analyzers confirm this opinion. Such a piece of equipment may be easily operated by a technician. However, one cannot only operate some apparatus, one must also know what information to supply to that apparatus.

Qualitative analysis is a tremendous source of information, useful in the formation of a specialist in analytical chemistry. It may be useful in learning about properties of various substances and compounds, learning about their behavior in different reaction media, etc. Consider, for example, an ordinary reaction of which every beginner is aware: the reaction of Co (II) ions with SCN^-. This reaction is well known to result in a blue complex of Co (II) $[Co(SCN)_4]^{2-}$ which is very unstable in an aqueous medium. To stabilize this complex, either a modification of the reaction medium or the withdrawal of the complex from the reaction medium is required. Addition of acetone, miscible with water, changes the dielectric constant of the medium and contributes to the stabilization of the Co (II) complex, preserving the blue color. This tells the student about the importance of the dielectric constant of the solvent in chemical reactions and demonstrates that the most stable complex combinations of cobalt are given by Co (III). If, however, the compound is extracted in a mixture of amyl alcohol and ether, its stability is even higher. The student thus discovers the fundamentals of separations through extraction from solvents immiscible with water.

Downloaded At: 15:18 17 January 2011

The contest or, more correctly, competition between sensitivity and selectivity represents the driving force of qualitative analysis.

The analyst's art consists of balancing these operational parameters in order to optimize the analytical process. Obviously, the early qualitative and quantitative analyses were practiced without a substantial theoretical background. However, Oswald and, more recently, Kolthoff, Feigel, Belecher, and Charlot (to quote only some of the pioneers) set the theoretical foundation of qualitative and quantitative analysis both inorganic and organic.

As has already been mentioned, research on reactions led to the development of new analytical techniques. Let us return now to ion-selective membrane electrodes. The pioneering research carried out throughout the world (e.g., by the group of Pungor, Simon, and Guilbault) has shown that the glass electrode is a particular case which can tell us about the quality and quantity of hydrogen ions. Various membranes, be they solid or liquid, can pick up information about the solution. This is how the wide variety of ion-selective electrodes, which give us information about the quality and quantity of components in solution, emerged. The selectivity of the information supplied depends on the electrode membrane as well as on the complexity of the medium analyzed. These electrochemical sensors tell us, first, about the existence of a component in solution and then about its amount. These electrodes resulted in the publication of a large number of articles about the analysis of organic or biological compounds. The membranes evolved so that enzymatic and tissue sensors were produced. Although their sensitivity has continuously been improved, the selectivity ISE's are still poor. The present author looks forward to witnessing the improvement of their analytical performance.

The electrodes with a solid membrane have already become classic in the determination

Downloaded At: 15:18 17 January 2011

of some inorganic anions with a simple or automatic system. The study of reactions in solution, and of extraction with solvents, has recently made possible the development of electrodes for the characterization of organic compounds. These electrodes operate with liquid or solid membranes, consisting of internal complex combinations or ionic association complexes extracted in organic solvents immiscible with water.

To evidence the importance of ion-selective electrodes in organic analysis the present author published a monograph in 1977.[19] This book, as well as the original contributions presented therein, led to a study of reactions in solution, especially the study of complex combinations. To support this statement, two recent works should be noted that were issued by a group of co-workers aimed at determining with ion-selective electrodes sulfur drugs[20] and lidocaine hydrochloride.[21] Both have employed liquid membrane electrodes, the former complex combinations of silver and copper of two derivatives of aza-uracil, the second either an ion-pair complex of the lidocaine cation with dipicrylamine in nitrobenzene or lidocaine dinonylnaphtalene-sulfonic acid ion-pair complex in a PVC matrix.

Qualitative analysis will always be of prime importance. Do not forget that various chromatographic techniques such as paper, thin-layer, or capillary-gas chromatography initially supply the fingerprint.

Various studies of criminology and of environmental chemistry rely in the first stage on qualitative analysis to fingerprint in order to make known the presence or absence of some components in the samples.

Therefore, this author declares for the teaching of analytical chemistry and for chemical analysis that the detailed study of reactions is the first step rather than the final step.

B. Quantity

The quantitative aspect of analytical chemistry is discussed next as part of the general analytical process:

$$\boxed{\text{Input}} \rightarrow \boxed{\text{Black-box}} \rightarrow \boxed{\text{Output}}$$

Quantitative analytical chemistry is the logical continuation of the qualitative act because it determines the ratio between the components of the sample. It is supposed to determine most exactly the content (the amount) of components present in the sample to be analyzed. Its basic equation is

$$P = f(C) \tag{1}$$

i.e., it determines a physical property (P) which is concentration dependent f(C).

It turns out that there may exist a large number of methods of analysis because various physical properties may be employed to obtain information about the sample. Therefore, the methods of quantitative analysis may be gathered under the general term "physical methods of analysis in analytical chemistry", which this author considers the most adequate.

Referring to gravimetry and volumetry as chemical methods is inadequate because the methods are based on the determination of some physical properties, i.e., the mass and the volume, respectively. Likewise, physicochemical or instrumental methods of analysis (terms encountered especially in the American literature) represent inadequate names given to those methods.

In quantitative methods of analysis, the main role is played by the sample, and, therefore, its nature and complexity determines the classification of methods as destructive and nondestructive.

If fast analytical information is required, the nondestructive methods seem preferable (if we include all beam analysis methods, they should be called partially destructive). This is so because the time required for the preparation of a reproducible surface of the sample and for the obtaining of analytical information following the impact of a beam is shortest.

However, in the case of complex samples, it is impossible to acquire good selectivity in the presence of many components and, therefore, separation of components is compulsory. Besides being a laborious operation, separation exposes the sample to contamination (more so in the case of trace analysis).

The student has to be taught that to do quantitative analysis one has to work carefully, accurately, and, if you wish, with competence. The laboratory has to be clean, climatized, and, in special cases, pressurized because all these conditions may affect the analytical information.

It follows that the operator has to act rationally upon the sample. Only after obtaining the preliminary information about the sample by qualitative analysis can one choose the adequate method of analysis and equipment.

There are three basic correlations which should be taken into account in order to obtain correct quantitative results: method-instrument correlation; man-instrument correlation; method-man-instrument correlation. This is the only way of establishing the optimal conditions of analysis for various materials.

The art of the analyst consists of the adequate selection of a method based on the most complete knowledge of the history of sample.

Let us consider two further possibilities offered by the sample of quantitative analysis.

Destructive methods of analysis are more involved but may yield valuable information about complex samples. Nondestructive methods are complementary to destructive ones and apply efficiently mostly to trace analysis.

The range of the amount of sample subjected to quantitative analysis extends from tens of grams (fire-assay) to submicrogram quantities in the case of beam analysis.

During this author's undergraduate studies in the 1950s, the lower limit of the analytical method was 10^{-6} g (microchemical methods), but today the 10^{-6} to 10^{-18} g region (trace analysis) is ever more studied by analysts.

Bringing the sample into solution, in the case of destructive analysis, requires a knowledge of basic chemistry for greatest efficiency. Thus, several wet or dry methods of disintegration of the samples have been devised which are very laborious and, in certain cases, require expensive equipment (like a platinum crucible). Once Teflon® (a highly inert material) became available, special devices for sample disintegration under pressure were manufactured. These shorten the time of sample disintegration and avoid the danger of contamination. These devices must be introduced into undergraduate laboratories and used with conventional equipment.

This author believes that we cannot consider as skilled those analysts who are not able to carry out correctly an analysis of silicates, i.e., of their major and minor components. In the analysis of silicates, disintegration under pressure considerably shortens the duration of analysis. In the "normal" system of disintegration, hydrochloric acid (even if hot) is only partially successful; utilization of pressure ensures disintegration of the silicate lattice and the solubilization of all sample components.

With solubilization of the sample, the operations that precede the analysis are not over; depending on the complexity of the sample, there are more preliminary operations such as masking of some components in solution or separation of components by various experimental techniques. Selection of masking and separation techniques requires a solid knowledge of both complexation and solution chemistry.

The improvement of equipment for the separation and analysis of components from complex mixtures has caused a partial overlap of the separation and analysis processes. The

Downloaded At: 15:18 17 January 2011

best example is that of ionic chromatography, a relatively recent chromatographic technique, which affects the separation of mixtures of organic and inorganic ions. An efficient separation by chromatography together with a conductometric detector yield an analytical signal in a very short time (10 to 15 min in the case of complex mixtures).

Thus, the separation and analysis of anions was achieved, a process which is difficult by other physical methods of analysis.

In nondestructive analyses, sampling is relatively easy, espeically for gases and liquids. The improvement of chromatographic methods of separation like GC and HPLC and the utilization of high-performance detectors such as MS and FTIR have ensured the finding of highly reliable analytical information.

Sampling is more difficult in nondestructive analyses on solid samples because reproducible surfaces must be obtained either by grinding the surface or by pelletization at reproducible pressure (in case of powdery samples).

A philosophical critical evaluation of quantitative analysis methods will now be presented.

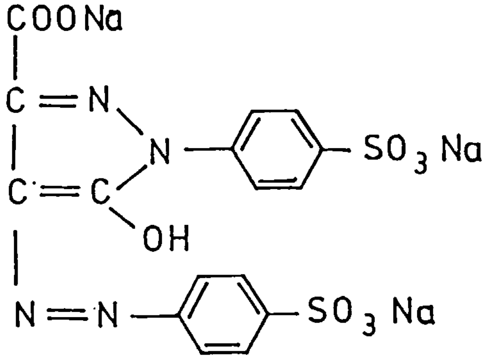
This evaluation will begin with the so-called classical methods: gravimetry and volumetry. They are now, in the present author's opinion, as efficient as in the past and they will remain so in the future. They may be applied to various samples and for various contents of components in the sample. Although other methods of chemical analysis have restricted their importance, they retain an instructive value and must be taught to students.

Some departments of chemistry give them little importance, if any at all, and this is a mistake. These techniques get the students acquainted with the "alphabet" of research in analytical chemistry and chemical analysis and help them learn about the formation of precipitates and the theory of solutions.

Several organic compounds may be titrated in nonaqueous or partially aqueous media.

It has been shown previously that several organic reagents may be employed efficiently in gravimetric and volumetric methods of analysis. Here is an example of an organic reagent utilized in the gravimetric and amperometric determination of zirconium. This reagent is tartrazine:

Downloaded At: 15:18 17 January 2011



The examination of this formula shows that it is an azopyrazolonic derivative; it forms a unitary precipitate with zirconium. The reagent exhibits a good selectivity and is competitive with the best reagent utilized in the gravimetric determination of zirconium, such as mandelic acid.

The method is certainly good because it has been used for the determination of zirconium in various materials in Romania, the U.S., Poland, and the U.S.S.R.[2]

It turns out that one can devise good methods from the standpoint of operational analytical parameters through a systematic study of the behavior of various analytes.

Now consider a more usual reagent which, during the 3rd decade of this century, contributed to the elaboration of new, quite important gravimetric techniques. Studies carried out in Cluj (Romania) by Spacu and Dick on complex combinations of pyridine with other addends have resulted in a new, highly efficient gravimetric method based on the formation of the compound:

$$[\mathrm{Cu\ Py_2\ (SCN)_2}]$$

The compound has a unitary composition which allows the immediate weighing of the precipitate after washing in special conditions with ethanol and ether and drying in a vacuum desiccator over P_2O_5. The method was adopted for titration as well as for absorption spectrometry (by dissolution of copper complex in chloroform); it was employed in the separation of copper from other metal ions with adequate masking agents. The method is extensively referred to in a monograph[22] which was signed by a distinguished contributor to the field of organic reagents in analytical chemistry.

In this discussion about the gravimetric method, one should not overlook the technique of precipitation in homogeneous media, e.g., thioacetamide.

Applying this reagent in laboratory practice, the student learns about the kinetics of hydrolysis of this compound in various media, with the *in situ* evolution of hydrogen sulfide.

Thus, one may notice that methods which are at first very simple may turn out to bear a substantial educational role for students in thorough analysis. Therefore, these "classical" methods of analysis must be awarded the attention they deserve, at least in the process of education.

This may be best emphasized by the benefits to analytical chemistry and chemical analysis of gravimetry.

