Československá spektroskopická společnost

Bulletin

25

1977

The Czechoslovak Spectroscopic Society, one of the scientific societies of the Czechoslovak Academy of Sciences, has at present 982 members, working in 290 institutes and enterprises.

The Society covers in its two divisions (Atomic spectroscopy and Molecular spectroscopy) all of the main areas of spectroscopy. The Society's activity is organized in a number of Working Groups (see annual reports below) and four Subcommittees (for nomenclature, for reference materials, for education and for instrumentation). The Society holds its Plenary Sessions once a year, the Executive Committee, elected for a period of three years, meets twice a year. The Society's daily life is run by the Presidium of the Executive Committee, that meets monthly or in shorter periods when necessary. The members of the Presidium are :

RNDr.J.Kuba, CSc	- president
Doc.Ing.E.Plško, DrSc	- vice-president
Ing.A.Švehla, CSc	- vice-president
RNDr.I.Rubeška, CSc	- secretary for science
RNDr.B.Moldan, CSc	- executive secretary
Ing.Z.Kosina	- treasurer
RNDr.A.Nová-Špačková, CSc	- secretary for foreign relations
Doc.Ing.J.Lego, CSc	- secretary for Subcommittees
RNDr.J.Mráz	- chairman-atomic division
Ing.J.Moravec, CSc	- chairman-molecular division
Doc.Dr.D.Papoušek, DrSc	

The main means of communication of the Society is the quarterly Bulletin, publishing organizational news, a digest of selected papers from the Society's meetings and other practical information, e.g. reviews of books on spectroscopy, recommended nomenclature, instruments for sale, etc. On the occasion of the XX.CSI and 7th ICAS a special edition of the Bulletin is herewith published in English in order to give our guests a brief idea of the Society's life. The briefs from the 1976 Annual Reports of the Atomic and Molecular Divisions are given followed by information on several of the main spectroscopic laboratories in Czechoslovakia.

The 1976 Annual Report of the Division of Atomic Spectroscopy.

The Division has organized two general sessions, twelve meetings of specialized Working Groups (W.G.) and one Subcommittee meeting.

The 21st Working Session with 98 participants was held in České Budějovice. The lecturers, including Dr.Krasnobayeva from Bulgaria, dealt with recent trends in analytical spectroscopy. One of the lectures was devoted to developments in excitation sources. Information on activities of foreign Spectroscopic Societies and on the preparation of the XX. CSI and 7.ICAS were also presented.

A Course of Applied Spectroscopy for 46 participants was organized. Applied research was part of the final examination.

W.G. for spectroscopy of metals, led by O.Staňková, organized a joint meeting together with the W.G.s for automated spectrometry and laser microanalysis.

W.G. for spectroscopy of nonconducting materials, led by K.Flórián, held a four-day Seminary. 40 participants including Hungarian and Polish spectroscopists listened to 29 lectures dealing with problems of trace analysis.

W.G. for AAS and flame spectroscopy, chaired by I.Rubeška, organized a Seminary on plasma discharges. 11 papers were presented including one by prof. de Galan from the Netherlands. 65 spectroscopists took part. A second Working Meeting was devoted to applications of AAS in petrochemistry. 14 papers dealing both with flame and electrothermal methods were presented to 58 participants.

W.G. for automated spectroscopy, managed by K.Kuboň, coorganized a training course in automated spectroscopy with 11 lecturers for 35 students.

W.G. for X-ray spectrometry, chaired by J.Waňková, organized a training course of automated X-ray spectrometry. 35 students and 13 lecturers took part.

W.G. for laser microanalysis is led by V.Jánošíková. A meeting on applications of a laser microprobe to analysis in geology attended by 15 spectroscopists was held.

W.G. for X-ray microanalysis, chaired by F.Štorek, organized a seminary on energy dispersive X-ray analysers (system EDAX) attended by 115 specialists. A further Working Meeting with 6 lecturers and 52 participants was also held.

W.G. for instrumental radianalytical methods, led by M.Vobecký, organized a Seminary "IAA 76" dedicated to improvements in detection limits and elimination of interferences in activation analysis. 20 papers were presented to 40 participants.

The Subcommittee for reference materials, led by K.Bičovský, organized a seminary on production certification and utilization of reference materials in Atomic Spectroscopy. 58 specialists from various W.G.'s listened to 20 lectures.

The 1976 Annual Report of the Division of Molecular Spectroscopy

The Working Groups (W.G.) of the Division have organized ten scientific meetings.

The most important event was organized by the W.G. for high-resolution molecular spectroscopy, chaired by D.Papoušek; this was The IV.International Seminary on high-resolution infrared spectroscopy held at Liblice, September 6-10. 101 participants from 14 countries took part.

The Seminary was focused on the laser, sub-mm, and microwave spectroscopies as well as on the problems of the spherical gyroscope molecules, Fourier spectroscopy, the theory of vibration-rotational spectra, molecules with high vibrational amplitudes, and potential functions. In all, 62 lectures were presented.

In a paper published in European Spectroscopy News J.T.Hougen has compared this Seminary to the outstanding Gordon Conferences, a flattering remark, indeed. Only two Conferences of this kind will be regularly organized in Europe; one in France (Tours) and one in Czechoslovakia, in odd and even years, respectively.

The W.G. for vibrational spectroscopy, managed by P.Klíma, organized a meeting on analytical applications of infrared spectroscopy and a second meeting on the use of computers for treating and evaluating.

The W.G. for Mössbauer spectroscopy, led by J.Sitek has arranged two meetings, aimed at practical application of this method. Though the members of this group are not numerous (about 15 persons usually take part in the sessions), its work has good results and its activity is highly appreciated.

The W.G. of magnetic resonance spectroscopy, chaired by J.Komenda, organized a Seminary on the NMR of solids and EPR Seminary on free-radical mechanisms in combustion processes and other problems solved by the EPR methods in the CSSR. In general about 30-50 members participate at the meetings.

The W.G. on electron spectroscopy and photochemistry, led by M.Nepraš, is the smallest in the Molecular Division but its activity can be successfully compared with those of the other working groups. Two meetings on photolytical effects, photochromism, and luminiscence spectroscopy techniques were held.

A personal change occured in the leader position of the W.G. for the solid state spectroscopy. A.Vaško, who had led this group for many years, has resigned and the group is now being chaired by M.Závětová. A meeting on spectroscopic and photochemical properties of calomel and new polarizer types was held. About 50 specialists took part.

The W.G. for mass spectrometry, led by Z.Herman, has about 50 members. Currently it is being reorganized to include not only the problems concerning organic chemistry but also isotopic composition and inorganic analysis.

X-ray fluorescence spectroscopic laboratory
Research Institute of Inorganic Chemistry (VÚAnCh)
Ústí nad Labem
(Member of Chemopetrol, Concern for Chemical Industry and Oil Processing, Praha)

The laboratory was established as a part of the X-ray diffraction group in 1963. The laboratory is equipped with a Philips PW 1410 vacuum X-ray spectrograph.

The laboratory has been developing new analytical procedures for intermediates and products of various processing lines. X-ray fluorescence analysis, as a rapid and accurate analytical method, is used for both research purposes and for routine analysis in manufacturing plants.

The main applications of X-ray fluorescence analysis in the VÚAnCh are as follows :

a) Development of analytical methods for the determination of biogenic elements in new types of phosphatic fertilisers and in agricultural plant materials.

When evaluating the efficiency of new types of fertilizers, the essential question is that of the capability of plants to accept the biogenic elements added into the soil by a fertilizer. A large number of samples must be treated owing to the great number of factors influencing this complex process. The conventionally used methods of chemical analysis do not meet these requirements mainly because they are tedious and time-consuming. On the other hand, the X-ray fluorescence method is very suitable for these purposes.

A method for the quantitative determination of the macroelements P, K, Ca, S, Cl and the microelements Mn, Zn and Cu in plant materials has been developed. The original material is pulverized without any further chemical treatment. The calibration curve method, based on a series of standards analyzed accurately by chemical methods, is used for the evaluation of the content of all the 8 elements to be determined. The method of scattered X-ray is used for the correction of the matrix effect. An accuracy of $\bar{\sigma}$ = 0.7 - 1.8% rel. has been achieved for the macroelements. An accuracy for the microelements at a concentration range of 10^{-3} - 10^{-4}% was 5 - 7% rel. The method is used for series analyses of plant materials.

b) Development of methods for the evaluation of the thickness of thin surface layers.

