

DEPARTMENT OF ANALYTICAL CHEMISTRY

Head of Department:

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I. STAFF

Full Professors :

Dušan Bustin PhD, DSc; Ján Krupčík, PhD, DSc, Jozef Lehotay, PhD, DSc., Eva Matisová, PhD, DSc, Ján Mocák, PhD, DSc

Associate Professors :

Ernest Beinrohr, PhD, Eva Brandšteterová, PhD, Miroslav Čakrt, PhD, Ján Labuda, DSc, Drahomír Oktavec, PhD, Miroslav Rievaj, PhD, Jozef Polonský, PhD, Viktor Vrábel, PhD

Assistant Professors :

Eva Benická, PhD, Tatiana Buzinkaiová, PhD, Andrea Hercegová, PhD, Elena Korgová, PhD, Pavol Májek, PhD, Alena Manová, PhD, Pavol Tarapčík PhD, Mária Vaničková, PhD

Research Fellows::

Miriám Bučková, PhD, Adriana Ferancová, PhD, Katarína Hroboňová, PhD, Jarmila Laštincová, Jana Sádecká, PhD, Ivan Skačáni, PhD, Ivan Špánik, PhD, Peter Tomčík, PhD, Magdaléna Valachovičová

PhD Students:

Branko Balla, Eva Blahová (till 1.10.2001), Jana Ďungelová, Peter Korytár, Petra Kotianová

Technical staff :

Marta Benešová, Zuzana Cifrová, Jana Otrubová, Juraj Žemlička

II. TEACHING AND RESEARCH LABORATORIES

Laboratory of capillary gas chromatography

Laboratory of high performance liquid chromatography

Laboratory of electroanalytical methods

Laboratory of molecular spectrometry

Clean laboratory for trace analysis with atomic spectrometry (AAS, OES-ICP)

Laboratory of organic elemental analysis

Laboratory of organic synthesis

Laboratory of fluorescence analysis

Laboratory of capillary isotachopheresis

Laboratory of electrochemical pre-concentration for atomic spectroscopy

Laboratory of chemometry

Laboratory of bioanalytical chemistry

III. TEACHING

A. Undergraduate Study

4th semester

Analytical Chemistry I. (2-2h)

Beinrohr, Krupčík, Labuda, Polonský, Vrábel

Laboratory Practice AC I.

(0-4 h) Valachovičová

5th semester

Analytical Chemistry II.

(2-2 h)

Bustin, Čakrt, Lehotay, Polonský

Laboratory Practice AC II.

(0-4 h)

Korgová

Testing and Quality Control

(1-1 h)

Čakrt, Tarapčík

6th semester

Semestral Project

7th semester

Atomic Spectrometry

(2-0 h)

Beinrohr, Manová

Anal.Chem.of Complex Inorg. Mixtures

(2-0 h)

Oktavec, Polonský

Anal.Chem. of Complex Org. and

Biological Mixtures

(2-0 h)

Brandšteterová, Skačáni

Lab.Practice I.

(0-1 h)

Matisová

Biosensors

(2-1 h)

Labuda, Mocák

Computer evaluation of anal. measurement

(2-0 h)

Májek, Mocák

8th semester

Electrochemistry and Electro-analytical Chemistry

(2-1 h)

Bustin, Mocák

Techniques of Mixtures Separation

(2-2 h)

Matisová, Valigura

Analytical Separation of Compounds	(2-1 h)	Krupčík, Matisová
Lab.Practice II	(0-6 h)	Matisová
Nuclear Analytical Chemistry	(2-0 h)	Tarapčík, Májek
Trace Analysis and Microanalysis Methods	(2-0 h)	Beinrohr, Čákr
Environmental Analytical Chemistry	(2-0 h)	Benická, Buzinkaiová
Automatisation of Analytical Chemistry	(2-0 h)	Rievaj, Dzurov
9th semester		
Bioanalytical Chemistry	(2-1 h)	Labuda, Mocák
Identification of Chemical Substances	(2-1 h)	Lehotay, Liptaj
Lab.Practice V.	(0-1 h)	Matisová
10th semester		
Laboratory of Diploma Work	(0-30 h)	
Selected Subjects		

IV.