The earlier studies of Duval and collaborators in France, continued in different countries, set the foundation of thermogravimetry. This technique is today a method of analysis of composition as well as of the structure of compounds. The importance of this technique is emphasized by the large number of publications and by the existence of specialized journals, such as the *Journal of Thermal Analysis*. Likewise, thermal analysis equipment has improved continuously. Important to mention here is the high-performance apparatus, called the Derivatograph, produced in Hungary.

In the previous discussion of the moral aging of analytical methods,[2] it was noted that those methods of analysis which seem "obsolete" at a certain moment are sometimes renewed, thus retaining their viability and value.

The new methods of analysis arise from the need to cope with problems of technology, which is in a state of permanent progress. They do not replace the existing methods, but compete with other techniques at various ranges of component concentration and samples of varying degrees of complexity.

It is interesting that some of the methods of analysis renew themselves at certain stages of development, as supported by progress in theory and equipment.

In a recent paper by Hieftje,[23] it is specified that "application drives theory" and "for the field to progress as rapidly as possible, then, it is desirable that communications between the fundamental and applied sides of analytical chemistry be as thorough as possible and that development in both areas find their way into the other efficiently".

The catalyst for this process is often instrumentation.

$$\text{Theory} \qquad \underset{\rightleftarrows}{\text{Instrumentation}} \qquad \text{Application}$$

An obvious example is mass spectrometry (MS). Like thermal analysis, this is a technique of analysis as such, as well as a technique of structural analysis. It combines features of

Downloaded At: 15:18 17 January 2011

separation and analytical techniques. Employed at an early stage for separation and analysis of isotopes, it later became useful in structural analysis. Adaptation of a spark source turned it into a technique of high sensitivity and selectivity for trace analysis (spark MS). All these techniques today retain their usefulness.

Moreover, due to the performance of the method, it has been utilized in a high-efficiency detection system, coupled with gas and liquid chromatography of high performance (GC-MS and HPLC-MS).

Those who teach classes in physical methods of analysis in analytical chemistry are accustomed to classifying the methods into large groups according to the principles which underlie them.

Thus, groups of methods, such as electrometric methods, optical methods, thermal methods, and magnetic methods, are common.

Some theoretical and practical aspects related to the multitude of physical methods in chemical analysis will now be discussed.

As shown above in the discussion about gravimetric and volumetric methods, the final goal of any method is to acquire information about the sample with some certainty. In order that information be the most reliable, the analyst has improved some methods and has devised new ones.

It is interesting to follow the evolution of various methods of analysis. Electrometric methods, unlike optical methods, apply only to samples in solution. The signal is picked up from the sample containing solution by a system of electrochemical sensors. Depending on the quantity which is measured, a wide variety of electrometric methods exist: potentiometry, conductometry, polarography, amperoometry, etc. Each of these techniques has diversified into techniques which are very sophisticated from a theoretical and practical viewpoint. Many of these techniques may be too complicated to fit into an automatic control system.

From among the electrometric methods, three are distinguished for their analytical implications: polarography, potentiometry with ion-selective electrodes, and last, but not least, anodic stripping voltammetry (ASV). All of these have been developing, and will certainly continue to develop, because their theoretical basis was elaborated by outstanding people, like Heyrovsky, Pungor, Simon, Guilbault, and Kemula, to name only some of the top scientists.

Like MS, polarography has regenerated itself. Although its beginnings may be traced to the 3rd decade of this century and resulted in a Nobel Prize for Heyrovsky, it seemed obsolete for a long period. It was the pioneering research of Zuman and Kemula which revitalized it.

Zuman contributed substantially to the utilization of polarography in organic analysis, which represented a spectacular enlargment of its sphere of applications.

Kemula instituted ASV, which is a great improvement over classical polarographic techniques in sensitivity (its sensitivity extends to parts per billion). The high performance of this technique is explained by the fact that it combines two analytical procedures in harmony; one of separation (pre-electrolysis with deposition of analyte on the stationary electrode) and the second anodic resolution (which supplies the analytical signal).

Due to its high sensitivity and precision, this technique has become exceedingly useful in the determination of traces in solution; its selectivity is also very good. The method may be utilized jointly with AAS in the testing of various samples because of the similar performance of the two methods. Vydra et al.[24] have written a good book on ASV.

The great advantage of electrometric techniques over other analytical techniques is the simple and inexpensive instrumentation they use. They may be utilized in flow analysis and adapted to continuous automatic control. Some ion-selective electrodes are employed in the automatic control of water quality.

Downloaded At: 15:18 17 January 2011

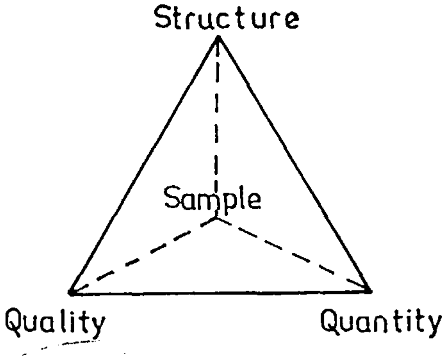


FIGURE 1. Sample implementation in the analytical process.

In teaching these techniques, one should emphasize their theoretical aspects in order to give the students a good foundation for a correct application in the laboratory.

Spectrometric methods of analysis are also very diverse. In the teaching of these methods, one should consider them comparatively according to their performances. The student should know the physical basis of the various spectrometric techniques. As will be shown in another section, the improvement of instrumentation requires a knowledge of electronics and computer use.

The sensitivity of spectrometric methods of analysis vary within wide limits, that of emission spectrometry is now at the higher limit of trace analysis. The appearance of AAS, especially with electrothermal atomization, and ICP-AES have brought these techniques to the same level of sensitivity as radiochemical methods of analysis, which are famous for their sensitivity.

IR spectroscopy used to be a fingerprint-type analytical technique; today, the introduction of transformation techniques in chemistry has turned it into a technique suitable for rapid analysis of small concentrations of components. FT-IR will be further discussed when we approach the methods of structural analysis.

The analyst must put the method on the instrument without being influenced by the latter. An instrument, however intricate, works only when operated or programed by man.

In a recent paper, Ewing and Van Swaay wrote: "A good curriculum should provide students with enough understanding of electronics, mechanics, optics and computer science to allow them to cooperate effectively with experts in those fields."[25]

This does not mean the analysts have to process far-reaching knowledge in all these fields; they need only basic knowledge so that they are able to employ the instrumentation and adapt it to the experiments in question.

The latest concept in analytical chemistry — the structure — will now be considered.

C. Structure

Perhaps the most interesting aspect of analytical chemistry and chemical analysis is structural analysis. It is the highest art of the analyst. Structural analysis refers to the sample as well, but while qualitative and quantitative analyses supply information "along the horizontal", structural analysis gives total information upon the sample, i.e., information along the vertical. This is shown in Figure 1. The sample was left on purpose in a plane behind that of the paper leaf because it is the one which influences the decision of the analytical chemist about the type of analysis to be performed.

There has existed, and still exists, the opinion, which this author considers inadequate and which has unfavorable effects upon the process of teaching, that structural analysis

Downloaded At: 15:18 17 January 2011

should be taught in departments of physical chemistry. In many higher education institutes, this opinion has robbed analytical chemistry of its most interesting part — the characterization of matter. The papers on education in analytical chemistry, and through analytical chemistry of future chemists, should grant this important aspect of chemical analysis the proper attention.

To argue for this point of view, consider that in order to do structural analysis on an unknown sample which is common today because of the large number of compounds synthesized, it is first necessary to know the history of the sample and the quality and quantity of its constituents. To do structural analysis as such means to do "art for the sake of art", and the resulting information has low reliability.

However, structural analysis bears its own specific features, which make it hard to approach. This was so because to obtain certain data about the structure of a component implies the correlation of data, originating in various techniques of structural analysis. The analyst has to gather solid information about the theoretical and practical aspects of various techniques of structural analysis. Because one individual cannot generally be a specialist in several techniques of structural analysis, the determinations are carried out in various laboratories by specialists and then put together for correlation. Therefore, in the curriculum of analytical chemistry, quantitative analysis should precede structural analysis. Moreover, the course of structural analysis should be a general one for all students, rather than a specialization in analytical chemistry or physical chemistry.

From among the methods of chemical analysis, the course should present those which are useful to structural analysis.

The classification in the present author's recently published book[1] is maintained here as well: infrared spectrometry (IR)-Raman spectrometry (RS); mass spectrometry (MS)-nuclear magnetic resonance spectrometry (NMR); and X-ray diffraction-Neutron diffraction.

This pairing of methods does not rule out the possibility that useful data can be supplied by other techniques, e.g., UV spectrometry or thermal analysis. However, the six techniques mentioned are those required in order to obtain certain data about the structure of a compound.

This author believes that the topic of a general course must include the theoretical and practical aspects of the methods of structural analysis; this would be a necessary and sufficient condition for the training of a future analyst.

Thus, the interpretation of a structure implies the correlation of data obtained by various techniques of structural analysis; this may be done if the student has practiced it. Fortunately, handbooks have been issued about the interpretation of structures on the basis of structural information supplied by various techniques.

In this respect, it is worth mentioning a complete textbook published recently in Romania; it includes 60 detailed examples of structural interpretation for some organic compounds.[26]

While the literature on structural analysis (books, reviews, and articles in specialized journals) are fairly numerous, the instrumentation existing in the laboratories of university chemistry departments is scarce. The rapid modernizing of laboratory equipment has resulted in high prices, far above those many institutions can afford.

"A survey initiated by a task force of the American Chemical Society (ACS) Joint Board — Council Committee on Science was conducted in 1982 to obtain information on the inventory of scientific instruments in college and university chemistry departments, and on the perceived needs of those departments for new instruments."[27] This survey emphasized the need to replace laboratory instrumentation with competitive equipment.

In conclusion, the survey remarks, "With rapid changes in technology taking place, instruments can become quickly outdated. A continuing program to update academic instrumentation is needed if we are to provide academic researchers with tools sophisticated enough to deal with today's complex scientific challenges, and provide an educational experience relevant to employment in industry or pursuit of breakthrough research."[27]

Downloaded At: 15:18 17 January 2011

It is obvious that an attempt should be made to ensure adequate instrumentation for structural analysis in university departments.

Next a brief comparative and critical discussion of the previously mentioned six techniques of structural analysis will be undertaken.

Examining the first pair — IR and RS — one may realize that the data they supply are complementary. In their "classic" variant, both techniques, although useful, are slow. The newer improved systems are much faster, e.g., IR interferometric spectrometry, referred to as Fourier transform (FT)-IR. The introduction of the laser as a light source has eliminated a series of disadvantages of the mercury-arc in excitation for RS. These improvements have made the methods faster and the analytical information more reliable so that the techniques are competitive for structural analysis.

As was mentioned earlier, in its FT-IR variant, this technique may be the ideal detector for GC and HPLC and so are the adaptations as detectors of FT-MS or FT-NMR.

Much has been published about these techniques but it is only necessary to mention two references.[28,29]

These tandem techniques are very useful in the characterization of some samples of extremely complex composition like oil products and flavors.

Things are similar with MS and NMR. MS as a technique of structural analysis benefits today by the existence of high performance equipment.

The perfectly reproducible mass spectra are listed and represent the fingerprint of the compound in question. The art of the analytical chemist who studies structure is to reconstruct the entire initial molecule from fragments.

Some specialists in MS have compared it with its opposite, organic synthesis, which obtains the whole from fragments. In the correct interpretation of mass spectra, a substantial contribution has been made by chemometrics through its pattern-recognition.

To prove the efficiency of MS as a detector in GC, one can mention that a GC-MS computer system, with a capillary column for separation, allows the identification and determination of about 20,000 pollutants in the atmosphere. This figure could be improved given the efficiency of this tandem technique, but it is not necessary.

The invention of RS was rewarded with a Nobel Prize, as was NMR spectrometry launched by Bloch's team (Stanford University) and Purcell's team (Harvard University) in 1945.

Initially, the magnetic technique was applied in the protonic system, then other resonant nuclei were utilized, the most important of which was ^{13}C (^{13}C-NMR).

NMR spectrometry yields interesting data on the structure of compounds; for the analytical chemist, it supplies data about the structure of complex combinations.