When dealing with the problems of increasing the service life of activated titanium anodes in electrolytic cells, it is necessary to study the thickness and homogeneity of the ruthenium layer on the anode surfaces. The X-ray secondary fluorescence method cannot be applied for this purpose because of the size of the anodes. With respect to this fact, a method based on the use of X-ray fluorescent radiation excited by a radionuclide combined with energy dispersive X-ray fluorescence, has been developed.

The suggested method uses ^{109}Cd (activity = 3 mCi) as the primary source. This radionuclide emits X-ray radiation $AgK\alpha$ at 22.2 keV, which brings about a very efficient excitation of X-ray radiation $RuK\alpha$ at 19.3 keV in the RuO_2 layer. The $RuK\alpha$ radiation is detected by a semiconductor counter Ge(Li) or by a scintillation counter. A Mo filter is used in the measurements. Model anodes, whose Ru layer thickness has been determined by weighing, are used as standards. The method is applicable for the measurement of Ru surface layers thicknesses within the range of 0 - 15 g/m^2.

Since the Ti anodes are made in the form of a wire grid, a special adaptor for the scintillation counter head has been designed.

All the instruments for this method are portable (single-channel set Tesla NZQ 714 T coupled with an NKQ 321 scintillation counter) and, therefore, can be used for field measurements.

The VÚAnCh X-ray fluorescence laboratory also provides expert training and consulting services. The head of this laboratory is the chairman of the Working Group on X-ray Fluorescence Methods of the CSSS. The Working Group aims at increasing the qualification of Czechoslovak specialists active in this field, e.g. by organising expert meetings and training courses.

Spectroscopic laboratories
Research Institute of Fine Chemicals (VÚČCH)
Brno

Research Institute of Fine Chemicals - VÚČCH of the Lachema national enterprise in Brno, which is part of the UNICHEM group in Pardubice, is the main Czechoslovak specialized institute for the research and development of laboratory and special chemicals. A glance at the product selection of the Lachema Works covering among other items ultra pure inorganic compounds, luminophores, organic reagents, biochemicals, sets for clinical diagnoses, materials for gas and liquid chromatography etc., shows to what an extent it is necessary to apply a highly qualified analytical control. Naturally it is necessary to use all standard analytical methods, the most important one being spectroscopy.

Absorption spectroscopy in the UV and visible range.

In recent years, in connection with the development and production of organic reagents, close attention has been directed to general problems of the photometric study of the origin of relatively dissociated complexes (determination of the complex compositions and of their basic parameters) and their use in analytical chemistry. Similarly the properties of the reagents TAMP, TRAMP, TEAMP and MAGON have been studied as well. Nowadays the spectrophotometric analytical methods are being developed for the production of diagnistic Bio-La-Test sets. The Lachema Works produce at present 35 different sets, all with photometric evaluation, most of them patented.

NMR analysis.

All intermediates and products of organic syntheses are analyzed by means of NMR spectroscopy not only for their structures but also for purity. Moreover, this method is used for studying some of the kinetic problems and as to polymers used in our research institute it was applied to the determination of mean molecular weights. These purely practical studies in the case of series of similar substances make a basis for profound investigations of different properties, e.g. conformational analysis, transfer of the substituent effects etc. Results are published in special papers. A relatively large range of organic compounds studied in this way enables the elaboration of different measuring methods. Results of this research are utilized in the TESLA Works in Brno where Czechoslovak NMR spectrometers are produced. In this field VÚČCH has been in a long and fruitful collaboration with the TESLA Works.

Atomic spectroscopy.

Atomic spectroscopy is employed particularly in the trace analysis of impurities in developed and produced special reagents, raw materials and intermediates. Emission technique (powders and to a minor extent solutions) is used in the identification analyses, in controlling the purity of some extra pure substances when minute contents of several elements should be determined simultaneously and finally when analyzing elements for which no discharge tubes with hollow cathodes for atomic absorption exist or when the particular element is difficult to be solubilized (Si). In the sixties a technique of the direct spectrographic determination of metallic impurities in organic chemicals without preceding mineralization was developed.

Facilities for atomic absorption spectrophotometry have been used in our laboratories since 1969. This recent method has proved useful owing to its rapidity and sensitivity and today cannot be disregarded not only in analyses for research purposes but it was introduced also in standards for the quality control of several products. In addition to routine analyses our chemists investigate some methodology problems of atomic absorption spectrophotometry, especially those of thermoelectric atomization in graphitic furnaces (HGA-74, PERKIN-ELMER).

Lectures and practical training courses are organized or co-organized widely, especially in the NMR field. The students appreciate the possibility of direct measuring of the spectra in the NMR laboratory of the institute. Growing demands for the development and production of new fine and special reagents and for their quality are the guarantee of the future development of spectroscopic methods in the laboratories of VÚČCH of the LACHEMA national enterprise.

Spectroscopic laboratories
Research Institute ČKD PRAHA (VÚ ČKD)
Praha

The Institute solves the scientific and technological problems of one of the biggest and most advanced concerns of the engineering Czechoslovak.

A wide range of research cannot be imagined without a thorough application of modern spectroscopy.

Optical emission spectrometry.

VÚ ČKD was in fact a pioneer Institute in the country in the field of metallurgical analysis.

In the mid - fifties a handy and precise steeloscope was developed and manufactured in a series sufficient to cover completely the needs of the country. Together with the manufacture of the instrument the laboratory was a leading factor in the methods of application. Visual spectroscopy research has not been abandoned till today ; the research of the quantification of this unfairly underestimated branch of spectroscopy still proceeds in the emission spectroscopic laboratory.

VÚ ČKD was also among the first in the country in automated spectrometry - quantometry. From the first instrument, a Cameca sequential spectrometer in 1957 up to the two ARL 310 000 spectrometers today the scientific research and routine application of metal analysis has formed an important part of the Institute's activity. The thorough study of matrix effects in steels, irons, aluminum and copper alloys is perhaps the most important.

A further step towards modern industrial application of spectrometry is computer controlled production with an on-line automated spectrometer. VÚ ČKD is the first institute to solve this problem in the country's metallurgy - the result will be computer- quantometer controlled casting of steels and nodular irons.

Reference materials.

Suitable reference materials are doubtless a necessity for any automated spectrometry. A lack of these led to the first attempts to produce them on VÚ ČKD grounds already in the fifties. Today, after wide and tedious research of concept, technology, homogeneity and certification analyses of monolitic metal reference materials, the Institute supplies reference materials to 35 countries. To mention one significant example - the set of cast irons cannot be matched as far as the universality and the size of the usable part of the samples are concerned.

Atomic absorption spectrometry.

Though rather a new field (a PERKIN-ELMER 300 Since 1973), AAS gained in VÚ ČKD an important role, both in reference materials analysis an in the solution of special tasks, like the determination of wear metals in oil, water waste pollutants etc. A routine versatility of the method has been systematically enhanced, e.g. a method for sample dissolving, extraction and AAS measuring in one flask has been developed.

Scanning Electron Microscopy and X-ray Microanalysis.

The science of metallurgy has been enhanced by the introduction of scanning electron microscopy and X-ray microanalysis, both opening new possibilities in the research of metals by overcoming disadvantages and limitations set up by former equipments.

The Electron Microscope Laboratory of VÚ ČKD utilizes since 1973 a JXCA-50A computer-controlled X-ray microanalyzer with an Image Analyzer, all from JEOL (Japan). This equipment enables the laboratory to cover most of the requirements of the ČKD-works both in the field of research and routine service. As an example of research programs solved, the laboratory deals with stainless steels, aluminum-silicon alloys and different types of cast irons. However, routine service includes a variety of materials, even non-metallic, making the analyst a versatile research worker. The laboratory is capable of making scanning electron micrographs, computer-controlled high speed qualitative and quantitative analysis of flat specimens with wavelength spectrometers and quantitative metallography using the Image Analyzer.

In the near future it is expected that an energy-dispersive spectrometer will be added to the system to speed up preliminary analysis and to enable, at least qualitatively, the performation of the X-ray microanalysis of rough surfaces such as fracture surfaces.

Wide national and international co-operation helps VÚ ČKD to keep pace with up-to-date spectroscopic development. As a member of the COMECON committees with a number of direct co-operations with foreign institutes, the spectroscopic laboratories of VÚ ČKD solve their problems efficiently, with sufficient information.

The spectroscopists of VÚ ČKD played an important role in the founding of the Czechoslovak Spectroscopic Society and still carry out a good deal of its activities. The Institute itself organizes a number of training courses and symposiums, helping thus the popularization of spectroscopy and, consequently, its proper role in the Nation's economy.