CURRENT RESEARCH PROJECTS

A. Development and Application of Direct Injection Assays for HPLC Analysis of Some Drugs and Toxic Compounds in Biological Samples (E. Brandšteterová). The aim of the project was the development and the application of new assays with the possibility of direct injection of biological samples into the HPLC system. SPE (Solid Phase Extraction) precolumn was integrated directly into the HPLC system what improves validation parameter values and minimizes the personal contact with biofluids. Automated HPLC procedures for the analysis of chosen drugs, natural and toxic compounds were compared with applied electromigration methods.

B. New Electroanalytical and Spectroscopic Systems for Ultra-trace and Speciation Analysis with Special Emphasis to Environmental and Clinical Problems. Optimization of Analytical Procedures (D. Bustin). The project was oriented to the development of analytical methods for ultratrace analysis of some biologically and environmentally important analytes. Flow - through galvanostatic chronopotentiometry has proved to become a calibrationless method for the determination of electrochemically active species. The method enables the measurement of extremely low (below $\mu\text{g/l}$) as well as medium and high analyte concentrations, e.g. over g/l . The method provides reliable results and owing to its robustness it can also be applied for unattended monitoring of toxic species in waters. Porous electrodes facilitates the electrochemical generation of hydrides for their consecutive determination by atomic spectroscopy. Flow-through coulometry was successfully applied for the measurement of metal layer thickness. Using new chemometrical and statistical procedures, the original multidimensional analytical data were transformed into ulticomponents, which enable a detailed characterization of clinical, environmental or food samples. Thus, it was possible to find the way how the original chemical and physical parameters affect the main diagnostic features or desired properties of the analyzed subject or object. New chemometrical procedures were utilized for obtaining the resolution of the overlapped voltammetric signals as well as the modelling of the signal shape and height under different conditions of an electrochemical experiment. The IUPAC- recommended method for determining the limits of detection and quantification was reformulated in a new, more comprehensive way. This way applies easy calculation, however, it is still statistically perfect.

C. Grant GAV 1/6104/99 Development of selective methods for analysis of biologically active compounds by selected chromatographic and electroanalytical methods (J. Krupčík). This project intends to contribute to development of methods for: (a) Computer assisted optimization of chromatographic separation of mixtures of biologically active compounds in several columns coupled in series under multicolumn and multidimensional separation conditions, (b) Modeling of gas-chromatographic separation and explanation of chiral recognition of enantiomers in gas chromatography on chiral columns using knowledge and data obtained by theoretical chemistry (semiempirical and "ab initio" methods) and by structure -retention data correlations for enantiomers of analyzed compounds. (c) Computer assisted signal processing for deconvolution together with an increase of signal to noise ratio of weak signals which are not resolved experimentally. Elaborated algorithms shall be exploited for processing of signals in gas chromatography and electroanalytical methods. Procedures for the determination of uncertainties and evaluation of signal errors shall be implemented in elaborated algorithms, too. (d) Analytical characterization and application of new types of chemical modifiers for electrodes, based on DNA, proteins and substances with the chiral properties.

D. Trace analysis of selected analytes in complex organic systems by combination of preconcentration techniques and capillary gas chromatography.(E. Matisová) The project is oriented to the development of methods for the trace analysis of selected, particularly volatile and semivolatile compounds in complex organic systems - in environmental matrix utilising preconcentration techniques in combination with capillary GC. A part of the project is devoted to the miniaturisation in analytical chemistry: - to the development of

microextraction methods for the sample preparation, large volume injection in capillary GC. A part of the project is connected with the development of high speed GC, - devoted to trace analysis.

D. CEEPUS PL-110 01/02. Development and improvement of modern analytical methods for monitoring the environmental pollution and introduction of the quality systems and accreditation to routine analytical laboratories. It is an educational project enabling the mobility of the students and teachers among 5 partner universities: The University of Mining and Metallurgy, Cracow, Karl-Frenzens-University of Graz, Graz, The University of Ljubljana, Ljubljana, The University of Pardubice, Pardubice, and Slovak University of Technology, Bratislava). The Slovak national coordinator is J. Mocák. Project duration: academic year 2001/2002.