An interesting variant of NMR is the dynamic NMR spectrometry which supplies information about the conformational equilibria in acyclic systems, configurational inversion, valency tautomerism, etc.

The considerable potential of NMR spectrometry has extended its applications far beyond the limits of analytical chemistry. Today "proton nuclear magnetic resonance imaging has provided a major breakthrough in diagnostic medical imaging and biomedical research. More than 200 instruments are now in use worldwide."[30]

Doubtless, the most fascinating field of structural analysis is that of X-ray diffraction and neutron diffraction. Both techniques supply total information about the structure of samples. The equipment now available rapidly yields useful information, when data are computer processed. Unfortunately, although neutron diffraction produces the most valuable information about the structure of compounds, the technique is far from being generally used because of experimental intricacies.

Modernized X-ray diffraction was utilized with very good results in the characterization of the structure of some complex materials such as polymers, proteins, nucleic acids, etc. In the case of compounds of complex compositions, even the most sophisticated automatic

Downloaded At: 15:18 17 January 2011

equipment, assisted by high performance computers, will not make interpretation of data any easier, therefore this problem is for the future to solve.

The analytical chemist has to be well aware of the basic principles of these techniques to be able to make use of them in the near future.

This concise discussion of the three basic concepts of analytical chemistry suggests the following conclusions:

1. Any analysis should begin with an exact knowledge of the history of the sample.
2. This knowledge ensures the adequate choice of the methods of analysis.
3. The analyst's art consists in adapting the method to the instrument.
4. Teaching and education in analytical chemistry should include, to a balanced extent, quantitative, qualitative, and structural analyses.

IV. CONNECTIONS

In this section, emphasis will be placed on the point that to be educated in the field of analytical chemistry or chemical analysis one should acquire knowledge in collateral fields as well. It is only in this way that one can understand and apply the new techniques of analysis and operate the instrumentation which is ever more sophisticated. The curricula have to balance the topics and ensure the perfect correlation of the various courses.

It is true that this is not easy to accomplish because it requires objectivity in the issuing of teaching programs.

To learn analytical chemistry, one needs the fundamentals of such branches of science as basic chemistry, physics, automatics, mathematics, biology, science of the environment, etc. The implications of the first four items mentioned, which are certainly involved in the understanding of analytical chemistry, will be mentioned.

Previously considered was the octahedron of analytical chemistry;[2] here inorganic chemistry, organic chemistry, and physical chemistry in fundamental chemistry should be included (Figure 2).

To do analytical chemistry, or to develop it along the vertical, assumes possessing a basic knowledge of other fields of science. Therefore, courses taught in chemistry departments have to deal only with those studies, or parts of them, which are helpful in the understanding of chemistry. Sometimes physics or mathematics is taught which have no direct implication in the chemistry of interest.

It is difficult indeed to do this selection because this requires that the professor teaching physics or mathematics in a chemistry department possess dual qualifications: a good knowledge of his own field and at least awareness of the problems of the chemical science in question.

Here are some general examples, and some others inspired by the present author's teaching activity, which are given in order to emphasize the implications of the subject under discussion.

A. Fundamental Chemistry

The knowledge of fundamental chemistry is the most important element in the formation of an analytical chemist. It is a good practice to give Freshmen a concise class of general chemistry. It is useful to set the foundations for further understanding of inorganic, organic, and physical chemistry.

There are very few good handbooks of general chemistry the world over; this is so because it is extremely difficult to gather, in a relatively brief number of pages, the knowledge which is useful in the understanding for all the rest of chemistry.

Downloaded At: 15:18 17 January 2011

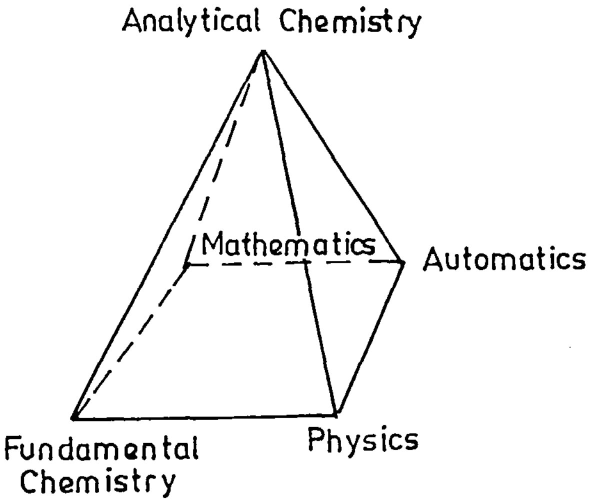


FIGURE 2. Connections of analytical chemistry.

Downloaded At: 15:18 17 January 2011

Fortunately, Romania has a high quality treatise on general chemistry written by a great specialist in organic chemistry, Professor C. D. Nenitescu.[31] The book was first issued in 1949 and has since served to instruct many generations of students; it has been translated into many languages. The study of a specialized domain may begin only after the assimilation of general knowledge.

How can analytical chemistry employ knowledge from other fields of chemistry? This question will be answered, starting from the history and properties of a sample.

Analytical chemistry participates in the discovery and exploitation of new sources of ores by describing the composition of those ores. The analyst should know mineralogy and geochemistry; they are taught as part of classes of general chemistry or as independent classes of mineralogy or geochemistry, which are very useful.

The present trend of teaching chemistry as theoretically as possible may have adverse effects, for instance, by excluding from the topic basic knowledge which is considered too simple.

Playing a musical instrument requires one to know the notes and to be talented. In any human endeavor, talent is fertile from simple to complex matter. Therefore, one has to weigh properly the information in chemistry and for chemistry.

Modern geochemistry enjoys the cooperation of analytical chemistry, which supplies it with the methods of geochemical and hydrogeochemical prospecting. In this respect, it is worth mentioning the importance of geochemistry and the position its relation to analytical chemistry has been awarded in the U.S.S.R. A famous research institute in Moscow is called Vernadskii Institute of Geochemistry and Analytical Chemistry, which is sponsored by the U.S.S.R. Academy of Science. In this institute are conducted remarkable studies of cosmochemistry and analytical cosmochemistry, a top priority of this field of research.

The teaching of inorganic chemistry and its understanding informs the future analytical chemist about the properties of various inorganic materials. Although controversial, a course in descriptive inorganic chemistry, if it is taught through correlations (which is rather difficult), gives the basic concepts about chemical elements and their properties.

To practice any kind of chemistry, one needs to know about the chemical elements which is equivalent to knowing the notes on the score in music. Then one can "play music", i.e., do inorganic or organic synthesis or perform analyses.

A very important field of inorganic chemistry, which contributes substantially to analytical chemistry and chemical analysis, is that of complexation. In a department of chemistry, an independent course on complexation is not the specialization of inorganic chemistry. Such a course should be a general one because the ever wider utilization of organic reagents in practice is equally interesting for students from organic, analytical, and even physical chemistry departments.

The notion of complexation introduced by Werner represents a useful concept which, besides being rewarded with a Nobel Prize, has given momentum to research in all of the chemical sciences. The applications of complexation in the practice of analytical chemistry are very diverse. For example, masking and masking agents and the utilization of complexations in a wide variety of methods of determination can be mentioned.

A distinguished professor from the Chemistry Department of Bucharest University, Gheorghe Spacu, forerunner in the study of complexation, gave a matchless course on this subject. The present author had the priviledge to associate with Professor Spacu for only a short time, yet this proved long enough to be engrossed in his passion for this study. There are indeed great professors, who, like great actors, fascinate and inspire.

The development of analytical chemistry has benefited enormously from inorganic chemistry and the chemistry of complexation.

Organic chemistry has been, and is, in continuous "cooperation" with analytical chemistry. The possibility of directed syntheses of new compounds useful as analytical reagents has ensured the continuity of this interdisciplinary cooperation. Think of the utilization of organic solvents in methods of separation and determinations and of the synthesis of new organic reagents, whose analytical performance is ever more favorable.

This author's long experience with organic reagents shows that there is no domain of chemical analysis which manages without reagents.

It is enough to mention as another example the utilization, at a growing rate, of macrocyclic ligands in analytical chemistry.

Physical chemistry has contributed to the theoretical foundation of analytical chemistry (Ostwald), but it should not be forgotten that the various branches of physical chemistry enjoy an equitable cooperation with analytical chemistry.

The study of atomic structure has resulted in the elaboration of theoretical systems which serve as a foundation for the techniques of chemical and structural analysis with X-ray diffraction and neutron scattering.

Kinetic studies have led to new, highly sensitive and selective methods of analysis: kinetic methods (Yatsimirskii) and enzymatic methods (Guilbault).

Thermodynamic studies have catalyzed the development of thermal analysis, whose methods have brought a substantial contribution to the study of the composition and structure of compounds.

Finally, consider studies in the chemistry of colloids and macromolecular compounds.

The theory of the formation of precipitates is closely related to colloidal chemistry, e.g., ponder the formation of precipitates of silver halides.

Without studies in the field of macromolecular compounds with dedicated properties, there would not exist a wide range of ion exchangers. These hard, soluble, polyelectrolytes find the most diverse applications in the practice of chemical analysis of simple or complex systems. Consider the outstanding performances of extraction chromatography and back-extraction chromatography.

All these facts confirm the idea that doing high-grade chemistry requires, first, a solid knowledge of chemistry, which is then applied creatively.

Downloaded At: 15:18 17 January 2011

B. Physics

The present author includes in physics electronics and does not make it a separate branch of science as some other people consider it. Because of its explosive evolution and its applications in so many domains, it is of key importance.

The physics classes for chemists should be taught such that, besides learning basic theoretical notions, students relaize how some principles apply to given studies of chemistry.

The definition given for the physical methods of analysis in analytical chemistry is "*quod erat demonstrandum*", a recognition of the role of physics in the development of methods of chemical analysis.

It is an understanding of the principle that underlies the method of analysis which ensures the correct operation of the instrumentation.

This analytical instrumentation, which has crossed many stages in its evolution, has been given "a hand" by electronics and automatics to become what it is now, a fast and reliable source of analytical information.

The specialization of future analysts requires the teaching of a course of electronics for chemists. This kind of course has proven its utility. The handbook by Malmstadt and Enke,[32] which now is classic, should be mentioned here.

In Romania, great importance is attached to teaching future chemists some electronics. A book has been published by Cordoş and Marian[33] (the first author worked for some time with Professor Malmstadt) which is useful to students as well as to specialists.

Downloaded At: 15:18 17 January 2011

Regarding the "inoculation" of a knowledge of electronics into analytical chemists, who are sometimes adverse to it, a good job is done by the journal *Analytical Chemistry*. Raymond Dessy publishes excellent "lessons" of electronics in this journal. In this regard, two of these lessons,[34,35] which are excellent, should be mentioned.

These facts show once more that physics has contributed to the growth of new methods of analysis, to the improvement of other methods, and to the optimization of the analytical process. Examples are numerous:

1. The improvement of classical polarographic methods by the use of the oscillograph has resulted in the higher performance technique of oscillopolarography.
2. The utilization of the Zeeman effect to optimize AAS.

The presence of physics in analytical chemistry is large and this is to the benefit of the analyst.

This discussion could not be concluded without mentioning the utilization of the laser in chemical analysis, beginning with emission spectrometry and continuing with RS. This powerful instrument, the laser (Nobel Prize for physics in 1966 to Alfred Kastler), has made possible the creation of new instrumentation and the enrichment of existing instrumentation.

Studies on plasma in various countries (Grigorovici and Truţia in Romania) have led to its adoption in analytical chemistry and to the development of the ICP-AES technique.

C. Automation

Think of the instrumentation of chemical analysis 40 to 50 years ago — only a balance and a buret — then realize how fast analytical chemistry has grown.

The development of the theoretical principles of automation or, in other words, the birth of a new science has obviously been sensed in technology by the creation of more and more automated devices. Chemical analysis has dually benefited from automation, e.g., the creation of semiautomated laboratory equipment (automatic balance included) and automated control systems such as on-line chromatography.

Downloaded At: 15:18 17 January 2011

The adaptation of microprocessors, computer terminals, and high-performance computers has turned automated analysis into an ultrafast process.