Spectrographic laboratory
A.S.Popov Research Institute of Radiocommunications
(Tesla-VÚST)
Praha

The development of electronics, and, especially, of semiconductors, requires a whole range of special high purity materials. Therefore materials introduced into the technological processes demand the determination of a whole range of impurities, which effect the electrical parameters of both devices and complete electronic equipment.

Among a number of methods used for the evaluation of these materials, spectroscopy is doubtless the most important one.

In the early fifties a spectrographic laboratory was founded in the Tesla - VÚST to back the research work on the preparation of high purity materials. The first spectrograph used in the laboratory was a Soviet ISP 22, which was later complemented by a Soviet KS 55 prismatic spectrograph. In later stages the equipment was extended by a Q 24 spectrograph and a

PGS 2 grating spectrograph from Zeiss, GDR. An HFO 1 spark source, an ABR 1 generator of an alternating disconnected arc and, especially, a generator of a direct arc with the range of 1,5 to 100 A were used as excitation sources.

The spectrographic laboratory determines many trace impurities in various matrices, e.g. semiconductor raw materials (sythesized polycrystalline and monocrystalline materials - Ge, Si, GaAs, InSb, etc.), or some high purity metals and non-metals used in semiconductor and instrument production technology (Pb, Bi, Sn, Cd, Ta, Se, Ag, Au, S, Te). Trace impurities were also determined in various chemicals (acids, hydroxides, inorganic salts). For the production of capacitors and resistors various powder metals (Ta, Nb, Ti), alloy metals and oxides for evaporation (Al, Ta, NiCr, SiO, TiO, cermets, etc.) were inspected, using spectrography. As far as the research of oxid ferromagnetics of spinel and granate structure is concerned, methods for the determination of impurities both in basic raw materials and in final products were developed. The raw materials as Fe_2O_3, NiO, MgO, MnO, ZnO, BaO, Y_2O_3, Li_2O, Ga_2O_3 were analyzed most frequently.

Trace impurities are usually determined by direct emission spectroscopy in open air or in inert atmosphere. For a quantitative evaluation both reference materials and standard solutions are used. To obtain a higher sensitivity of direct spectroscopic analysis, methods for preliminary concentration of traces by a wet process were developed. Thus it was possible to achieve, after the matrix has been removed, detection limits for some metal impurities of the order of 0,01 ppm.

The reasons why spectrographic analysis plays such an important part among chemical and physico-chemical methods used for the evaluation of the material purity lie in a relatively high sensitivity (especially for metal impurities), in the rapidity and in the possibility to determine a whole range of elements simultaneously from one sample, in many cases available in minimum quantity.

The spectrographic laboratory at Tesla - VÚST introduces the developed methods into Tesla enterprises and partially provides analyses for them.

Tesla - VÚST also takes part in the scientific cooperation between COMECON countries, mainly in the unification and standardization of methods for the evaluation of materials. The preparation of these materials is a subject of The Coordination Centre for the research of semiconductor materials and high purity metals.

Spectroscopic laboratories of the Iron and Steel Research Institute (VÚHŽ) Dobrá (Frýdek-Místek)

VÚHŽ is a central research base in the iron and steel industry. VÚHŽ has established co-operation with a number of Czechoslovak specialized research institutes and universities in the application of spectroscopic methods in metallurgical research. A direct co-operation exists also with the COMECON's Permanent Commission on the Peaceful Use of Atomic Energy, the theme being the application of radiometric methods.

The laboratories of VÚHŽ work both in research and applications of various spectroscopic techniques.

Atomic absorption spectrometry (AAS).

The laboratory of AAS has been equipped with a Varian-Techtron M 1000 since 1971 and with an M 63 electrothermal Carbon Rod Atomizer since 1975. This laboratory has to meet the current requirements for the determination of Cr, Ni, Mn, Cu, Ca, Mg and Al in steels.

Systematic research has been directed towards the analysis of trace elements in steels, stimulated by the development of steels for the nuclear industry and microalloyed steels.

A rapid determination of very low contents of Pb, Sb, Bi and As in various types of steels has been satisfactorily solved by flameless atomization, as well as of Cu, Zn and Pb in Mn and of Cr in low-carbon ferroalloys.

In the near future systematic research will be aimed at the development of the determination of the other elements in steels and isolates of nonmetallic inclusions in those cases, where an increase in the sensitivity and accuracy of the determination or time saving is required.

Optical emission spectrometry.

The VÚHŽ laboratory of optical emission spectroscopy was built in 1973 and houses a Q 24 UV-Spectrograph from Zeiss, GDR. This laboratory meets the requirements for the microanalytical determination of isolates of oxidic inclusions and selected types of carbides. A solution method with a rotating graphite electrode is applied for the determination of SiO_2, Al_2O_3, FeO, Cr_2O_3, MnO, CaO, MgO, TiO_2 and CuO in oxides and Cr, Nb, V and Mo in carbides.

During the next years systematic research will be devoted to the increase of the reliability of the results and to the extension of the methods to sulphide inclusions.

Neutron activition analysis.

In 1969 the laboratory for neutron activation was built at VÚHŽ and equipped with a Na-2 neutron generator and a NTA-512 channel analyzer. The nondestructive method for the determination of oxygen in steels has been checked and later extended to other metals. Silicon and chromium have been determined in high-silicon and chromium steels.

The method has also been successfully applied to the analysis of non-metalic inclusions, giving valuable additional information on their origin and formation.

Mössbauer spectroscopy.

Although VÚHŽ has no Mössbauer spectroscopic laboratory, it has taken part in the joint research with the Faculty of Nuclear Physics of the Technical University in Bratislava. The problems, connected with the production of transformer steel sheets, included the development of methods for the determination of the occupation of coordinated shells with silicon atoms in the Fe Si phase and for the study of the surface stresses, arising as a consequence of the heat resisting insulation of oriented electrical sheets. The application of the Mössbauer effect in this field makes it possible to compare various kinds

of insulation and their effect on the final electrotechnical properties of the products.

Another contribution of this work lies in the possibility of introducing Mössbauer spectroscopy to VÚHŽ and its application for the solution of specific problems in specialized laboratories in the country.

Spectroscopic laboratory
National Research Institute for Materials (SVÚM)
Foundry Research Division
Brno

The Brno Division of the SVÚM is the main Czechoslovak foundry research centre. Its spectroscopic laboratory, with 25 years' experience, plays an important part in the solution of problems of cast metals.

Optical emission spectroscopy.

In 1953 an ISP 22 Soviet quartz spectrograph was installed and served for the determination of metal elements in cast alloys up to a content of 5%. Lack of suitable reference materials soon led to the development of a number of the solution methods, using the rotating disc electrode.

The installation of a FES-1 Soviet photoelectric steelometer in 1962 was a further step towards a rapid and versatile spectral analysis. The instrument, a glass-optics simple one-element quantometer with rapid change to another selected element, determined a number of elements in cast irons and steels, foundry copper and aluminum alloys, high alloyed steels and even foundry slags. The SVÚM laboratory developed methods and working manuals for this instrument and introduced it to the industrial laboratories.

The third phase started in 1967, when a PGS-2, high resolution grating spectrograph from Zeiss, GDR with an ABR 3 arc generator made it possible to determine allmost all trace elements in iron.

Laser local microanalysis.

A LMA - 1 laser microanalyzer from Zeiss, GDR, recently combined with the giant pulse source (Q-switch) opened a new field in the SVÚM Brno branch research work. There have been developed methods for the qualitative and quantitative micro-analysis of individual structural phases. These methods made it possible to analyse particles over 2 um, thus being suitable for coarse-grained inhomogeneous materials such as e.g. various types of cast irons, foundry aluminum and copper alloys, and for the qualitative and quantitative analyses of non-metallic inclusions in cast irons.

The SVÚM Brno spectroscopic laboratory closely co-operates with specialized laboratories in Czechoslovakia and with the laboratories in the foundry research institutes of the COMECON countries.

Department of Analytical Chemistry
J.E.Purkyně - University
Brno

The Department is associated with 9 sections in Czechoslovak Universities and Technical Universities. Co-operation on some problems of metallurgical analysis, multicomponent trace analysis and analysis of pollutants in water and air, takes place with a number of institutions. Scientific contacts are established with a large number of co-operators at Universities abroad, especially at the Mining University Freiberg, GDR, K.Marx-University Leipzig, GDR, M.V.Lomonosow-University Moscow, USSR and T.G.Shevtchenko-University Kiew, USSR.

The Department aims its effort at the instrumentation and methodology of molecular and atomic absorption spectroscopy, emission spectroscopy in high-temperature flames and conventional sources.

Molecular spectroscopy in the UV and visible range.