E. The development of modern method for teaching analytical chemistry supported by PC (P. Tarapčík)
The main drawbacks of traditional teaching method of analytical chemistry are: low individual activity of students in classroom; unified work rhythm not considering individual abilities of students; low variability; high cost of modern laboratory method. There is a possibility to overcome this drawbacks applying relatively individual work in front of PC. The main goals of this project are:
- to provide simulating software for various analytical methods, mainly in the area of chemical equilibria in analytical chemistry,
- to provide teaching procedures supported by simulating software, combining work methods in whole group by traditional method and in small groups (two-three students) with PC.
The spreadsheet „EXCEL“ is widely used calculating product with high poverty of graphics, statistics..., the supporting software will be made on this basis as the EXCEL sheets, partly with macros in VBA language.

V. COOPERATION

A. Cooperation in Slovakia

Department of Microelectronics Faculty of Electrical Engineering and Information Technology, Slovak University of Technology Bratislava

Department of Petroleum Technology, Department of Biotechnology and Environment, VURUP, Slovnaft a.s. Bratislava

Department of Plant Physiology, Faculty of Natural Sciences, Comenius University, Bratislava
Faculty Hospital, Bratislava

Food Research Institute, Bratislava

Hospital for Tuberculosis and Respiratory Diseases, Department of Clinical Chemistry, Kvetnica, 058 87 Poprad

Hospital of the Ministry of Defence, Division of Clinical Laboratories, 833 31 Bratislava

National Institute of Oncology, Bratislava

Pharmaceutical Faculty, Comenius University, Bratislava

Slovak Institute of Metrology, Karloveská 63, 842 55 Bratislava

B. International Cooperation

Department of Analytical Chemistry, Chemical Technological Faculty, University, Pardubice, Czech Republic

Department of Analytical Chemistry, Palacky University, Olomouc, Czech Republic

Department of Environmental Chemistry and Ekoanalytics, Faculty of Chemistry

Department of Chemistry, Gilman Hall, Iowa State University, Ames, Iowa, USA

Chiral separation of optical active compounds by HPLC and HRGC

Department of Organic Chemistry, University of Gent, Gent, Belgium

Chiral separations by HRGC

Faculty of Material Engineering and Ceramics, The University of Mining and Metallurgy, Al. Mickiewicza 30, 30-059 Cracow, Poland

Institut de Physique Nucléaire, 91406 Orsay Cedex, France

Institute of Pharmaceutical Chemistry, University of Muenster, Germany

Nicholas Copernicus University, Toruń, Poland

Prof. Hans Puxbaum; Technical University Vienna, Institute of Analytical Chemistry, Vienna, Austria

Utilisation of Capillary GC in Combination with Preconcentration Techniques for the Analysis of Organic Compounds in Aerosols

C. Membership in Domestic Organizations and Societies

Chairman of Scientific Group “Chromatography and Electrophoresis”, Slovak Chemical Society, Slovak Academy of Sciences, Bratislava (E. Brandšterová)

Chemical Papers Editorial Board (D. Bustin)

Membership in the Editorial Board of the Slovak scientific journal *Laboratory Diagnosis* (J. Mocák).
Slovak Chemical Society at Academy of Science, Group of Analytical Chemistry (J. Krupčík)
Slovak Chemical Society at Academy of Science, Group of Analytical Chemistry (J. Lehotay)
Slovak Chemical Society (D. Bustin)
Slovak Society of Clinical Biochemistry. ISSN 1335-2644 (J.Mocák)

D. Membership in International Organisations and Societies

American Chemical Society (D. Bustin)
European Commission, Science, Research and Developments (J. Lehotay)
Chemical Analysis Editorial Board (J. Lehotay)
IUPAC (D. Bustin)
UICC (International Union Against Cancer), Geneva, Switzerland, (Eva Brandšteterová)

E. Tempus Programme :

F. International Scientific Programmes :

Grant No. 002-98, Slovak – US Universities Co-operation. Mechanistic study of chiral recognition in HPLC and HRGC (J. Krupčík). The main objective of the project is to study mechanistic aspects of chiral recognition in the direct separation of enantiomers by HPLC and HRGC. The influence of structure and polarity differences in substituents bonded to the asymmetric carbon atom in enantiomers, and selectivity of a chiral selector in HPLC and HRGC shall be studied in detail. Elaborated optimum separation system shall be used for two dimensional separation of optically active compounds in natural samples.