Practically all the steps of the analytical process have been automated, except, partially, the sampling of solids. The present equipment is provided with automated sample exchangers, automated measuring and data processing systems, etc.

The most recent trend is the application of robotics to laboratory practice, especially to sampling. In this respect, Dessy, who published two pioneering papers on this subject,[36,37] must be acknowledged.

Several companies have already begun to produce such analytical robots.

It will be shown in the next section on development that automation and robotization are means of improving the analytical control and of eliminating manual opeations. Thus, human errors are eliminated and the analytical system becomes more objective.

However, one should not forget that the automated and robotized systems are man's creations, programed by man, and they will never replace human thinking. Or will they?

Students of advanced study should be taught automation and robotization, but professors have to emphasize that analytical chemistry remains for man to practice and develop. Chemical analysis will be partially or totally automated, but still conducted by man.

D. Mathematics

In a recently published paper, Devlin pointed out: "In 1983 the German mathematician Gerd Faltings put a finite limit on the number of solutions of Fermat's equation."[38] This proves the great power of mathematical research, which may solve practically impossible problems such as Fermat's problem.

Mathematics is present in all science as a factor of the theoretical progress and optimization of some applied systems. Analytical chemistry and chemical analysis are among the beneficiaries of mathematics.

There exists now a field of study, chemometrics (Kowalski), which deals with the optimization of the brut analytical signal and to which the contribution of mathematics is substantial. It includes, e.g., the statistical processing of analytical data or pattern recognition.

The development of computer science was also significantly supported by mathematics.

Mathematics is present in the new laboratory instrumentation, e.g., in transform techniques which serve to obtain multiple analytical information. FT-IR is the result of the perfect cooperation of analytical chemistry, physics, mathematics, and computer science.

It can be concluded that analytical chemistry may develop only through the cooperation with chemistry, physics, automation, mathematics, and, especially, biology and biochemistry.

V. DEVELOPMENT

In writing this section, this author encountered some difficulties because the problems of development in general and the development of science in particular are dealt with by specialized persons and organizations and are hard to approach since they are problems of prospective, of futurology.

Therefore, this section has been written on the basis of some analytical systems which are prominent now and have promising futures: reactions, surface, automation, robotics, and teleanalysis.

This discussion should be restricted by the titles of the sections mentioned. To make it more interesting, and more general, the discussion will focus on three correlated problems: method, instrument, and man. One can notice that this is the third correlation already mentioned. Man (the analytical chemist) is the last in the discussion as the main contributor to the future of analytical instrumentation.

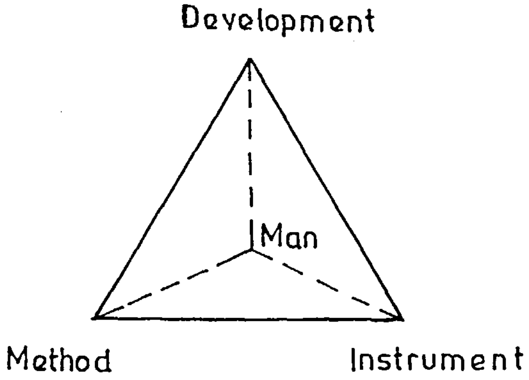


FIGURE 3. Correlation of method-instrument-man and development.

This correlation, method-instrument-man, represents a real force for the development of analytical chemistry and of chemical analysis. This correlation is pictured in Figure 3.

Downloaded At: 15:18 17 January 2011

A. Method

The various definitions of analytical chemistry show that this science deals with the elaboration and improvement of the methods of analysis, including the methods of separation and sampling. Optimization of analytical control implies the development of new methods of separation and analysis and the improvement of the old ones.

Some problems of prospective from the standpoint of the philosophy of analytical chemistry shall be discussed here.

Professor Ron Belcher, who for many years was in charge of the reagent commission of IUPAC, has mentioned several times the importance of the study of chemical reactions and the importance of finding new reagents and methods of analysis. In discussions with this author, he emphasized the primordial importance of these studies for the future development of analytical chemistry. The analyst finds in such studies practically unlimited fields of activity. The art of the analyst consists of studying the chemical phenomena in depth, of selecting the general out of the particular in order to develop competitive and optimized systems of analysis.

It is exactly the study of reactions, of reactivity, and reaction mechanisms which may be helpful in judging the capability, correctness, and creativity of the analyst. To develop new methods of analysis and to improve the existing ones requires creative thinking about the subject in question.

Let us consider sampling. As specified earlier, sampling means mostly separation, and in this field there is much room for improvement, especially through the coupling of several separation methods into a more efficient whole. Returning to two systems of separation which were already mentioned, i.e., extraction chromatography and back-extraction chromatography (the latter term was introduced by the present author's co-worker Pisoschi[39]), both techniques rely on coupling solvent extraction with chromatographic methods, but differ in their principle.

In extraction chromatography, or chromatography of extraction, an inert support (Teflon® or Kel F), an organic stationary phase (which generally includes a complexing agent), and a mobile aqueous phase are employed, while in back-extraction chromatography an active support (a pretreated ion exchanger), aqueous stationary phase, and a mobile organic phase (a product resulted following solvent extraction) are used.

Downloaded At: 15:18 17 January 2011

The first system has the drawback of lower efficiency (lower amounts separated) because of the porous substrate being impregnated with solvent. It was utilized with very good results for the separation of radionuclides. Several years ago, a book was written by Braun and Ghersini[40] about this subject; it was well conceived and retains its freshness. This technique is useful to the analysts who work in radiochemical separations, yielding radionuclides of high purity and competitivity.

The second system, with active support, offers the advantage that the ion exchanger is utilized to full capacity so that the separation efficiency is much higher. In the present author's laboratory, studies began on this system in 1971. Then the present author and colleagues utilized as extraction agents naphtenic acids and various solid ion exchangers and succeeded in separating U (VI) from Th (IV).[41] Afterwards, this author and co-workers tried to improve the efficiency of the separation system. Thus far, the best results were obtained with the mixed action of liquid ion exchangers (as "extraction" agents) and solid ion exchangers.

Pisoschi and Tătaru[39] made use of hydroxyoximes developed by General Mills (Tucson, Ariz.) under the name Lix. For example, Lix 64 N is a mixture of 5,8-diethyl-7-hydroxi-6-dodecanoxime and (2-hydroxy-2-nonyl-phenyl)phenyl-metanon oxime. The results obtained in a concentration of Ni^{2+} are very promising.

Recently, in cooperation with Pisoschi, the present author conducted a new study on back-extraction chromatography which has produced very interesting results regarding the separation and concentration of Ni^{2+}, Cu^{2+}, and Co^{2+}.[42] Back-extraction chromatography may be used with good results at technological levels to obtain products of high purity.

Regarding the separation systems, more attention should be given to studies on synergism. The performances of synergistic extraction are common knowledge. These should be improved and coupled with systems of ionic exchange to obtain highly efficient separations based on back-extraction chromatography.

Let us return now to another technique of separation — ion chromatography. It was originally developed by Small and co-workers in 1975[43] and is applied routinely for the analysis of organic cations and anions, organic acids and amines, transition metals, carbohydrates, and alcohols.[44]

In a recent paper about the chromatographic separations of anions and cations from water samples, it was stated: "This original method used two columns attached in series packed with ion exchange resins to separate the ions of interest and suppress the conductance of the eluant, leaving only the species of interest as the major conducting species in the solution. Once the ions were separated and the eluant suppressed, the solution entered a conductivity cell, where the species of interest were detected."[45] This definition of ionic chromatography was selected because it is very concise and complete. Acting upon the chromatographic column one can substantially improve this technique, which is very promising.

Before approaching the methods of analysis, note that other methods of separation may also be considerably improved and that, in general, the performance of all techniques of separation, either through extraction with solvents or ionic exchange, as well as other chromatographic techniques, will have to be improved.

The methods of analysis have already been classified into destructive and nondestructive. The former consist merely of a study of chemical reactions. It is obvious that the improvement of some methods and the creation of new methods of analysis depend on the research carried out on reactions. To illustrate the importance of this research for chemical analysis, only two types of reactions are mentioned, which were famous in their time and still are. The first reaction is the basis of the method for determination of SO_2 advanced by West and Gaeke.[46] It is characterized by high sensitivity and good selectivity; it is still employed in the determination of SO_2 from polluted atmospheres. Although the mechanism of this reaction has been the subject of numerous studies, it has not been perfectly elucidated.

The second reaction is among the very few for spectrometric determination of F^- ion; it makes use of the absorbance of the ternary complex formed between cerium (III) complex with Alizarin complexan[47] and fluoride ion.[48] The Alizarin complexan reagent was proposed in 1958[47] as a complexometric indicator in the determination of some elements. This reaction evidences the art of the research mentioned, which has come out with a reaction considered practically impossible: a color reaction for fluoride ions. Due to its performance, this reaction is competitive with fluorine determination by means of the ion-selective electrode, using a LaF_3 membrane.

The study of reactions remains a top priority of analytical chemistry and in chemical analysis. Studies on organic reagents, and their reactions with ions and molecules, should be continued. Studies should also be conducted on kinetic reactions, both simple and enzymatic, especially on reactions of chemi- and bioluminiscence. The study of these systems will certainly contribute to the improvement of analytical methods.

One should not forget that more importance should be given to the reaction mechanisms because reactions are successfully employed today in analytical practices whose mechanisms are either partially elucidated or totally unknown.

Partially destructive and nondestructive methods have received considerable attention and have made spectacular progress in the past years. The history of these methods begins, practically, with the use of X-ray fluorescence in analytical practice. This technique is the basis of modern methods, referred to as beam analysis, methods of wide variety. From the standpoint of operational parameters, these culminate with secondary ion mass spectrometry (SIMS), a technique with outstanding applications in analytical practice.

The analyst changes the "reagent" with the beam and is interested in picking up information from the surface of the sample or the layer immediately beneath. In this technique, the analyst has a tremendous instrument for the investigation of samples.

First in importance are the techniques of surface analysis, e.g., electron spectrometry for chemical analysis (ESCA), which utilizes electron beams and total surface analysis. There are also secondary effects, e.g., Auger electrons, which have suggested a new technique of surface analysis — Auger spectrometry, whose foundation was set in 1925 by Auger.

Beam analysis has recently been enriched with new techniques, such as the Raman microprobe, proton microprobe, and synchrotron radiation (one of the most efficient and expensive techniques of surface analysis).

Utilization of surface analytical techniques (beam analysis) has represented immense progress in analytical methodology because it allows qualitative, quantitative, and sometimes structural characterization of a microscopic fraction of samples; this is impossible by destructive techniques.

The importance of surface analyses is underlined by the prestigious journal *Analytical Chemistry* which, after 1982, included surface analysis in its April issue dedicated to fundamental reviews.

Before going to the next section, one of the most interesting achievements in analytical reagents in the last 20 years should be mentioned. This was left to the end of this section to reiterate the idea that the study of reactions is, and will be, of primary importance for chemical analysis and analytical chemistry.

This achievement is the application of macrocyclic compounds to chemical analysis. The dean of analytical chemists, I. M. Kolthoff, remarked "As early as 1939, a few macrocyclic compounds have been prepared by organic chemists, but their complexing properties with cations were not recognized until in the sixties."[49] The pioneering contribution to this field is ascribed to Pedersen.[50]

Studies have been conducted by various schools of chemistry around the world, among which the one in Strasbourg headed by Lehn[51] produced an "explosion" in the synthesis of the compounds in question. The syntheses of macrocyclic compounds continue and special mention is deserved by Japanese researchers.

Downloaded At: 15:18 17 January 2011

The analytical applications of these macrocyclic compounds are diverse, from ion-selective electrodes, reagents for spectrometric determinations, to direct synthesis of such compounds for chromatographic purposes, etc. One can compare the introduction of these compounds in analytical practice with the revolution produced in analysis by the synthesis of new complexans.

The final conclusion of this section — analytical chemistry and chemical analysis have no limits!

B. Instruments

The second part of the correlation presents as much interest as the first (i.e., the method); this section will next examine some ideas regarding the instrumentation that the analyst employs throughout the entire analytical process.

The evolution of instrumentation was similar to that of the methods of analysis. The sample, through its volume and composition, dictates the type of method of analysis and the instrumentation. The analyst employs various instruments in order to obtain information, a signal from the sample, as fast as possible.