The parameters of metal-organic reagent complexes have

been studied, using computerized methods. A number of polyphenols, azo - and trifenylmethane - dyes etc. have been investigated in this way, both in aqueous and non - aqueous media. The criteria were set up for the selection of reagents, optimizing of procedures and evaluation methods.

Atomic absorption spectroscopy.

The basic physico-chemical properties of the method have been studied, e.g. the hyperfine structure of resonance lines, nebulization and atomization efficiences, using a computer. The application of the Zeeman effect for selective modulation of absorption lines in a strong alternating magnetic field made it possible to lower the detection limits and eliminate non-selective background absorption. The influence of organic acids (sulphosalicylic, chromotropic etc.) on the increase of atomisation efficiency has been the topic of another interesting research work. The flame and flameless (graphite rod or tube) AAS helped to solve various sophisticated analytical and physical-chemical problems.

Emission spectroscopy.

The hot flame (laminar N_2O - acetylene) has been studied as a an excitation source for atomic emission. The internal standard method and computerized evaluation allowed a determination of a number of elements in widely variable matrices.

Both theory and practice of spectroscopy are taught in courses, managed by the Department. During the last five years 35 papers have been published by the Department's members. Plasma emission sources and special branches of electron spectroscopy are the themes of the most important research projects in the near future.

Spectroscopic laboratories
Geological Survey of Czechoslovakia (ÚÚG)
Praha

The spectroscopic work at ÚÚG started in 1947, when a large Hilger spectrograph was installed. At that time emission spectrographic methods were rather rudimentary and only slowly achieved the required accuracy and precision. When the Central Geological Bureau incorporating all geological institutions in the country was established, the laboratory was entrusted with methodical developments of spectrochemical applications in geology. A course on qualitative and quantitative emission spectroscopy was organized by the spectroscopists of the Geological Survey.

Later X-ray fluorescence spectrometry for trace element determination in rocks and isotope mass spectrometry have been introduced. The latter is nowadays used for Pb and K-Ar dating as well as for the study of oxygen and sulphur isotopes in geological materials.

Atomic absorption spectroscopy was introduced as early as 1963, thus pioneering on a national scale. In 1967 the ÚÚG sponsored the 1st International Conference on Atomic Absorption Spectroscopy thus starting the series of ICAS conferences.

The scope of spectroscopic techniques has recently been extended by acquiring an electron probe microanalyzer.

The laboratory has participated in several international projects on the establishment of analytical data and certification of geochemical reference materials, contributing data by optical emission, X-ray fluorescence and atomic absorption spectrometry methods.

The laboratory also offers training facilities in the above techniques for spectroscopists from geological surveys and similar institutions from developing countries. This is realized either by direct cooperation or by assisting in UNESCO sponsored postgraduate courses on modern analytical methods (UNALCO) or on geological exploration (GEOCHIM).

Spectroscopic laboratories
Institute of Organic Chemistry and Biochemistry
Czechoslovak Academy of Sciences
Praha

The Institute of Organic Chemistry and Biochemistry of the Czechoslovak Academy of Sciences, is one of the institutes established after World War II and incorporated into the system of scientific centres of the Czechoslovak Academy of Sciences immediately after the foundation of the Academy in 1952. As suggested by the name of the Institute, the main topics of its interest are organic chemistry and biochemistry. The attention is particularly focussed on investigations of structure, chemical properties and synthesis of components of the living matter. The spectroscopy is regarded in the Institute as a methodic tool; for this reason, the theory of spectroscopy has been no object of a special research.

In the very beginnings of the Institute, modern physical methods have been introduced, inter alia the spectroscopic methods. From these methods, the first attention has been paid to the ultraviolet spectroscopy as an analytical method. The infrared spectroscopy has been then successfully applied to elucidation of structures of the naturally occurring substances. Later on, it proved advisable to transfer some investigations, particularly those on the theoretical background of infrared spectroscopy, into the present Jaroslav Heyrovský Institute of Physical Chemistry and Electrochemistry (Czechoslovak Academy of Sciences). Concerning the nuclear magnetic resonance, the Institute gave a pulse (1958-1960) to the construction of an original Czechoslovak apparatus for the measurement of ^{1}H-NMR spectra. The apparatus was made at the Institute of Instrumental Analytical Chemistry (Czechoslovak Academy of Sciences) in Brno, and then, on an industrial scale, in the Tesla Works. In recent years, the application of NMR to structural problems of organic chemistry and biochemistry has undergone a phenomenal growth in this Institute.

The Laboratory of Mass Spectrometry was founded in 1962 under the auspices of this Institute and the Institute of Physical Chemistry and Electrochemistry (Czechoslovak Academy of Sciences). At the present time, the Laboratory is a part of this Institute and collaborates with numerous other institutes in Czechoslovakia.

The application of chiroptical methods to structural problems of organic chemistry and biochemistry began in this Institute in 1960 with a construction of a primitive apparatus. Since 1965, the Department has used a spectropolarimeter for the measurement of optical rotatory dispersion, since 1971 an apparatus for the measurement of circular dichroism. The Department offers collaboration with other research institutions.

The above mentioned spectroscopic methods, which were applied for the first time in Czechoslovakia in this Institute, are used in determinations of the structure of naturally occurring substances and of compounds obtained by chemical procedures. In the recent time, the spectroscopic methods have been combined with chemical methods and the semiempirical methods of quantum chemistry, depending on the nature of the particular problem. In the early beginnings of the Institute, the infrared spectroscopy played an important role in investigations on sesquiterpenes. The IR spectra of the particular hydrocarbon skeletons and derivatives were measured, published, and used in structural determinations not only in this Institute but also in many research centres abroad, until the NMR method was developed. Thus, the IR spectroscopy proved helpful in investigations on the conformation of six-membered rings, polycyclic compounds, and medium-sized rings and in investigations on hydrogen bonds. Recently, the IR spectroscopy has been applied to compounds with an amide group (lactams, amides, and peptides) in order to examine the nonplanar nature of the amide group in some types of these compounds. The data obtained by IR spectroscopy are compared with those resulting from measurements of the chiroptical behaviour. Furthermore, the chiroptical methods are used in this Institute in investigations on numerous optically active naturally occurring substances, particularly nucleosides and the oxo or lactone derivatives of isoprenoids.

The Laboratory of Mass Spectrometry participates in investigations on various nitrogen-containing components of nucleic acids, peptides, and alkaloids. Remarkable results were obtained with the rhoeadine- and isopavine-tape alkaloids from the family Papaveraceae. Of a special interest are structures of some insect pheromones as inferred from measurements and analysis of mass spectra.

The Laboratory of Nuclear Magnetic Resonance helps to elucidate the structure of naturally occurring substances, for example terpenes and related compounds. The NMR investigators in our Institute try to reduce the need of additional chemical transformations to a minimum. Some specific reactions such as acylations with trichloracetyl isocyanate or the thermally initiated Cope rearrangements are performed in situ in cells.

The fruitful symbiosis of spectroscopic and chemical methods could be illustrated by elucidation of the structure of exotoxin from Bacillus thuringiensis in this Institute. This exotoxin contains a purine base, three monosaccharide units connected by an anomalous ether bond, and a residue of phosphoric acid. The proposal of the exotoxine structure was confirmed by a total synthesis.

From more than 3000 communications published by this Institute up to 1975, about 1500 papers contain spectral characteristics of the isolated or synthesized compounds and in about 20% of papers, the spectral data are reported and discussed in detail. The contribution of spectroscopy to elucidation of structural problems of organic chemistry and biochemistry in this Institute is thus enormous, not speaking about the fruitful personal contacts between spectroscopists and chemists.

It is hardly possible to propose the future trends of this Institute without taking into account a close collaboration between spectroscopists and organic chemists or biochemists both in theoretical problems and experimental praxis. Such a collaboration makes possible a qualified proposal, synthesis, and measurement of the most suitable model compounds in numerous theoretical questions. In this Institute, such an approach has been successfully used in systematical investigations on the nonplanar architecture of the amide (peptide) bonds as significant structural units of proteins and peptides. On the basis of the above mentioned collaboration and with the use of modern apparatus and modern spectral methods, higher forms of spectroscopic analysis could be attempted, for example structural analysis of the whole systems instead of isolated components.