G. Visitors from Abroad

Prof. D.W.Armstrong Department of Chemistry, Gilman Hall, Iowa State University, Ames, Iowa, USA, August 2001 (5 days)

Prof. Andrzej Bobrowski, DSc., The University of Mining and Metallurgy, Cracow, Poland, March 2001 (10 days)

Prof.A.Manschreck, Department of Organic Chemistry, University of Regensburg, Germany

Prof.P.Sandra Department of Organic Chemistry, University of Gent, Gent, Belgium, August 2001 (5 days)

Dr. Matija Strlič, The University of Ljubljana, Ljubljana, Slovenia, January-February 2001 (44 days)

H. Visits of Staff Members and Postgraduate Students in Foreign Institutions

B. Balla Prag, Czech Republic, May 31 2001 (1 day)

B. Balla Austria, June 10-30 2001 (21 days)

E. Beinrohr Gent, Belgium, February 10-13 2001 (4 days)

E. Beinrohr Budapest, Hungary, February 27 2001 (1 day)

E. Beinrohr Fridek – Mistek, Czech Republic, June 12 2001 (1day)

E. Beinrohr Czech Republic, conference, November 13-14 2001 (2 days)

M. Bučková Dortmund, Germany, February 15 – May 14 2001 (1 month)

M. Bučková Dortmund, Germany, September 3 – November 2 2001 (2 months)

M. Čakrt Fridek-Mistek, Czech republic, June 12 2001 (1day)

J. Ďungelová Czech Republic, November 19 – December 19 2001 (1 month)

A. Ferancová Czech Republic, January 5 – January 31 2001 (1 month)

A. Ferancová Czech Republic, conference, June 19–23 2001 (5 days)

A. Ferancová Prag, Czech Republic, July23 – August 18 2001 (1month)

S. Hrouzková Sophia, Bulgary, June 6-10 2001 (5 days)

P. Korytár Deutchland, January 1 December 31 2001 (12 months)

P. Kotianová Wien, Austria, January 29 2001 (1 day)

P. Kotianová Wien, Austria, January 31 2001 (1 day)

P. Kotianová Wien, Austria, March 1 2001 (1 day)

P. Kotianová Las Vegas, USA, conference, May 18-28 2001 (11 days)

P. Kotianová Wien, Austria, May 4 2001 (1 day)

P. Kotianová Wien, Austria, June 29 2001 (1 day)

P. Kotianová Wien, Austria, July 31 2001 (1 day)

J. Krupčík Wien, Austria, conference, January 23-24 2001 (2 days)

J. Krupčík Belgium, coneference, May 27 – June 2 2001 (7 days)

J. Krupčík USA, conference, July 10–23 2001 (14 days)

J. Krupčík Bruxelless, Belgium, conference, September 17–22 2001 (7 days)

J. Krupčík Deutchland, conference, October 7–10 2001 (4 days)

J. Krupčík Bruxelles, Belgium, November 14–16 2001 (3 days)
 J. Krupčík Paris, France, conference, December 3–6 2001 (4 days)
 J. Labuda Czech Republic, June 19–23 2001 (5 days)
 J. Labuda Deutschland, September 29 – October 5 2001 (8 days)
 J. Labuda Czech Republic, November 1–2 2001 (2 days)
 J. Labuda Czech Republic, November 14–16 2001 (3 days)
 J. Labuda Czech Republic, November 27–December 1 2001 (5 days)
 J. Lehotay Wien, Austria, conference, January 23–24 2001 (2 days)
 J. Lehotay Czech Republic, conference, May 22–25 2001 (4 days)
 J. Lehotay, Danmark, conference, June 4–7 2001 (4 days)
 J. Lehotay Czech Republic, conference, June 12–14 2001 (3 days)
 J. Lehotay USA, conference, July 10–23 2001 (14 days)
 J. Lehotay Siofok, Hungary, September 1–5 2001 (5 days)
 J. Lehotay Poland, September 19–21 2001 (3 days)
 J. Lehotay Deutschland, conference, October 7–10 2001 (4 days)
 J. Lehotay Czech Republic, October 30 2001 (1 day)
 J. Lehotay Belgium, November 14–16 2001 (3 days)
 E. Matisová Spain, conference, May 9–18 2001 (10 days)
 E. Matisová Wien, Austria, June 29 2001 (1 day)
 E. Matisová Deutschland, conference, October 7–10 2001 (4 days)
 J. Mocák Brno, Czech Republic, May 23–24 2001 (2 days)
 J. Mocák Graz, Austria, June 15–30 2001 (15 days)
 J. Mocák Poland, September 14–21 2001 (8 days)
 P. Oswald Gent, Belgium, February 6 - July 5 2001 (5 months)
 I. Špánik USA, August 1 – December 31 2001 (5 months)
 A. Žiaková Pardubice, Czech Republic, June 18–19 2001 (2days)