As was shown in the final chapter of *Education and Teaching in Analytical Chemistry*,[1] to be useful the analytical signal has to have three qualities: rapidity, reproducibility, and reliability. This section will now examine these same qualities with some examples.

Downloaded At: 15:18 17 January 2011

Rapidity refers to the time interval between the introduction of the sample and the output of the analytical information; it is the shortest time possible. This may be achieved by improving the methods of analysis and the analytical instrumentation. Therefore, there must be agreement between the operational parameters of the method and the functional parameters of the instrument.

Present technology requires performing a large number of analyses in the shortest time, both sequentially and continuously. Therefore, some modifications had to be made in instrumentation. First of all, the analysis equipment was mechanized, automated, and robotized. This was done through the cooperation of analytical chemistry with other fields of study.

As soon as analytical methods were automated, they became partially or totally independent. Since the methods were rapid, they were fit for automated production control. Thus, automated devices for the continuous monitoring of the degree of environmental pollution were manufactured which employed methods whose performances were comparable to those of existing systems.

In the study of water pollution, ion-selective membrane electrodes were employed, while in the study of atmospheric pollution, several automated methods were utilized, e.g., a high sensitivity and selective analysis of carbon monoxide in the atmosphere based on IR nondispersive spectrometry.

A method could be adapted to an automated system of measurement and control only if it met the performance criteria required by the general analytical process.

The cooperation of the various branches of science has resulted in the development of analytical instruments which promptly give a reproducible and reliable signal. The reproducibility of the analytical signal depends both on the method and on the instrument, while the reliability of the analytical signal depends to a greater extent on the instrument.

If the methods of laboratory analysis, as well as those of automated production control, require fulfillment of the three qualities of the analytical signal, this is no less than that required of hyphenated methods of analysis. These latter were developed to give more information in shorter times; this was achieved through the adequate coupling of separation and analysis techniques. The present studies make use of tandem techniques, such as GC-MS, HPLC-MS, CG-FTIR, HPLC-FTIR, TA-GC, etc.

As Hirschfeld very well remarked, "By combining several analytical methods, it is possible to pool their virtues. To start with, as described above, we can combine a high discriminating power in one of the instruments, with a high separation power in the other, to accomplish general qualitative tasks more easily."[52]

It is now possible, from a technical point of view, to couple two methods of analysis as detectors, for one method of separation, e.g., GC-MS-FTIR. Obviously these tandem techniques imply the availability of adequate interface systems.

The separation analysis systems can also be attached to a computer, which processes and refines the signal to make information available more rapidly.

The hyphenated methods are comparable — from the point of view of the volume of information and of its efficiency — with surface analysis; unfortunately, the two techniques compare well with respect to the high cost of instrumentation.

An examination of the evolution of laboratory instrumentation shows that during the last decade it has reached scandalous proportions and the analysts are to blame for this. They studied the techniques of separation in the laboratory and created the technological conditions for obtaining chemicals of extremely high purity. This set the foundation for research on semiconductors and therefore the foundation of electronic concerns such as those in Silicon Valley, California became possible. Thus, microelectronics was born and a new generation of equipment which processes and stores analytical information was conceived. In many branches of science and technology, this equipment was implemented so that it contributed to scientific research and practical applications which will be discussed in the next section.

Next, let us examine an interesting domain of analytical chemistry and chemical analysis — teleanalysis. This field resulted from analytical chemistry.

To be suitable for such a system of analysis, the equipment has to be automatic and be made of reliable parts. This equipment collects samples, performs analyses on the spot, and broadcasts the results at the same time. The teleanalysis systems were improved as a result of the development of cosmochemistry, whose beginnings lie in the American and Soviet lunar and outer space programs. An interesting achievement is the automated analysis, through X-ray fluorescence, of the surface of Venus.[53,54] The technique of analysis and data processing was remarkable. Although the temperature on the surface of Venus was about 450°C, the equipment on both spacecrafts Venera 13 and Venera 14 worked very well, sending to Earth very interesting data on the chemical composition of Venusian rocks.

Samples were picked up with a special drilling device and irradiated in the internal chamber of the lander with radioisotopes. The induced fluorescence was recorded by an energy-dispersive X-ray spectrometer. The perfect preparation of the experiment, to ensure that it would work in the inadequate "climatic" conditions of Venus, was remarkable. This is one of the outstanding achievements of analytical chemistry in recent times.

In an editorial in *Analytical Chemistry* in July 1982, Professor Morrison wrote,[55] "On March 1 and 5 this year, however, Veneras 13 and 14 touched down on the planet and provided the first detailed analysis of Venusian surface rocks. We are particularly indebted to Professor Yu. A. Surkov and his colleagues of the Vernadski Institute of Geochemistry and Analytical Chemistry in Moscow for the cooperation in sharing with our readers the details of one of the more exacting analytical measurements of recent time."

The example above shows the importance of the method-instrument-man correlation and of a knowledge of the sample to be analyzed. It should not be forgotten that the program of this experiment was conceived in the above-mentioned institute which specialized in geochemistry and, more recently, in cosmochemistry.

Now let us deal with the third element of the correlation — man.

C. Man

Without interfering with the discussion in the final section of this paper, the focus here

Downloaded At: 15:18 17 January 2011

will be on the qualities of the researcher in analytical chemistry. Such qualities are very well summarized in the present author's recent book as the three Cs, i.e., capability, correctness, and creativity. Without repeating what was written there, the idea should be stressed and, through some examples, the role of the researcher during the whole analytical process should be emphasized.

It is obvious that to be creative in science one should be both capable and correct. These qualities belong to someone who is intelligent and educated in a given branch of science and are acquired in the process of education of the future researcher.

The professor has to induce into the student a passion for laboratory work and for research. This is best done through personal example. Both in teaching and in laboratory activity, the professor should be a "model" to the student.

The best way of shaping a specialist in a certain field is to work on a research team. The members of a team should be alike in preoccupations and thinking abilities. The team should be able to work under various conditions regarding the quantity and quality of research.

The beginner learns on the team how to program the experiment and how to solve research problems. Regardless of the nature of the research, the beginner should learn how to learn or how to search permanently for the new and be well informed.

The first and most important step of any research is the literature search. The information on the subject in question should always be updated; this has been mentioned elsewhere[1] and, although this tenet is generally accepted, it is not always obeyed. The student should be taught very early how to handle the various systems of information. A prime principle should be: spare no cost as far as information is concerned, regardless of its nature. The most efficient form of information is the direct contact with specialists, either through work in reknown laboratories or participation in scientific meetings.

Downloaded At: 15:18 17 January 2011

Young participants in major international meetings (e.g., of the Euroanalysis type) are scarce, at least in Europe. At the five Euroanalysis Congresses held so far, the participants are generally the same, mostly professors and accomplished researchers. This may be good, but younger scientists, who could learn from their encounters with the noted personalities of science, are absent. The answer is given, on one hand, by the participation fees, which have grown fairly high of late and, on the other hand, by the inadequate support granted to the young scientist by the various chemistry societies.

If these things remain unchanged, there is a risk, at least in Europe, that major congresses (we do not refer to conferences on narrow specialities indeed attended by more young people sent there by various companies) will continue to be attended by a growing number of senior scientists. There are exceptions to these "rules", which evidence the role of a good team of research. Thus, Professor Pungor's team from the Technical University of Budapest is always present at scientific meetings with a good number of young people.

According to information from the literature of the U.S., the participation of young researchers at international meetings is substantial because of good support. This may be sensed if one considers a continuous increase of participants at Pittsburgh-type meetings.

At the 36th Pittsburgh Conference held in New Orleans (February 25 to March 1, 1985), more than 100 technical sessions were scheduled, including 18 planned symposia and more than 1200 papers.[56] Many young scientists were in attendance.

Participation at meetings should be active; this refers not only to the presentation of oral communications or posters, but also to contacts and discussions with specialists. Thus, some international cooperations and joint ventures may be established. A fruitful cooperation has been established in Europe between the research teams of Professor Pungor (Hungary) and Professor Simon (Switzerland).

To do "great" science, means to work on a team and for the team. It is the professor's task to select students from among the Freshman class and to lead them towards the various fields of basic and applied research.

Selection of young people for basic research should be done carefully, with objectivity and scientific maturity. Basic research in general, and in analytical chemistry in particular, can be performed only by teams of intelligent analysts, who put their minds to work. While for applied research capable and correct people can manage, for basic research the third condition — creativity — is compulsory.

Creativity in science requires information; the scientists should not simply reproduce, but, as already indicated, read between the lines, extract ideas from the ideas of others, scrutinize them, and turn them into new research subjects.

It is obvious that not every Bachelor of Science graduate, however well prepared, is able to perform scientific research. The selection should be severe, to seek the perspective researchers who will approach and develop new basic research projects.

Modern society has begun to realize that basic research has played an important role in its evolution. Many years have passed before research has asserted itself as a motivating force of society, through science initially and through technology secondly.

The analyst has to understand the two aspects of analytical chemistry as a science. Thus, either alone or supervised by a professor, one chooses the adequate profession.

Great countries such as the U.S., U.S.S.R., and Japan have invested considerably in basic research; analytical chemistry has been one of the beneficiaries of these investments. The example of Soviet cosmochemistry given above is convincing. All major comtemporary scientific endeavors such as nuclear energy, space, and the environment absorb huge investments for research and development.

Downloaded At: 15:18 17 January 2011

The process of theoretical and practical analytical chemistry has been stimulated by these investments. The nuclear program alone has imposed the development of new methods and adequate equipment. The radiochemical and radiometric methods of analysis were substantially improved. Sampling equipment and instrumentation for semiautomatic or automatic analysis were created. Studies were conducted on separation techniques such as extraction with solvents, ionic exchange, etc.

Space research imposed the development of automated equipment for cosmoanalytical research. A new field of chemical analysis and analytical chemistry — teleanalysis — took shape. These researchers gave momentum to current laboratory practice; some analytical methods were improved and the measure and control devices were miniaturized. The greatest successes of analytical chemistry came from its involvement in space programs; the equipment had to be more reliable and, as a consequence, the analytical information itself gained in reliability.

The few aspects regarding the implication of the analyst in scientific research discussed above suggest that to maintain the status of analytical chemistry a certain type of practical analytical chemist, a certain type of individual who conducts research in analytical chemistry, has to be educated.

In the final part of this section are listed some requirements which should be imposed on future analysts:

1. To know the history of analytical chemistry
2. To be well informed about analytical chemistry and chemical analysis. Information requires a solid knowledge of at least two languages widely used for chemistry, i.e., English and Russian
3. For those involved in fundamental research in analytical chemistry, development should take place along the "horizontal", i.e., widest and deepest knowledge of basic chemistry, physics, automatics, mathematics, etc.
4. For those involved in applied research, the development should occur along the "vertical", i.e., specialization in a narrow field of chemical analysis, e.g., chromatography

or spectrometry, and further on in even narrower domains, e.g., subdomains as GC, HPLC, AAS, etc. (the evolution of modern society requires the training of both groups of specialists)

5. To work with dedication for analytical chemistry and chemical analysis
6. To promote analytical chemistry in order to ensure it a rightful place among the basic and applied branches of science

The aim of this section was to demonstrate the importance of the method-instrument-man correlation. Although the formal ratio is 2:1 in favor of the former sequence of the correlation, it should not be forgotten that both the method and the instrument are man's achievements, designed to favor human activity.

Certainly the future will hold surprises in respect to both methods and instrumentation. Therefore, professors have to educate future specialists to be able to absorb "surprises".

Multilateral and multidisciplinary training is now a basic requirement for future specialists. Specialization and ultraspecialization may then build upon the support of basic knowledge.

Only intelligent and logical thinking can facilitate the progress of analytical chemistry and chemical analysis.

Under these circumstances, the role of the teacher becomes more and more important, but more difficult.

The teacher has to learn to be aware of all that is new in analytical chemistry. Courses will have to be updated and adapted to new "analytical technologies".

All these aspects will be demonstrated in the final two sections, which are shorter, but will discuss some problems related to the applications and aims of analytical chemistry, as well as to their correlation.