Spectroscopic laboratories
Institute of Solid State Physics (ÚFPL ČSAV)
Czechoslovak Academy of Sciences
Praha

The spectroscopic methods have been largely developed in the Institute of Solid State Physics namely in the field of X-ray spectroscopy, optical spectroscopy and electron-paramagnetic resonance. A cooperation has been established in this field with research centres for solid state physics in USSR, Bulgaria and GDR (Institute of Physics, Moscow, Physico-Technical Institute, Leningrad, both Academy of Sciences of the USSR, Moscow State University, with the Institute of Physics, Tartu-Estonian Academy of Sciences, Institute of Solid State Physics of the Bulgarian Academy of Sciences, Sofia, Central Institute of Electron-Physics, Academy of Sciences of GDR, Berlin).

In the field of X-ray spectroscopy, the Institute continues the traditions of the Spectroscopic Institute (1922) of the Charles University and the school of Prof. V. Dolejšek.

Since the beginning of the fifties attention has been paid to the rapid analysis of steels and ores. A rapid quantitative analysis of germanium in coal has been developed and later, in cooperation with SONP Kladno, an X-ray quantometer was constructed for the steel analysis. For the defect topography of crystal structures both older methods and newly developed ones were used in solving methodical problems of X-ray spectroscopy and for measurements on single crystals of semiconductors. A high resolution was reached by several double-crystal spectrometers with perfect germanium single crystals. A detailed theory of these devices was elaborated and the dynamical theory of diffraction was confirmed by means of a selfconstructed three-crystal spectrometer.

At the present time X-ray emission and absorption bands are studied in $A^{N}B^{8-N}$ semiconductors possessing sphalerite structure both experimentally and theoretically. Further the phase sestructure of crystalline and amorphous materials is studied and the density of the latter is determined by an original X-ray method. The study of X-ray absorption spectra under high pressu- is being prepared.

The results of X-ray analysis development were important for the national economy. The quantometer was constructed in the time when no similar device existed in Europe. (Prize of the Czechoslovak Academy of Sciences in 1964). The reward of the Czechoslovak Academy was awarded to the analysis of tungsten steels during melting. In 1976 the Academy Prize was given for the development of method with high resolutions.

Optical spectroscopy of the solid state has been developed since the second half of the fifties. By gradual extension of experimental methods it is now possible to study the spectral dependences of the transmission and reflectivity in a broad frequency range - from vacuum ultraviolet down to 200cm^{-1} in the infrared. Absorption edge and its fine structure is studied in the 10 k - 500 K temperature range. The aim of the experimental studies performed on non-metallic materials, in crystalline, amorphous glassy and liquid states, is to determine the optical constants. The knowledge of their spectral dependence constitutes the starting point for deeper understanding of the electronic structure of these materials and its further development based on the modern microscopical quantum mechanical theory. The correlation between the details of the spectra and the main features of the band structure has been comprehended (e.g. works on orthorombic CdSb, tetragonal Hg_2Cl_2, GeS layer crystal, amorphous germanium and silicon, glassy $CdGeAs_2$, As_2Te_3 etc.).

Especially the information obtained from IR spectroscopy is used for determination of the undesirable impurities (e.g. oxygen in germanium, carbonates in PbO and in other semiconducting materials). The rewards of the Czechoslovak Academy of Sciences were awarded for the above metioned works.

The study of the nature of the impurities and of the luminiscence centres in ionic crystals and the understanding of their microscopical mechanism is closely connected with the development of the exoelectron emission spectroscopy. Since 1952 the complex investigation of these phenomena has been performed in the Institute in a broad frequency and temperature range and under the influence of further external conditions.

Models of the colour centres and impurity states of some transition and heavy metallic atoms and of the anionic complexes in ionic crystals were proposed and theoretically solved.

Results of this research work were applied in the field of the dosimetry of radiation, and in the construction of detectors. These results were also very useful in understanding of the physical factors, affecting the disease of silicosis.

In the field of the electron-paramagnetic resonance the research program which started in 1965 is mostly oriented on the study of the ions of the transition elements in diamagnetic crystals. The results have a basic importance for the understanding of the properties of magnetically ordered systems and for the study of the crystallochemical aspects of solids. The basic contribution in the field was the investigation of the vanadium ion in the tetrahedral coordination in garnets. The work was rewarded by the Czechoslovak Academy Reward in 1976. In another work the ions in the S-state (Mn^{2+} Fe^{3+}) in a group of diamagnetic garnets were studied. The correlation was found between the constants of the spin Hamiltonian of these ions and the parameters characterizing the geometrical structure of the garnets studied.

A large number of original papers dealing with above mentioned methods and their applications were published and reported at the international coneferences. Theses of the students of University as well as those of the post graduate students using spectroscopical methods are elaborated in the Institute, the members of the Institute present lectures on spectroscopy at the Charles University and at the Czech Technical University.

Further perspectives in the field of spectroscopy and of the development of the spectroscopical methods in the Institute of Solid State Physics are connected with the complex investigation of the condensed systems.

Spectroscopic laboratory
Research Institute of Agrochemical Technology (VÚAT)
Slovchemia
Bratislava

The Institute collaborates with research institutes and corporations of the Ministry of Agriculture and Food, the Ministry of Health, and with university research centres in the country. On the international scale, the Institute takes an active part in the work on problems coordinated by COMECON and INTERCHIM bodies. Intense collaboration also exists with similar institutes in the USSR, Poland, and the GDR. The Institute also takes part in the CIPAC and IUPAC projects on the analytical chemistry of pesticides and pesticide residues.

This year, the Research Institute of Agrochemical Technology celebrates 30th anniversary of its foundation. From its very start in 1947, the research work had been connected with solving problems of quantitative colorimetric analysis of some products based on biologically active compounds, i.e. pesticides. The further development of spectroscopic methods led via photometry to spectrophotometric methods. In 1958, the laboratory was equipped with an UR-10 IR spectrophotometer of Zeiss, G.D.R. and in 1964 with an UNICAM SP-100 IR spectrophotometer. Present equipment of the laboratory includes a SPECORD IR-71 IR spectrophotometers and IR-75 from Zeiss, and an UNICAM SP-8000 UV spectrophotometer.

As demanded by the research of the technological processes, the development of spectroscopic methods has been oriented from the very beginning towards quantitative analysis. The work on the analysis of the mixture of chlorine-substituted phenoxyacetic acids by UV-spectrophotometry, the use of IR-spectrophotometry for the analysis of trichloromethyl sulphenylchloride, heptachlor technical product, the mixture of the triazine herbicides simazinatrazin, and propazin, and the determination of phosdrin cis-isomer in technical product are only fen examples. The results achieved influenced directly the work on the technology, which was of an enormous technical and economical importance. The data obtained by those methods helped to optimize the technology of the particular products.

The development of spectroscopy is documented by a series of 15 papers, and 5 patents, among them the patent for the automatic colorimetric analyzer for the analysis of the working atmosphere of chemical plants.

An accent was laid on the development of quantitative spectrophotometric methods for determination of the particular components in technical products without preliminary separation. As early as in 1960 an IR-spectrophotometric method for the analysis of a 10-component mixture of the isomers, present in technical hexachlorocyclohexane, was published. The concentrations of the components were calculated by the method of successive approximations. The method was applied in 1965 in the analysis of technical hexachloro cyclopentadiene. In 1967, a universal spectrophotometric method for quantitative analysis of multicomponent mixtures was worked out, based on the solution of an overdetermined system of linear equations by the method of least squares. The method has been used in the analysis of an 8-component mixture of chlorinated benzene derivatives. This method has been further studied on different model systems, where the absorption bands corresponding to the particular components were approximated by Lorentz functions. A method was developed for the choice of optimum analytical positions and a three- to fourfold overdetermination of the linear equations was found satisfactory. The research resulted in a generalized method that makes possible the analysis of multicomponent mixtures without preliminary separation and offers satisfactory results even in case of overlapping of the component spectra.

The generalized method found its practical use in chemical and technological research of pesticidal compounds. In collaboration with the Chemical Institute of the Komenský University in Bratislava, a UV-spectrophotometric method was worked out for the analysis of a three-component mixture of technical pyrazon. It is a modern analytical method, making possible an on-line connection of the spectrophotometer with a computer, thus eliminating errors, caused by an operator. This method, together with other spectrophotometric methods developed in the Institute (IR-method and UV-method after separation by column chromatography),

was accepted by CIPAC for international testing.

The laboratory collaborates intensely with universities, giving the students facilities to work on their MS and PhD theses.

The general conception of the further development of this branch of analytical chemistry in VÚAT is based on extending the present instrumental equipment. A spectrophotometer with digital output and a programmable calculator will be added soon to the present equipment, supposed to make possible the direct processing of experimental data. Another project suggests the application of spectral methods in the studying of a reaction kinetics of multicomponent systems.