VI. THESES AND DISSERTATIONS

A. Graduate Theses (MS Degree) for state examinations after five years of study in Analytical Chemistry (Supervisors are written in brackets)

Adamcová Z.: HPLC ultratrace analysis of some nitroaromatics compounds in soil samples (K. Hroboňová)
 Baranová V.: Analysis using DNA biosensor (J. Labuda)
 Blahová E.: The study of sorbents of new generation in sample-handling before HPLC analysis of morphine and its metabolites (E. Brandšteterová)
 Čonka K.: Extraction of persistent organic compounds from biological samples using the methods of SPE (A. Kočan)
 Gašpířiková K.: Study of the sorption of some cations on the teflon surface (P. Tarapčík)
 Heldiová E.: In – electrode coulometric titrations: determination of some metals (E. Beinrohr)
 Hrašková L.: Preparation and application of DNA modified electrode. (M. Vaníčková)
 Krascenits Z.: Determination of fluorides in waters (M. Čákrť)
 Křištof J.: Detailed twodimensional and multidimensional data analysis (J. Mocák)
 Malychová Z.: Isotachophoretic determination of citric and isocitric acid (J. Sádecká)
 Martinkovičová K.: In – electrode coulometric titrations: determination of acids and bases (E. Beinrohr)
 Rexová D.: Application of microelectrodes in voltammetric analysis (M. Rievaj)
 Rojkovičová T.: The HPLC separation of some enantiomers using macromolecules of antibiotics as stationary phase (J. Lehotay)
 Senková Z.: Determination of antidepressive drugs and pollutants using electrochemical biosensors (E. Korgová)
 Skaličanová A.: Electroanalytical application of interdigitated microelectrodes arrays (D. Bustin)
 Slaná I.: Determination of phosphates in waters by flow –trough coulometry (A. Manová)
 Šimeková M.: Fast gas chromatography and its utilisation in the analysis of organic compounds (E. Matisová)
 Škrabáková Z.: Optimization of the Determination of High Boiling Petroleum Hydrocarbons by UV and IR Spectrometry (D. Oktavec)
 Štorcel M.: Possibility of determination some chosen drugs by capillary isotachopheresis (T. Buzinkaiová)
 Tóthová A.: Crystal structure and biological activity of 1,4 dihydropyridine derivatives (V. Vrábel)
 Verčík T.: The analysis of persistent organic contaminants in food products (E. Benická)
 Žabka M.: Optimization of separation of enantiomers by dual column capillary gas chromatography in chiral columns (J. Krupčík)

B. Dissertations (PhD.):

Ferancová A.: Development and utilization of sensors based on β - cyclodextrin modified electrodes (J. Labuda)

Hercegová A.: Utilization of some electromigrating methods in pharmacy ITP determination of some selected nonsteroidal antirheumatics in body fluids and pharmaceutical preparations (J. Polonský)