Downloaded At: 15:18 17 January 2011

VI. APPLICATIONS

This section relates to the final section — aims — and the two are complimentary.

A short discussion about the applications of analytical chemistry and chemical analysis is practically inconceivable. However, the present author shall attempt in the following to present a discussion of present and future applications of analytical chemistry. These discussions will be split into two parts, i.e., basic research and applied research, which really cannot be clearly separated from each other.

A. Basic Research

It is important first to emphasize, without flattering analytical chemistry, that practically every domain of basic research benefits from analytical chemistry. This author will not trace the contribution of analytical chemistry to every field of research; instead an outline of its enormous impact on the development of scientific research, through some examples selected from the innumerable examples known, shall be undertaken.

Analytical chemistry and chemical analysis have contributed substantially to the development of chemical science in general and its branches, inorganic and organic chemistry, in particular, based upon the synthesis and characterization of compounds.

This discipline of chemistry resorts to methods of quantitative and structural analysis. The characterization of the immense number of synthesis compounds would not be possible without "a hand" from analytical chemistry.

As has already been shown in this paper, inorganic chemistry is present in analytical chemistry through the field of complexation, however, conversely, to the study of complexation chemistry an important contribution is brought by analytical chemistry. Besides establishing the composition of complexes, analytical chemistry, through structural analysis, has contributed, does contribute, and will contribute to the elucidation of the structure of chemical complexes.

NMR spectrometry is one of the techniques of structural analysis which describes the constitution of complexes in general and of those complexes with organic reagents. Utilization of this technique for the study of the structure of inorganic compounds has facilitated the establishment of their structure and contributed to the development of new studies.

From among the numerous articles and reviews on NMR, a recent paper by Fedorov,[57] which updates and discusses the implications of this technique in the chemistry of complex formation with organic reagents, can be mentioned. In that paper are examined the applications of NMR in establishing the structure of reagents and complexes with various types of ligands.

Research on the chemistry of high-purity inorganic compounds has been assisted by analytical chemistry, with methods of high purification and trace analysis. Such substances as silicon, germanium, or GaAs, with very important applications in microelectronics, were tested for their high purity by methods of analysis of high sensitivity and selectivity. Only such techniques as spark MS or ICP-AES could meet the requirements of high-purity analyses.

Thus, the study of ultrapure materials and semiconductors is not possible without chemical analysis.

Research in organic chemistry, which is obviously a speciality based on synthesis, has also felt the effects of the development of analytical chemistry. The study and characterization of simple compounds, or of those whose composition and structure is complex (e.g., polymers), was possible through the utilization of adequate methods and instrumentation.

The progress of theoretical studies in organic chemistry with implications in adjacent fields, such as pharmaceutical chemistry, has created possibilities for establishing correlations between structure and properties. Thus, the controlled synthesis of new compounds, with "predicted" characteristics and properties, has become possible.

The analyst has been carefully watching these studies because his task is to characterize not only the final reaction products but also the intermediate ones as well. One may state that without the development of separation techniques such as solvent extraction and chromatographic techniques, especially GC and HPLC, as well as of techniques of analysis, especially structural ones, organic chemistry would not have reached its present stage.

From the multitude of examples, this author will focus on two to emphasize that controlled organic synthesis results in reagents of high interest in analytical practice. Leitis et al.[58] recently published a review article on analytical reagents derived from pyridine aldehydes. These derivatives exhibit very interesting and promising analytical properties. It suffices to say that these reagents are remarkable for the large number of competitive reactions they can produce and which can be used for the determination of transition metal cations, aminophenols, substituted hydrazines, and other substances.

The second paper, also published recently by Lehn,[59] refers to supramolecular chemistry, controlled synthesis of some macromolecular compounds of very diverse implications. The author, who is a famous specialist in the field, notes, "Macropolycyclic architectures, in principle, meet the requirements. Being large (macro), and highly connected (polycyclic), they are suitable for the construction of molecules containing the cavities, clefts, and pockets that provide the framework for management of binding sites, reactive groups, and bound species."[59]

Thus, a new branch of chemistry was born in which analytical chemistry has invested important capital, from which it now profits. Do not forget the numerous analytical applications of these compounds.

The research of physical chemistry is not, by far, the smallest beneficiary of analytical chemistry. Practically, several measurements of phycial chemistry bear a pronounced ana-

Downloaded At: 15:18 17 January 2011

Downloaded At: 15:18 17 January 2011

lytical character such as the kinetic studies carried out using thermal methods of analysis and the studies of equilibrium through membranes on reaction mechanisms in solution or in flames. Thus, physical chemistry receives useful information from analytical chemistry and, in turn, supplies the theoretical basis of a large number of methods and techniques of analysis.

Analytical chemistry is, and will continue to be, present in biological sciences. It has contributed to the development of some special methods of analysis such as enzymatic analysis, immunochemical analysis, and radioimmunochemical analysis, (radio immunoassay [RIA]). Thus, analytical chemistry, through chemical analysis, has become an instrument that has been playing an increasing role in the development of biological sciences. Biology, and mainly biochemistry, are the legitimate beneficiaries of analytical chemistry. This is most illustratively defined by Warren,[60] "Analytical chemistry and the analytical chemist will play an important role and participate more fully in the area of biotechnology and genetic engineering in the future. The analytical chemist will be working with the biochemist, the molecular biologist, and the process engineer to scale up bench scale synthesis of biomolecules for processing large amounts of product." Further on Warren says, "The demands of modern biology will require the development of a breed of biotechnologically competent analytical chemists. This breed of analytical chemists will not only have to be well grounded in traditional theory and skills, but must also be aware of the basic principles of biology, biochemistry, microbiology, molecular biology, biocatalysis, and the basic engineering process associated with the production of biomolecules."[60]

Indeed, the research in science prefixed by "bio" is fascinating. During this author's undergraduate studies, a very good Romanian specialist in endocrinology — Professor C. I. Parhon — was conducting research on the implication of hormones in the growth of plants. Today, this research has developed considerably, and the contribution of analytical chemistry is substantial; special techniques of analysis were required for this study. As Davis et al. remark,[61] "Plant hormone research has paralleled the historical development of chemical methods of analysis. Although many phytohormones have been identified, routine analyses of these molecules continue to challenge even the most modern chemical and immunological methods." and further on, "The marriage of current methods of chemical analysis with immunosorbents and immunoassays promises to extend detection limits and our understanding of phytohormones' roles in plant growth and development."[61]

All these considerations, to this author's satisfaction, prove the role of analytical chemistry and chemical analysis in some top fields of basic scientific research. The applications are practically unlimited. Next, the applications of analytical chemistry in some applied research will be examined.

B. Applied Research

This is practically an unlimited field and, therefore, discussion is restricted to three applications of vital importance for human life, i.e., the quality of the environment, the quality of food, and the "quality" of man, which comes to the analyses of the environment, of food, and clinical analysis, respectively. In all these domains, analytical chemistry has made an outstanding contribution. This discussion will begin with the analysis of the environment, in which remarkable progress was achieved with respect to methods, instrumentation, and automation of equipment. The analysis of environmental purity implies a programing of experiments according to the special nature of the samples to be analyzed. When the present author mentions the environment, he is generally referring to atmosphere, water, and soil. While the analyses of soil and soil pollution are not attached, sampling problems, in atmosphere and water-analyses sampling, is difficult (one has to deal with unique samples). The diffusion of atmospheric components and the flow of liquids cause the composition of samples to change rapidly. Therefore, in the case of unique samples, the devising of automatic, continuous systems of analysis was necessary to characterize the pollutants of waters and air. Analytical chemistry has solved these problems by selecting the methods and improving the equipment.

Unfortunately, while the techniques developed are quite good from a sensitivity view point, there is still much to do to improve their selectivity. There are, for example, electrometric methods adaptable to the automatic and continuous analysis of water and air whose selectivity is poor. Consider the instrumental methods for the electrometric determination of SO_2, which offer all the advantages except selectivity. The method mentioned, advanced by West and Gaeke,[46] has been utilized with good results for the determination of the concentration of SO_2 in the atmosphere.

To a certain extent, things are similar with the selectivity of electrochemical sensors used for the pollutants of water. Most sensors, except the pH and LaF_3 electrodes, do not yield adequate signals and reliable analytical information because of their lack of selectivity.

Analytical chemistry may be useful in another way in studies regarding the pollution of the atmosphere, i.e., by finding the precise location of potentially polluting industrial plants. Studies have been carried out of atmospheric diffusion; radioactive or fluorescent (fluoresceine) tracers are released from a fixed point. The chemical analysis of diffused pollutants at various distances along different directions indicates the precise location of the pollutants.

Analytical chemistry has also contributed to studies regarding the background pollution of the atmosphere; for this study, samples are collected through automatic sampling devices placed in zones of minimum pollution.

It is worth noting that analytical studies regarding the quality of the environment play an educational role because the automated instrumentation displays quantitative data in public locations. This is the case of automatic analyzers of CO in the atmosphere; these analyzers are based on the nondispersive infrared technique. Through these analyzers, the community at large can find out about the degree of atmospheric pollution and act to lower it.

Studies on environmental pollution have resulted in the elimination of some dangerously noxious pollution sources. Thus, the large number of analyses carried out by AAS have revealed the extent of lead pollution in cities; this is due, to a great extent, to the use of tetraethyl lead as an additive to improve the octane rating of fuel. Now, in several countries utilization of this very noxious substance is forbidden.

Last, but not least, analytical chemistry has contributed through its studies on pollution of the environment to an improvement in the technologies of some products. The so-called ''nonpollutting technologies'' were created, which will certainly increase in number in the future.

Now consider a brief examination of the role of chemical analysis in the characterization of food. The permanent increase in the level of pollution, on one hand, and of the amount of food produced, on the other, has imposed continuous quality controls on food. This control consists of determining the content of inorganic or organic toxic substances which are introduced either in production or by accident. Specialized laboratories are now supplied with competitive equipment which routinely perform such analyses.

There are a good deal of potentially noxious substances in food, of which the most dangerous are metallic or organometallic substances such as Pb, Hg, organic derivatives of mercury, and insecticides such as DDT and organophosphoric or organothiophosphoric pesticides.

Analytical research in food technology has introduced methods of separation and analysis of these dangerous substances in food. Thus, AA on one side and chromatographic techniques on the other have contributed to the characterization of the quality of food. Some foodstuffs of more complex composition, such as wine or coffee, required the use of high-performance separation techniques and analysis. This is the case in GC which utilizes capillary columns

Downloaded At: 15:18 17 January 2011

to separate with efficiency flavor components. The chromatograms of such products are very complicated (hundreds of peaks) and their interpretation may be done only with an improved system in tandem with a MS or FT-IR detector.

Finally, the third field of analytical application is clinical analysis. This is the most efficient role of analytical chemistry because it refers to the "quality of man".

Clinical chemistry today employs excellent analytical methods, for example, there are automated systems which carry out in a very short time the analysis of the various components of blood.

The methodology of clinical analysis is in a permanent state of improvement; it is enough to mention, as an example, the introduction of enzyme immunoassay techniques (enzyme-linked immunosorbent assay [ELISA] or enzyme-multiplied-immunoassay technique [EMIT]) of high sensitivity, whose performance compares with RIA.

Analytical chemistry has successfully been used in the study of intoxication and diseases such as diabetes. Analytical studies have been conducted on intoxication with alcohol, and adequate methods of research and analysis were introduced. An interesting paper on blood alcohol concentration (BAC) was published recently.[62]

In the case of diabetes, research is being done on the general metabolic system and glucose metabolism in particular. Chemical analysis has contributed substantially to the diagnosis and treatment of this disease as well as of other illnesses. A remarkable achievement is the introduction of the RIA technique (Yalow and Berson, 1960). A bird's eye view of analytical implications in diabetes was published by Free and Free,[63] who note, among other things, "The contributions of analytical chemistry to understanding and providing effective treatment of diabetes are greater with the disease than any other disorder of mankind." This is obviously taking into account the competition between various techniques of analysis to find the diagnosis and treatment of this sickness of the civilized world.

Dealing with the applications of analytical chemistry and chemical analysis, the present author referred to basic and applied fields, which are related to the "protection" of man. However, chemistry, through chemical analysis, is involved in the development of methods of investigation and control of goods made by man and sometimes in the protection of these goods (criminalogical research).