Department of Molecular Spectroscopy
The J.Heyrovský Institute of Physical Chemistry
and Electrochemistry (ÚFCHE J.H. ČSAV)
Czechoslovak Academy of Sciences
Praha

Basic as well as applied research of the relations between the structure and infrared spectra of polyatomic molecules has had a long tradition originating from the need to develop physical methods for elucidating molecular structures of compounds in natural substances or prepared in chemical laboratories. Extensive studies of these problems led to the recognition that a deeper insight into the complex nature of the relations between molecular structure and infrared spectra requires a detailed investigation of the vibration-rotation states of small polyatomic molecules. Because of this, a systematic research started about 15 years ago on the problems of the anharmonicity of molecular vibrations, vibration-rotational interactions, determination of precise molecular geometry, barriers to internal rotation and molecular inversion. Theoretical investigation of these problems using methods of quantum mechanics, group theory and advanced numerical methods has been supported by a design, construction and completion of the high resolution infrared spectrometer.

Computerized methods for automatic assignments of vibrational bands in the infrared spectra of complex molecules discovery and explanation of the role of weak collision complexes in intermolecular interactions, or a proposal of methods for determination of the degree of branching in alkanes by infrared spectroscopy are examples of the results of the chemically oriented spectroscopy research.

Determination of the anharmonic potential functions in a series of small polyatomic molecules belongs to the pioneer work in this important field of molecular physics. This research was later extended to the socalled nonrigid molecules, especially to the problem of molecular inversion in ammonia-type molecules. For the first time, a complete theoretical interpretation has been given of the physically important effect of inversion in ammonia, NH_3. This led to a discovery and theoretical interpretation of the anomalies in the inversion splitting of energy levels of ammonia. A new value of 1800 cm^{-1} has been determined for the inversion barrier in NH_3 which is about 200 cm^{-1} lower than the previously accepted value and is in an excellent agreement with the value obtained by the ab initio quantum mechanical calculations.

Last but not least, the high resolution infrared spectrometer has been put into operational condition recently and produced first high resolution data. This single beam, double--pass spectrometer equipped with a 15 x 25 cm echelle type grating, liquid nitrogen cooled photoconductive detectors and advanced lock-in-amplifiers, operates now with a 0.03 cm^{-1} resolution in the 3500-6000cm^{-1} region. An extension of the spectrum range of the spectrometer to greater wavelengths will be possible in the near future.

International cooperation has always played an important role in the research work of the Department. For example, cooperation with the group of Dr.A.F.Krupnov at the Radiophysical Research Institute in Gorky, U.S.S.R. made it possible to extend our research to submillimeterwave spectrum range. The unique sensitivity of Krupnov's submillimeter spectrometer can be used to detect rotational transitions in the excited vibra-

tional states of polyatomic molecules. The joint paper on the detection on the rotational-inversion transitions in the ν_2 states of $^{14}NH_3$ and $^{15}NH_3$ is among the first results. Another joint work will appear soon in which the detection of the pure inversion transitions in the ν_2 state of ammonia will be reported for the first time.

The Department has also a fruitful cooperation with the Infrared Lab of Prof. K. Narahari Rao from the Department of Physics at the Ohio State University in Columbus, U.S.A. in the field of high resolution infrared spectroscopy. For example, a joint analysis of the 21 infrared bands of $^{14}NH_3$ measured at the Ohio State University with resolution better than 0.03 cm^{-1} in the 700 - 3500 cm^{-1} range will be finished soon.

An important part of the international activity of our Department is the organization of International Seminars on High Resolution Infrared Spectroscopy. Starting in 1970, we have already organized four seminars with the participation of the top high-resolution infrared and microwave spectroscopists from practically all important European and many U.S.laboratories. The forthcoming fifth Seminar will be held in September 1978 at the Liblice Castle near Prague.

Experimental and theoretical research in the field of high resolution spectroscopy is planned to continue at the Department in the above mentioned directions. However, it will be extended towards the excitation of the high vibrational states of polyatomic molecules in the infrared laser field. Selective stimulation of chemical reactions vis excitation of molecules to high vibrational levels belongs to a new promising field in chemical reactivity with interesting advanced technological applications. High-resolution vibration-rotational spectroscopy can be expected to play an increasing role in the discussion of the mechanism of these reactions.

Central Control Laboratory
Nuclear Research Institute (ÚJV)
Řež u Prahy

The Central Control Laboratory (CCL) has continued the tradition of the Analytical department and other departments of the Nuclear Research Institute (ÚJV); it has been developing various spectroscopic methods for 15 years and solving by them the scientific-research and application problems.

The CCL's research is mainly concerned with the mass and infrared spectroscopies as well as electronic spectrophotometry, emission spectrography, (also with a laser excitation), and gamma spectrometry in connection with activation analysis.

In the last years, a unique mass-spectrometric laboratory was established at the CCL for an isotopic analysis of nuclear materials and the methods esp. for determining the isotopic composition and content of uranium and plutonium have been worked out. During a short period, the laboratory has become a top-level one and, therefore it was chosen by the International Atomic Energy Agency (IAEA) in Vienna for a collaboration within the framework of Safeguards. Thus, the CCL participates by highly difficult analyses of the samples taken by IAEA inspectors in different countries for checking the Treaty on Non--Proliferation of Nuclear Weapons. The CCL has already taken part successfully in 2 international experimentes organized by IAEA, in competition with 8 foreign laboratories.

The infrared spectroscopy helped in solving various problems od radiolytic cleavage of substances, reprocessing of irradiated nuclear fuel by the fluoride method, preparation of nuclear and nonnuclear materials by the sol-gel method, studying the structure of uranium compounds, and other problems of the nuclear programme and other branches of the national economy the CSSR. - By the development of a method for analyzing heavy water and by many routine analytical determinations, the laboratory of infrared spectroscopy collaborated with the first Czechoslovak atomic power plant A-1 and participated, together with the laboratory of emission spectrography and laser microprobe, in the first step of the Czechoslovak nuclear power engineering.

Using the infrared spectroscopy, the CCL contributed to an essential extent to the preparation of a highly efficient and selective sorbet for the separation of radioactive strontium and was awarded in 1971 by the Award of the Czechoslovak Academy of Sciences.

In collaboration with the institutes of the Czechoslovak Academy of Sciences, some fundamental problems of the solvent effect in solutions and of the measurement of thermodynamic functions for different complexation reactions were solved and the laboratory of infrared spectroscopy was awarded in 1967 by the Award of the Czechoslovak Academy of Sciences. - Recently, a Czechoslovak patent was obtained for a cell-less measurement of infrared spectra of aqueous solutions and suspensions.

The methods of nuclear spectrometry, esp. gamma and also alpha, are being developed and applied. In past years, these methods have predominantly been used for the activation analysis purposes whereas now they are used also for controlling the nuclear fuels. The experimental device has been gradually improved from a simple equipment with a scintillation detector up to a highly advanced gamma-spectrometric apparatus with Ge(Li) detector with a high resolving power and an in-line computer for treating the experimental data. - In connection with activation analysis, the gamma spectrometry serves for the destructive but esp. instrumental determinations of trace elements in different materials both for the research and national economy. As the industrial utilization is concerned, e.g. the purity of semiconductor and extrapure materials (Si, Ge, Ga, Al, Sb) of La in steels and slags are estimated. For medical purposes, the determinations of uranium in blood of the uranium-industry workers, Cu and Zn in neural tissues to explain the mechanism of intoxication with CS_2, and some elements in carcinomas are performed. To a great extent, this method is also used for controlling the environment pollution. Routine analyses for factories and research institutes, bringing the sum of about 400.000 Kčs in a year, are performed; the actual benefit brought by this activity to the national economy and health cannot be evaluated of course.

Besides the activation analysis, the gamma spectrometry serves for analyzing fissile products and as a comparative method for determining the isotopic composition of uranium. These problems are solved also within the framework of COMECON collaboration and the results are used for controlling the nuclear fuels and determining the burnup for the Czechoslovak Atomic Energy Commission, the atomic power plant, and IAEA.

The emission spectrography laboratory took initially part in the control of radioisotope manufacture (^{131}I, ^{32}P, ^{35}S) but gradually its contribution to the development of nuclear power engineering increased. Methods for determining about 40 elements in metal uranium were developed, now being used in routine analyses. A method for analyzing the corrosion products from the primary circuit was worked out for the A-1; also the impurities on the surface of fuel element shells were analyzed using laser excitation. - The methods for estimating 25 elements in metal Na, used as a coolant in fast reactors, were developed as well as important methods for the determination of impurities in intermediates from the production of polycrystalline germanium ($GeCl_4$, GeO_2).