VII. Publications

A. Journals (* registered in Current Contents)

- [1]* Balla B., Mocák J., Pivovarníková H., Balla, J., Kavková D., Varmusová E.: Application of Multivariate Analysis in Laboratory Medicine. *Clin. Chem. Lab. Sci.* 39, 285 (2001)
- [2]* Beinrohr E.: Flow-through coulometry as a calibrationless method in inorganic trace analysis. *Accred. Qual. Assur.*, 6, 321-324 (2001)
- [3]* Blahová E., Bovanová L., Brandšteterová E.: Direct HPLC analysis of trimethoprim in milk. *J. Liq. Chrom. & Rel. Technol.*, 24 19, 3027-3035 (2001)
- [4]* Borošová D., Mocák J., Beinrohr E., Bobrowski A.: Determination of arsenic in water quality assurance and calculation of metrological characteristics. *Centr. Europ. J. Public Health* 8, 196-198 (2001)
- [5]* Brandšteterová E., Endresz E., Blaschke G.: Chiral separation of verapamil and some of its metabolites by HPLC and CE. *Pharmazie* 56, 7, 536-541 (2001)
- [6] Buzinkaiová T.: Úlohy z chemickej praxe. *Chemické rozhľady* 4, ISSN 1335-8391 (2001)
- [7]* Buzinkaiová T., Polonský J., Skačáni I.: Determination of fendiline and gallopamil by capillary isotachopheresis. *J. Chromatography B* 757, 215 (2001)
- [8] Čakrt M.: Entries in: *Encyclopaedia Beliana. 2. zväzok, Bell – Czy, EÚ SAV & Veda, Bratislava, ISBN 80-224-0671-6* (2001)
- [9] Čakrt M., Berek J., Beinrohr E., Dostálek P., Dzurov J., Enge J., Glatz Z., Heyrovský M., Karliček R., Katrlík J., Krofta J., Křivánková L., Kvasnička F., Labuda J., Ledvinka K., Opekar F., Sádecká J., Vespalec R., Vyskočil L.: *Elektroanalytické metody. Ing. Václav Helán – 2THETA, Český Těšín, ISBN 80-86380-07-6, 316 pp.* (2001)
- [10]* Čakrt M., Hercegová A., Leško J., Polonský J., Sádecká J., Skačáni I.: Isotachopheretic determination of naproxen in the presence of its metabolite in human serum. *J. Chromatogr. A* 916, 207 – 214 (2001)
- [11] Čaniová A., Argalášová-Šútovská K., Brandšteterová E., Lux A.: Porovnanie obsahu peroxizomicínu A_1 v *in vivo* a *in vitro* kultúrach rastlín rodu *Karwinskia* metódou HPLC (in Slovak). Comparison of the content of peroxizomicine A_1 in vivo and in vitro cultures of plants *Karwinskia* by HPLC. *Farmaceutický obzor*, 9, 215-218 (2001)
- [12]* Čaniová A., Brandšteterová E.: HPLC analysis of phenolic acids in *melissa officinalis*. *J. Liq. Chrom. & Rel. Technol.*, 24, 17, 2647-2659 (2001)
- [13]* Čaniová A., Brandšteterová E.: HPLC analysis of phenolic acids in plant material. *Chem. Anal.*, 46, 757-780 (2001)
- [14] Čižmárik J., Lehotay J., Bednáriková B.: Štúdium lokálnych anestetík. CLVII. Chromatografické vlastnosti pentakainu, karbizokainu, heptakainu a jeho 3- a 4- polohových izomérov v RP systéme HPLC. *Čes. a slov. Farm.* 50, 5, 233-238 (2001)
- [15]* Ferancová A., Korgová E., Buzinkaiová T., Kutner W., Štěpánek I., Labuda, J.: Electrochemical sensors using screen-printed carbon electrode assemblies modified with the β -cyclodextrin or carboxymethylated β -cyclodextrin polymer films for determination of tricyclic antidepressive drugs. *Anal. Chim. Acta* 447, 47-54 (2001)
- [16]* Ferancová A., Labuda J.: Cyclodextrins as electrode modifiers. *Fres. J. Anal. Chem.* 370, 1-10 (2001)
- [17]* Ferancová A., Labuda J., Kutner W.: Electrochemical Quartz Crystal Microbalance Study of Accumulating Properties of the β -Cyclodextrin and Carboxymethylated β -Cyclodextrin Polymer Films with Respect to the Azepine and Phenothiazine Type Antidepressive Drugs. *Electroanalysis* 13, 1417-1423 (2001)
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- [29]* Lehotay J., Hroboňová K., Čižmárik J., Renčová M., Armstrong D.W.: Modification of the Chiral Bonding Properties of Teicoplanin Chiral Stationary Phase by Organic Additives. HPLC Separation of Enantiomers of Alkoxy-substituted Esters of Phenylcabamic Acid. *J.Liq. Chromatogr.* 24, 5, 609 – 624 (2001)
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