Research in analytical chemistry has resulted in methods of analysis (very often standardized) for natural products and synthetic products employed in various modern technologies.

Without approaching the field of industrial production, or more correctly the control of the quality of the industrial production, this paper will examine some instances in which analytical chemistry was implemented through chemical analysis.

Analytical chemistry possesses today methods useful for research in archeology (utilization of ^{14}C radioactive tracers) or for the authentication of works of art (trace analysis).

Methods have been devised for the detection of drug abuse in athletes; trace analysis and chromatographic separation are the methods employed in this application.

So far, competitive methods were described for the characrterization of some raw materials through research in analytical geochemistry or analytical hydrogeochemistry.

Several modern technologies have made use of the continuous automatic control offered by analytical chemistry, e.g., large steel mills, which are controlled continuously by automatic emission or fluorescence X-ray spectrometers come to mind.

It is hoped that the facts presented thus far prove that through its numerous methods and techniques of analysis analytical chemistry has asserted itself in modern society as a well-defined science which contributes to the improvement of the quality of human life.

In the final section, it will be shown that the "circle closes". That is, we learn analytical chemistry and conduct research in analytical chemistry in order to optimize the process of production of material goods. The optimization of this process contributes to the improvement of the conditions of education and research. Therefore the title of the final section is Aims.

Downloaded At: 15:18 17 January 2011

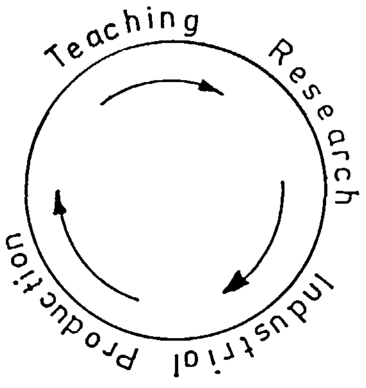


FIGURE 4. The great correlation: teaching-research-industrial production.

VII. AIMS

Downloaded At: 15:18 17 January 2011

This final section refers to the important correlation of teaching and research and industrial production which forms a closed circle (Figure 4) with clockwise circulation.

Let us "launch" some original ideas about this correlation. There is one paper which approaches the problem of the future of analytical chemistry and predicts its prospectives as a science and the prospectives of chemical analysis as an applied branch.

The author of that paper has stated something which is important, "We cannot continue to assign the responsibility for science education to those who are scientifically uneducated."[64]

In analytical chemistry, as in any branch of science, one has to start with teaching — efficient teaching makes research and industrial production possible.

A. Teaching

Opinions about teaching analytical chemistry, extracted from the literature, are numerous. The present author will now put all of them aside and on the basis of over 30 years of experience in higher education will discuss some aspects of teaching analytical chemistry. Here reference is made to teaching analytical chemistry in a chemistry department because in other departments (metallurgy, pharmacy, medicine, biology, etc.) its relative importance is different.

From the very beginning, one must consider that analytical chemistry is an independent chemical specialization with its own rules. Therefore, in chemistry departments it should be represented by a separate chair. To associate it with inorganic or physical chemistry — as in some countries of Europe — is a mistake. It is true that analytical chemistry interacts with the specialities mentioned, but it should remain an independent field. To make this possible, its advertising should make use of all means to specify its object, aim, and prospectives.

The teaching of analytical chemistry is different in various departments and, therefore, the specialists should be offered opportunities to discuss and reach agreement on how to teach it (not uniformity). Therefore, the initiative of the European Working Party on Analytical Chemistry in convening from time to time and discussions on the Education of Analytical Chemistry are welcome because they help those involved to improve teaching and education in the field in question.

Assume the question arises: what should one teach in classes of analytical chemistry? The answer was contained in the section on concepts. Analytical chemistry should be taught starting from quality, continuing with quantity, and then to structure. This is the logical sequence of teaching it. The time shared by each of the three concepts should be well balanced. This requires that the members of the teaching team be well educated in analytical chemistry and chemical analysis and, especially, be objective. Only an objective scrutiny of the subject and object of analytical chemistry may result in unified, integrated, and interesting classes.

The professors' council in chemistry departments should ensure the teaching of chemical and nonchemical courses, such that information interpenetrates from the various classes and forms a unified whole.

Thus, every independent speciality may profit from the information supplied by the others. Moreover, discussions on the teaching of specialists in various domains may lead to establishing interdisciplinary cooperation along the logical sequence of the circle.

Formation of a cultured analyst requires the teaching of the "alphabet", which means teaching of qualitative analysis. In such classes, the student — future specialist — is informed about the basic laws of chemistry, about the properties and behavior of compounds, about reactions and their mechanisms. The study of reactions in the solid state and solution make up the major subjects of qualitative analysis. No matter how perfect the laboratory instrumentation, teaching qualitative analysis should continue because it is the basis of the understanding of the other branches of analytical chemistry. It should be taught as a basic course for one semester (first semester, 1st year of study), with a lecture-to-laboratory ratio of 2:6 weekly. This share of time is necessary and sufficient for learning qualitative chemistry. Although all methods of analysis assume a qualitative aspect, teaching and laboratories in qualitative analysis should center around the study of the basic elements of analytical chemistry, with the study of reactions taking the greatest share.

The assistant plays an important role in the qualitative analytical chemistry laboratory. He teaches the student how to program the experiment and to choose the most efficient way to identify compounds in a mixture. As an undergraduate, this author had attended a laboratory of qualitative chemical analysis supervised by well-prepared and passionate assistants and had the already mentioned feeling[3] that chemical analysis was an "art", which only a well-prepared specialist may practice.

As soon as the basic knowledge of qualitative analysis is taught, quantitative analysis normally follows. Although this is controversial, at the beginning of this course (and not at the end), the notions of sampling should be taught. The notions of sampling should include the general characterization of samples with respect to their history, processing, and techniques of separation.

This course should mirror the relative importance of sampling in the analytical process.

The classes should follow those of qualitative analysis in the second semester of the 1st year of study; the ratio of lecture to laboratory should be 2:6 weekly.

There will be objections, such as, how can one teach separation before methods of analysis. The problem is indeed disputable, but the solution is left to the logic of teaching analytical chemistry. To do quantitative analysis on some samples of complex composition, one should utilize one or more methods of separation. To check the efficiency of a separation method one may choose for the students' laboratory other methods which are very simple. Thus, one takes a step forward to quantitative analysis.

The course of quantitative analysis should be taught for all Sophomores for two semesters, with a lecture-to-laboratory ratio of 2:6. Handbooks of quantitative analytical chemistry should be issued (unfortunately there are very few at hand) which, in a relatively small volume (maximum 500 pages), have critical discussions of all the important areas of analytical chemistry. Repetitions should be avoided, e.g., the calculation of titration curves in volumetry.

The teaching of quantitative analytical chemistry is the "art" of the team whose members

Downloaded At: 15:18 17 January 2011

have to select the general out of the particular in order to balance theoretical and applied aspects.

In the quantitative analysis laboratory, experiments should advance from simple to complex. The student should understand that the instrument picks up a signal from an experimental program, which, in many instances, is part of some equipment. Intricate analyses on ores or organic compounds in mixtures, which imply a first step of separation, should not be avoided. Thus, the student gets accustomed to complex systems of analysis, which are the object of chemico-analytical production control.

It has been shown that structural analysis is the "concert master" of teaching and research in analytical chemistry. The course of structural analysis is at present programed in many chemistry departments, either in chairs of physical chemistry (most often) or in chairs of organic chemistry (fairly often).

This course should be a general one. It should be taught to Seniors in the second semester. This proposal is made because to understand and apply the techniuqes of structural analysis one needs a knowledge of physics and physical chemistry, and this knowledge, as well as that of mathematics, is taught before the course in question.

The course of structural analysis should include 2 hr of classes and 6 hr of laboratory weekly; its topic should include both basic knowledge and its applications.

Supervised by assistants well trained in analytical chemistry, the students have to elucidate the structure of some compounds of both simple or more complex structure on the basis of diverse methods of structural analysis.

The course and laboratory of structural analysis, like that of quantitative chemical analysis, should be accompanied by data processing (chemometrics).

This is simply a proposal regarding the teaching of basic courses of analytical chemistry in chemistry departments; it is obviously open for discussion.

The specialization and postuniversity courses of analytical chemistry should have the topics decided by the teams that teach them. The choice of courses, and their number, are related to the requirements of specialists in analytical chemistry and to the specialization of the team in the field in question.

Courses designed to update knowledge should be held at various time intervals which depend on the moral aging of the analyst.

Some characteristics of the process of teaching in analytical chemistry and chemical analysis have been listed and next some "philosophical" aspects of research shall be considered.

Downloaded At: 15:18 17 January 2011

B. Research

This section is practically a replacement for conclusions, thus it concentrates on a discussion about the present and future of research in analytical chemistry.

The selection of researchers for science in general, and analytical chemistry in particular, should be rigorous.

The research of analytical chemistry will be worthwhile if it approaches the basic aspects of the field and is conducted by interdisciplinary teams, which ensure the design of future analytical instrumentation.

In the method-instrument-man correlation, man is the decisive factor. The researcher should be granted an adequate climate in order to be productive. The researcher should be granted conditions to develop, to learn, and to work. The team is important in this respect.

The high-performance research in analytical chemistry is, and will be, effected only by those who are trained for research in the university. No effort should be spared in providing university laboratories with the newest equipment. In some universities, there still exists the concept that higher education is only formative and the student should learn the basis of science with relatively modest instrumentation. This concept introduces a substantial gap between the quality and quantity of research equipment in universities and in research laboratories (or even laboratories in industry which can afford the high-performance equipment).

We have to spread the idea that higher education is, besides formative, creative. The gifted student, supervised by an experienced faculty, has to learn the basics of research and contribute to them while a undergraduate. The students who specialize in analytical chemistry have to be selected rigorously to get them enrolled in the most efficient school of research — graduate studies.

Specialization through graduate studies is a higher stage of the formative process of the Bachelor of Science, which ensures the development of creative abilities. To safeguard the future of analytical chemistry as a science, an ever-increasing number of young people should be involved in this activity. The graduate research work, supported by a team, centers around the individual's initiative, imagination, and intelligence as a graduate student.

In order that the specialization through graduate studies be useful, the curriculum should be carefully selected. To develop the student's passion for scientific research, this curriculum must include subjects with pronounced basic character. Thus, the researcher is formed in the process of research and may bring useful contributions.

This paper has referred many times to teams and their role in successful research. Several research teams working in the same department may form a school of research, pooling their significant results. In the case of the present author, two such schools of research have been influential. These are known in Europe and other parts of the world: Spacu's school of chemistry of complexation and Neniţescu's school of organic synthesis. High-quality Ph.D. studies could be conducted in the atmosphere of such a school.

Thus, logic dictates discussing tradition in scientific research. It requires that members of a research school try to raise their qualifications continuously. The obtaining of a Ph.D. is only one step in the formation of a researcher. This degree should be obtained by a large number of analysts.

The Soviet Union's efforts to develop analytical chemistry bears mention here. To obtain the D.Sc. (they call it "Doctor"), one conducts sustained research throughout a long time interval after obtaining the Ph.D., the first degree (they call it "Candidate in Sciences"). The title of "Doctor" in the Soviet Union is a guarantee that the holder has performed a large amount of high-grade research.

In conclusion, far-reaching research in chemistry will continue to be performed provided that it is ensured the instrumentation and people needed.

And now the third and last aspect of this section — industrial production.

C. Industrial Production

Teaching in analytical chemistry is supposed to not only form researchers but also ensure industrial productivity with the personnel required for an efficient control of quality.

Quality control, and its optimization in view of the optimization of production itself, may be achieved through the permanent cooperation of the analyst and the control system.

The trend to automation and robotization of industrial production has dictated the use of highly automated measure and control equipment. Think of the pH-meters and on-line chromatographs which operate in continuous flux to ensure the control of production, the automatic setting and optimization of the systems controlled.

One may ask, what is the analyst doing in this case when the systems are all automatic and even robotic? The analysts who work in industry have to get ahead of themselves and become, besides analysts as such (in some cases less sophisticated methods are used), system supervisers. To do this, they should be well versed in automated systems of measure and control, be able to service them in case of damage, and be able to think of new improved systems.