During the last several years, the spectroscopic laboratories of the CCL have published their results in cca 200 papers in Czechoslovak and foreign scientific journals, lectures in different conferences, and research reports. Also an intense pedagogic activity can be observed at the CCL; many new specialists and experts were and are educated.

The CCL plays an important role in the collaboration with the USSR and other states of COMECON in solving the essential problems of the nuclear power engineering development.

Institute of Chemical Physics
Faculty of Chemical Technology
Slovak Technical University
Bratislava

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20 years ago, in December 1957, a group of several senior lecturers at the Department of Physical Chemistry of the

Slovak Technical University, together with some young physical chemists, physicists, and technicians, formed a scientific team of 12 members, a germ of the present Institute of Chemical Physics. The immediate aim of the Institute was to continue the scientific programme of the Department of Physical Chemistry, being led by Prof.V.Kellö and celebrating, at that time, the 15th anniversary of its foundation. The Department of Physical Chemistry began its activity without any scientific tradition in Slovakia, and only the purposeful effort of Professor Stehlík, coming from the University of Brno and being the head of the Department of Physical Chemistry from 1946 to 1951, formed the first research programme aimed at the study of molecular compounds and hydrogen bridges in water solutions with a combined method of diffusion and osmosis through semi-permeable membranes.

An important moment was the introduction of infra-red spectroscopy in 1950. Starting from the study of hydrogen bridges and from other smaller structural and analytical problems resulting from the requirements of the industry, a method of infra-red spectroscopy was developed and applied to the study of kinetics, mechanism and inhibition of the oxidation of natural and synthetic polymers initiated by heat and light. Later, this technique was applied also to the study of complicated natural hydrocarbon mixtures after the separation of crude oil fractions by molecular distillation and by chromatographic methods. Gradually, further spectroscopic methods in the visible and UV range were introduced, followed by Raman spectroscopy, X-ray structural analysis and the methods for the determination of molecular weight by light scattering. The elucidation of relations between dielectrical properties and oxidation stability enabled to propose an effective method of continual preparation of impregnation materials for high-voltage cables, for which the scientific workers of the Institute were awarded the State Klement Gottwald Prize in 1965.

The exact study of chain oxidation reactions of polymers and hydrocarbons by spectroscopical methods opened also the sphere of systematical study of reactivity of free radicals. This programme required to extend the absorption methods by resonance methods, as the ESR (X-band) and NMR 80 MHz spectroscopies were. By introducing the spectral computer Varian SS-100 in combination with ESR, the cryophysical methods, the high-vacuum technique, and the isotope exchange, the condition for studying the properties of free radicals on a professional level was given. The parallel use of resonance and absorption methods in combination with the measurement of dielectrical properties of low-conductive non-polar systems and the study of kinetic parameters enabled to correlate the catalytic activity with the paramagnetism of stereospecific catalysts synthesized on the basis of transient metals and organo-metals. In the case of three different types of catalysts it was shown that the paramagnetism is a necessary but not sufficient condition of their activity. It has been proved that the activity is conditioned :

1)In the case of a heterogenous catalytic system $TiCl_4$+$Al(i\text{-}but)_3$ of Ziegler-Natta type for polymerization of isoprene by crystal field of β-$TiCl_3$ after localization of the complex $[Ti^{+3}\text{-}Al]$ on the vacant places in the crystal surface.

2) In the case of a microheterogenous catalytic system $Ni(acac)_2$-$Al(C_2H_5)_3$-$BF_3O(C_2H_5)_2$ for stereospecific polymerization of the 1,4 cis-butadiene by the presence of the colloidal nickel as the carrier of active centre with an oriented π-allyl complex on Ni^{+1}.

3) In the case of a homolytic catalytic system $CuCl_2$ - cyclohexylamine for oxidative bonding of 2,6 - dimethylphenol to polyphenylether by coordination of two amine molecules to the central Cu^{+2} ion. The process of the polymer increase is controlled by the radical-ion polymerization, which has been unambiguously indicated by the ESR spectroscopy in combination with the simultaneous measurement of the electric conductivity.

On the one hand the theoretical interpretation of experimental results of the study of free radicals and of the structure of active centres of catalysts necessitated naturally a close cooperation with the quantumchemical group founded by Prof.Valko in the Department of Physical Chemistry. On the other hand, the experimental research gradually has come more and more in contact with the solution of topical economic research problems of chemical industry. On that account, there were developed methods and in own workshops constructed prototype arrangements for the

study of continuously passing chemical reactions and also of physical high-vacuum processes in thin-moving films. The continuous film reactions as well as the continuous film molecular distillation were developed not only in laboratory dimensions, but also in pilot plants and led to an intensification of chemical processes and to an introduction of the new technique and of production processes.

But as the centre of the theoretical research remains the sphere of free radicals. From the results of a common signification, reached in this field in the last years, it can be quoted :

a) Elaborating of the method and explaining of laws of stabilization of peroxy and alkoxy radicals in the coordinated sphere of transition metals. The fixed radicals RO_2 ° and RO ° with a lowered reactivity mutually differ by g-factor of ESR signals and they can be used for generation of secondary radicals for the study of their reactivity. In the case of this type of oxidative-additive reactions the generated radicals remain coordinated in the highest oxidative stage of the transition metal. The driving force of these reactions is the effort to reach the stable 18-electron valence configuration. In all systems studied the ESR technique pointed that the transition metals behave as free radicals with an unpaired electron, which remains preserved at oxidative as well as reduction reactions. The fixation of peroxy radicals was proved in the last time also in the case of the metalenzymes of the respiration chain (cytochromes, peroxidases, catalases).

b) The coordinated radicals were used in a large scale for the study of low-temperature oxidation and of bimolecular homolytic substitution reactions for testing the relations between the electron structure, the degree of delocalization, and the reactivity in the process of inhibition of phenolic, bisphenolic and tiobisphenolic antioxidants. By this spectroscopy technique it was first time possible to prepare free radicals in a sufficiently high concentration in liquid phase under laboratory temperature from N-heterocycle molecules of the pyrazole, benzimidazole and indole line. The elaboration of the so called "pulse" method of the simultaneous generation of several radicals of different reactivity gave a criterion for the study of the important process of H-transfer of radical cascades.

c) In the field of biochemical application, the ESR method gave valuable information about the effect of toxic substances during one-electron reactions, about non-enzymatic decomposition of peroxides and about non-controlled superoxy oxygen formation as well as about the attack of free radicals on biological targets (radiomimetic reaction). From this point of view the neutral cation and anion radicals of carcinogenic substances were studied (aromatic amines, azo-compounds, polyaromates) that interfere during the process of electron or hydrogen transfer with the active centres of metalenzymes connected with the loss of the identity of the cells.

d) A mechanism of molecular oxygen activation on cobalt controlled by redox properties of the ligand was detected.

e) The radical character of one electron transfer from a lone pair of $\bar{S}$, $\bar{P}$, $\bar{N}$ in organic compounds to peroxides was proved by the modified ESR technique.

f) In the sphere of electron-transfer study there was elaborated a new high-effective method of ketyl-radicals generation, catalyzed by transition metals in the presence of organometals. Controlling the reduction force by the choice of Grinard agents of different structure, of butyllithium, of triethylaluminum, hydrides in combination with various complexing agents and solvents as well as by isotope exchange it was possible to generalize this method for preparation of anionradicals giving high-resolved hyperfine ESR spectra. This technique made it possible to study a whole range of new spectroscopy effects as interactions of protons, formation of CT-complexes between anion radicals and complexing solvents, to correlate the spin density with quantum-chemical calculations and to contribute to the theory of ion pairs.

g) The elaboration of the method for isolation of free radicals from the pyrolysis of polymers by applying a rotating cryostat working at $-195^{\circ}C$ and 5×10^{-4} Torr in combination with a low--temperature ESR technique led to obtaining principially new information about the process of burning of polymers on a molecular level. In cooperation with industry the problem of retardation of polymer burning was successfully solved by this method on the basis of brominated and organophosphoric scavengers (disactivators) of free radicals.

In the field of quantum chemistry, the MO theory was applied to the study of mechanism of homogeneous catalysis on the base of transition metals. There were elaborated perturbation methods for calculation of interactions with closed and opened electric levels. The weak intermolecular interaction forces, using diagrammatic methods, were studied.

The establishment of a new up-to-date laboratory equipped with a NMR spectrometer 100 kHz with FT and possibility to measure also spectra C^{13} would be an important factor of the further development of spectroscopic methods. Thus the studies of inducted polarization of nuclei, as well as of double resonance aiming at examination of paramagnetic effect of radicals and transition metals using NMR spectra are prepared.

The Institute co-operates intensely with a whole range of research institutes of universities, of Czechoslovak Academy of Sciences, and of Slovak Academy of Sciences, as well as with the research institutes of chemical industry in Czechoslovakia and abroad. Together, about 350 papers and research reports have been published and 30 patents obtained.

Spectroscopic laboratories
Institute of Chemical Technology
Praha

The Institute participates in the development and utilization of various spectroscopic methods in Czechoslovakia.

Spectroscopy has been a traditional subject at the Institute (orginally one of the faculties of the Technical University in Praque) since pre-war days. This fact made it possible to organize large groups of highly skilled workers engaged in various fields of spectroscopy during the last fifteen years. The department laboratories have been gradually equipped with first class up-to-date devices and apparatuses for spectroscopic research. At present the Institute is one of the foremost and most prominent Czechoslovak centres of spectroscopy.

Besides maintaining contact in the field of fundamental research with almost all technical universities in Czechoslovakia, the Institute plays an important role in the solution of specific problems of the Nation's economy by spectroscopy.

One of the greatest contributions of the Institute of Chemical Technology to Czechoslovak spectroscopy is its activity in providing instructions and popularizing lectures. Each year hundreds of graduates leave with knowledge both of spectroscopic theory and practice, lot of them pursuing the deeper study of this subject further on.

A characteristic feature of the work in the departments of spectroscopy is its complexity; this is evidenced not only by a number of developed and applied techniques of atomic and molecular spectroscopy, but above all by an extensive co-operation among departments in the solution of particular problems.

Laboratory of Flame Spectroscopy

The Laboratory co-operates with a number of Czechoslovak plants and research institutes, providing analyses and consultations on applied atomic absorption spectrometry. To mention just a few : the Chemical Works at Záluží and Benzina Prague (oil and heavy organics), Kaučuk Kralupy (rubber), Tesla Rožnov (electronics), the Paper Research Institute, Prague and the Faculty Hospital, Prague 2. The seminars on new developments in AAS are organized yearly, frequently with international participation.

The laboratory, founded in 1968, aimed its activity at first at flame AAS developments and applications, then towards the analytical application of atomic fluorescence. At present the major research assignments involve the analysis of petroleum and petroleum based products by methods of atomic spectrometry, the study of flameless atomization and the development of electrothermal atomizers.

The activity of the laboratory is evidenced by 64 papers, 4 patents obtained and one monography on atomic fluorescence.

The Department of Electron -Probe Microanalysis and Microsopy of the Corporate laboratory for the Chemistry and Technology of Silicates
of the Czechoslovak Academy of Sciences and the Institute of Chemical Technology

The department co-operates with a number of institutions in Czechoslovakia and abroad, e.g. The Siberian Department of the Soviet Academy of Sciences - the Institute of Geology and Geochemistry, in the field of quantitative analysis of silicates. In co-operation with the Geological Institute of the Czechoslovak Academy of Sciences the department was the first in COMECON countries to begin with the quantitative microanalysis of lunar samples brought by Luna 16 and later analysed the samples from the Apollo 11 and 12 expeditions.

The department was founded in 1968 and is directed towards the possibilities of electron-probe microanalysis of silicates. Later on, the results of its work have been applied in the glass industry, where at present microanalysis plays an important role in production control of technical glass. The method of chemical microanalysis has been gradually extended to metals, mineralogical and other inorganic materials, polymers and organic materials to meet the requirements of the other departments of the Institute. The main activity, however, is still the application of electron-probe microanalysis of silicates. Such important problems as diffusion phenomena in glass melts and glasses, phase separation and crystallization of glass, the corrosion of refractory materials and electrodes during the electric melting of glass etc.are studied. A series of important industrial problems, such as e.g. the corrosion of molybdenum by elementary antimony during the electromelting of glass, or corrosion of fuel elements in reactor, exploitation of cermets, identification and explanation of occurrence of inhomogeneities in glass etc. have been solved.

The department's achievements have been internationally appreciated by honorary rewards, offers of foreign societies memberships and invitations to important conferences

Laboratory of Nuclear Magnetic Resonance

The laboratory collaborates with several institutes of the Czechoslovak Academy of Sciences, especially with the Institute of Inorganic Chemistry in Řež, and the Institute of Plant Instrumentation in Brno. The workers of the laboratory participate also in the evaluation of Tesla (ČSSR) NMR spectrometers. In 1977 the laboratory organized an international meeting of experts from COMECON countries, which received positive response among the participants, and will be organized again in 1978. The laboratory was established at the beginning of the year 1972, following the activity of the laboratory of NMR, existing at the Department of Instrumental Analytical Methods since 1967. At first the laboratory dealt mainly with the structural analysis of organic molecules. The study of "classical" problems was later extended to a systematic research of the possibilities of using shift reagents and to the problems of utilizing "off-line" computers in NMR spectroscopy. The results of this research were summarized in 35 papers and 12 lectures at various symposia. Owing to the existing instrumentation of the laboratory various methods were applied to the NMR of fluorine compounds. The methods have been successfully utilized also in analytical study of intermediates and reactions for the preparation of the manufacture of the anaesthetic Anecotan. Recently the laboratory has been developing NMR for the analysis and evaluation of high-boiling products of processing of petroleum and coal. This complex problem is in close connection with the contemporary requirements of the national economy.

Laboratory of Mass Spectroscopy

The laboratory co-operates with a number of scientific institutions in the COMECON countries, mainly VNIINP Moscow, which yielded a series of works on the analysis of Romaškin petroleum products. It was established in 1967 and at first aimed its activities predominantly at organic structure analysis. Gradually, however, more and more substantial analytical problems of technological importance have been solved, e.g. for Kaučuk Kralupy (rubber), the Pharmaceutical Works Spolana, Neratovice, the East-Bohemian Chemical Works, Rybitví Pardubice, the Society for

Chemical and Metalurgical Production, Ústí n/L., etc.

The major concern of the laboratory's scientific activity is oriented entirely to the environmental analysis. The two basic integrated activitiet are : the determination of volatile organic substances polluting water and environment and the investigation of organic substances in the burning process and slab pyrolysis. Apart from purely theoretical activities in this branch, the laboratory is engaged in solving numerous specific industrial problems, which is evidenced by more than 40 papers. The laboratory members have delivered a number of lectures on mass spectrometry at various conferences and seminars, most important being two seminars on the exploitation of mass spectrometry and other related techniques in the crude oil and petrochemical industry under the name Petromass '73 and '75. The seminars organized by laboratory gained wide international appreciation.

In consequence, the laboratory was selected as the co-ordination centre for the COMECON countries.

The Past and the Future of the Czechoslovak Spectroscopic Society.

The main goal of the Society is to integrate and co-ordinate the individual efforts in the vast field of spectroscopy, both in basic research and applications in the national economy. The annual reports and brief descriptions of different laboratories give the reader an idea of the Society's varied activities.

The contemporary life of the Society stems from both tradition and dynamic growth. The precursor of the Society, the "Association for Research in Spectroscopic Analysis", was founded already in 1949 and joined the Czechoslovak Academy of Sciences as one of its scientific societies in 1966. This step in particular provided for a solid scientific base and opened up the possibility to cover actually all branches of this rapidly growing science spectroscopy. Nevertheless, the Society remained attractive for the practising industrial spectroscopist as well. The lively and informal contacts between science and practice are characteristic features of the Society: they open the way both for an efficient utilization and a steady stimulation of spectroscopic research.

The recent programme and future plans of the Society are in full agreement with the Conclusions on the scientific & technical development of the XV. Congress of the Czechoslovak Communist Party. The effort of Czechoslovak spectroscopists thus aims at a search for possible applications of spectroscopy so that the production may be increased, for economizing of energy and raw material resources and for environmental protection.

These tasks are highly complex and demanding; they cannot be fulfilled without efficient co-operation both inside the country and abroad. The members of the Society are proud to take a fair part in the international effort towards scientific and economic co-operation as well as towards better mutual understanding.

This was the spirit in which the XX.CSI and 7.ICAS have been organized, this is the spirit in which the Society intends to enlarge its contacts with all who are ready for fair co-operation.

Vydává Československá spektroskopická společnost při ČSAV
se sídlem ve Výzkumném ústavu ČKD v Praze 9, Na Harfě 7.
Za ČSSS zodpovídá Dr.B.Moldan CSc.
Redakce Ing.J.Moravec CSc a Ing.K.Bičovský.
Redakční uzávěrka 15.dubna 1977.
Pouze pro vnitřní potřebu.