Downloaded At: 15:18 17 January 2011

The analysts who work in quality control have to think and prepare continuously to shift the man-instrument correlation to their favor.

It is well known that some industrial accidents in the chemical industry were due to damage in the systems of measure and control. The analysts who work in industry bear the responsibility of ensuring the reliability of measure and control equipment. This is the only way they can obtain reliable analytical data, which in turn ensure optimized quality inspection.

This paper could not be concluded without specifying once more that its aim was not to list the updated literature in analytical chemistry. The present author has tried to express some personal points of view about education in analytical chemistry, resorting sometimes to data in the literature.

Thus, this paper is the result. This author feels it is useful as it offers a basis of discussion about this branch of science — analytical chemistry.

Downloaded At: 15:18 17 January 2011

REFERENCES

1. **Baiulescu, G. E., Pătroescu, C., and Chalmers, R. A.,** *Education and Teaching in Analytical Chemistry,* Ellis Horwood, Chichester, England, 1982.
2. **Baiulescu, G. E.,** Moral ageing of analytical methods, *Analyst,* 105, 1045, 1980.
3. **Baiulescu, G. E.,** Analytical chemistry: science or art, plenary lecture presented on the occasion of the 4th Centenary of the Univ. of Zaragoza (Spain), October 1983.
4. **Baiulescu, G. E.,** Sampling, in *Collaborative Interlaboratory Studies in Chemical Analysis,* Egan, H. and West, T. S., Eds., Pergamon Press, Oxford, 1982.
5. **Baiulescu, G. E., Mircea, D., and Zugrăvescu, P. Gh.,** Consideraţii asupra probei de analizat, *Rev. Chim.,* 35, 1023, 1984.
6. **Baiulescu, G. E. and Ciucu, A.,** Concepts in analytical chemistry, 1st Int. Symp. Hist. and Philos. of Anal. Chem., Vienna, November 22—23, 1985.
7. **Pungor, E. and Kellner, R.,** Education of analytical chemistry in Europe, 1983/84, Fed. Eur. Chem. Soc., Working Party on Anal. Chem., OGMAC, 1985.
8. **Szabadváry, F.,** *History of Analytical Chemistry,* Pergamon Press, Oxford, 1966, 16.
9. **Szabadváry, F.,** *History of Analytical Chemistry,* Pergamon Press, Oxford, 1966, 6.
10. **Kallman, S.,** Analytical chemistry of the precious metals, interdependence of classical and instrumental methods, *Anal. Chem.,* 56, 1020A, 1984.
11. **Szabadváry, F.,** *History of Analytical Chemistry,* Pergamon Press, Oxford, 1966, 172.
12. **Popa, G., Negoiu, D., and Baiulescu, G. E.,** On functional analytical group for palladium, *Zh. Anal. Khim.,* 14, 322, 1959.
13. **Baiulescu, G. E., Greff, C., and Dăneţ, F.,** Contribution to the study of functional analytical groups in the determination of palladium(II), *Analyst,* 94, 354, 1969.
14. **Pérez-Bustamante, J. A. and Burriel-Martí, F.,** "Palladiazo": a new selective metallochromic reagent for palladium. I. The main characteristics of the pure reagent and its reaction with palladium (II), *Anal. Chim. Acta,* 37, 49, 1967.
15. **Khalifa, M. E. M., Stoicescu, D., and Baiulescu, G. E.,** Pallatriazo-A sensitive and selective reagent for the spectrometric determination of palladium (II), *Mikrochim. Acta,* 169, 1984.
16. **Baiulescu, G. E., Moldoveanu, S., and West, T. S.,** Nicolae Teclu (1839—1916), a pioneer of flame spectroscopy, *Talanta,* 30, 135, 1983.
17. **Smith, R. and James, G. V.,** The sampling of bulk materials, *Anal. Sci. Monogr.,* 8, 1981.
18. **Goldschmidt, B.,** *Le Complex Atomique, Histoire Politique de l'Énergie Nucléaire,* Librairie Arthème Fayard, 1980.
19. **Baiulescu, G. E. and Coşofreţ, V. V.,** *Applications of Ion-Selective Membrane Electrodes in Organic Analysis,* Ellis Horwood, Chichester, England, 1977.
20. **Baiulescu, G. E., Kandemir, G., Ionescu, M. S., and Cristescu, C.,** Determination of sulpha-drugs with ion-selective membrane electrodes-II, *Talanta,* 32, 295, 1985.

21. **Ionescu, M. S., Abrutis, A. A., Rădulescu, N., Baiulescu, G. E., and Coşofreţ, V. V.,** Lidocaine-selective membrane electrodes and their applications in pharmaceutical analysis, *Analyst,* 110, 929, 1985.
22. **Welcher, F. J. and Boschmann, E.,** *Organic Reagents for Copper,* Robert E. Krieger Publ., Huntington, New York, 1979, 217.
23. **Hieftje, G. M.,** The two sides of analytical chemistry, *Anal. Chem.,* 57, 256A, 1985.
24. **Vydra, F., Stulik, K., and Juláková, E.,** *Electrochemical Stripping Analysis,* Ellis Horwood, Chichester, England, 1976.
25. **Ewing, G. W. and Van Swaay, M.,** The teaching of chemical instrumentation, *Anal. Chem.,* 57, 385A, 1985.
26. **Balaban, A. T., Banciu, M., and Pogany, I. I.,** *Aplicaţii ale Metodelor Fizice în Chimia Organică,* Editura Stiinţifică şi Enciclopedică, Bucharest, 1983.
27. **Anon.,** Instrumentation needs of academic departments of chemistry. A survey study, *Anal. Chem.,* 56, 1225A, 1984.
28. **Griffiths, P. R.,** *Chemical Infrared Fourier Transform Spectroscopy,* Wiley Interscience, New York, 1975.
29. **Freeman, S. K.,** *Applications of Laser Raman Spectroscopy,* Wiley Interscience, New York, 1974.
30. **Smith, S. L.,** Nuclear magnetic resonance imaging, *Anal. Chem.,* 57, 595A, 1985.
31. **Neniţescu, C. D.,** *Chimie Generală,* Editura Didactică şi Pedagogică, Bucharest, 1979.
32. **Malmstadt, H. V. and Enke, C. G.,** *Digital Electronics for Scientists,* Benjamin, New York, 1969.
33. **Cordoş, E. and Marian, I.,** *Electronică Pentru Chimişti,* Editura Stiinţifică şi Enciclopedică, Bucharest, 1978.
34. **Dessy, R. E.,** The electronic toolbox. I, *Anal. Chem.,* 57, 1188A, 1985.
35. **Dessy, R. E.,** The electronic toolbox. II, *Anal. Chem.,* 57, 1298A, 1985.
36. **Dessy, R.,** Robots in the laboratory. I, *Anal. Chem.,* 55, 1100A, 1983.
37. **Dessy, R.,** Robots in the laboratory. II, *Anal. Chem.,* 55, 1232A, 1983.
38. **Devlin, K.,** The golden age of mathematics, *New Sci.,* 1452, 30, 1985.
39. **Pisoschi, A. and Tătaru, S.,** Concentration and separation of Ni^{2+}, Cu^{2+} and CO^{2+} by back extraction chromatography using LIX 64N and cationic resins. I. Concentration of Ni^{2+}, *J. Chromatogr.,* 201, 297, 1980.
40. **Braun, T. and Ghersini, G.,** *Extraction Chromatography,* Akademiai Kiado, Budapest, 1975.
41. **Popa, Gr., Baiulescu, G., Ciurea, I. C., Lazăr, C., Ioniţă, E., and Popescu, M.,** Proc. 3rd Natl. Conf. on Anal. Chem., Braşov, Romania, September 22—26, 1971, 99.
42. **Baiulescu, G. E. and Pisoschi, A.,** Concentration and separation of Ni^{2+}, Cu^{2+} and Co^{2+} by back extraction chromatography using LIX 64N and cationic resins, *Anal. Lett.,* 18, 1027, 1985.
43. **Small, H., Stevens, T. S., and Bauman, V. C.,** Novel ion exchange chromatographic method using conductimetric detection, *Anal. Chem.,* 47, 1801, 1975.
44. **Smith, F. C., Jr. and Chang, R. C.,** *The Practice of Ion Chromatography,* Wiley Interscience, New York, 1983.
45. **Jones, V. K. and Tarter, J. G.,** Simultaneous analysis of anions and cations in water samples using ion chromatography, *Int. Lab.,* p. 36, 1985.
46. **West, P. W. and Gaeke, G. C.,** Fixation of sulfur dioxide as disulfitomercurate (II) and subsequent colorimetric estimation, *Anal. Chem.,* 28, 1816, 1956.
47. **Belcher, R., Leonard, M. A., and West, T. S.,** The preparation and analytical properties of NN-di (carboxymethyl)-aminoethyl derivatives of some hydroxyantraquinones, *J. Chem. Soc.,* p. 2390, 1958.
48. **Belcher, R., Leonard, M. A., and West, T. S.,** A new spot test for the detection of fluoride ion, *Talanta,* 2, 92, 1959.
49. **Kolthoff, I. M.,** Application of macrocyclic compounds in chemical analysis, *Anal. Chem.,* 51, 1R, 1979.
50. **Pedersen, C. J.,** Cyclic polyethers and their complexes with metal salts, *J. Am. Chem. Soc.,* 89, 7071, 1967.
51. **Dietrich, B., Lehn, J. M., and Sauvage, J. P.,** Diaza-polyoxa-macrocyclic and macrobicyclic compounds, *Tetrahedron Lett.,* p. 2885, 1969.
52. **Hirschfeld, T.,** The hy-phen-ated methods, *Anal. Chem.,* 52, 297A, 1980.
53. **Surkov, Yu. A., Socheglov, O. P., Moskalyeva, L. P., Kirichenko, V. S., Dudin, A. D., Gimadov, V. L., Kurochkin, S. S., and Rasputny, V. N.,** X-ray fluorescence analysis of Venus' rocks by space probes — Venera-13 and Venera-14, *Zh. Anal. Chim.,* 37, 1349, 1982.
54. **Surkov, Yu. A., Socheglov, O. P., Moskalyeva, L. P., Kirichenko, V. S., Dudin, A. D., Gimadov, V. L., Kurochkin, S. S., and Rasputny, V. N.,** X-ray fluorescence spectrometry on the surface of Venus, *Anal. Chem.,* 54, 957A, 1982.
55. **Morrison, G. H.,** Planetary analysis, *Anal. Chem.,* 54, 1249, 1982.
56. Pittsburgh Conference, New Orleans, February 25—March 1, 1985, *Anal. Chem.,* 57, 134A, 1985.
57. **Fedorov, L. A.,** Nuclear magnetic resonance spectroscopy of organic analytical reagents and their metal complexes, *Zh. Anal. Khim.,* 40, 5, 1985.

Downloaded At: 15:18 17 January 2011

58. **Leitis, L. J., Skolmeistere, R. A., Rubina, K. I., Jansone, D. P., and Shimanskaya, M. V.,** Analytical reagents derived from pyridine aldehydes, *Zh. Anal. Khim.*, 40, 386, 1985.
59. **Lehn, J. M.,** Supramolecular chemistry: receptors, catalysts, and carriers, *Science*, 227, 849, 1985.
60. **Warren, D. C.,** New frontiers in membrane technology and chromatography: applications for biotechnology, *Anal. Chem.*, 56, 1529A, 1984.
61. **Davis, G. C., Hein, M. B., Neely, B. C., Sharp, C. R., and Carnes, M. G.,** Strategies for the determination of plant hormones, *Anal. Chem.*, 57, 639A, 1985.
62. **Hume, D. N. and Fitzgerald, E. F.,** Chemical tests for intoxication: what do the numbers really mean?, *Anal. Chem.*, 57, 877A, 1985.
63. **Free, H. M. and Free, A. H.,** Analytical chemistry in the conquest of diabetes, *Anal. Chem.*, 56, 664A, 1984.
64. **Isenhour, T. L.,** The future of analytical chemistry: will there be one?, *Anal. Chem.*, 55, 824A, 1983.

Downloaded At: 15:18 17 January 2011