

BOOK OF ABSTRACTS

FLOW ANALYSIS XV

KRAKOW, POLAND 2022



JUNE 26 – JULY 1

CO-FINANCING



Ministry of Education
and Science

PATRONAGE

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JAGIELLONIAN UNIVERSITY
IN KRAKÓW

SCIENTIFIC



EuChemS
European Chemical Society
—Division of Analytical Chemistry—

MEDIA



ANALITYKA
NAUKA I PRAKTYKA

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WIEDZA I PRAKTYKA

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Historical summary of FLOW ANALYSIS conferences

I	1979	Amsterdam, the Netherlands
II	1982	Lund, Sweden
III	1985	Birmingham, UK
IV	1988	Las Vegas, USA
V	1991	Kumamoto, Japan
VI	1994	Toledo, Spain
VII	1997	Piracicaba, Brazil
VIII	2000	Warsaw, Poland
IX	2003	Geelong, Australia
X	2006	Porto, Portugal
XI	2009	Mallorca, Spain
XII	2012	Thessaloniki, Greece
XIII	2015	Prague, Czech Republic
XIV	2018	Bangkok, Thailand
XV	2022	Kraków, Poland

Welcome message

It is a great honour and pleasure to welcome you to the international scientific conference Flow Analysis XV, organized by the Faculty of Chemistry of the Jagiellonian University from June 26 to July 1, 2022 in Krakow, Poland.

Organized every three years by various research centers around the world, Flow Analysis conferences, have a long tradition as a platform for meeting of scientists presenting the latest advances, innovative concepts, and directions for the development of methods of flow analysis as well as for exchanging experiences and creating new scientific contacts. The 15th edition of the conference, organized exceptionally due to the Covid-19 pandemic four years after the previous edition, is a good opportunity to assess the current position and the contribution of flow analysis to the development of modern analytical chemistry.

Together with colleagues who contributed to the organization of the conference, I am glad to have among us outstanding scientists – conference lecturers, who have accepted our invitations. The scientific programme of the conference will include opening, plenary, keynote, invited, and closing lectures as well as oral, also of Young Scientists, and poster presentations. The best presentations of Young Scientists will be awarded by the Organizing Committee and by the President of the Committee of Analytical Chemistry, Polish Academy of Sciences.

Traditionally, in addition to the scientific programme, the conference includes also a social programme. During this edition we cordially invite participants to join welcome party and the conference banquet as well as to Krakow Old Town or Kazimierz sightseeing, excursion to "Wieliczka" Salt Mine, and also visiting Royal Private Apartments and Cathedral on the Wawel Hill.

We acknowledge the Editors of *Analytica Chimica Acta* for they agreement to continue the conference tradition by organizing the Special Issue featuring the selected papers from the conference, entitled: *Advances in Flow Analysis*. Selected papers from Flow Analysis XV, Krakow 2022. We invite participants to submit research or review manuscripts. All submissions will go through normal peer review process per journal standards. The submission portal will be opened from July 7 to November 7, 2022.

We also need to share some sad news with you. With deep sorrow we received the messages about passing away Professors José L.F. Costa Lima and Miguel Valcárcel Cases, outstanding experts in the field of analytical chemistry, who contributed significantly to the development of methods of flow analysis, organizers of previous editions of the Flow Analysis conference, Flow Analysis X and Flow Analysis VI, respectively. In the Book of Abstracts, we have included a chapter In Memoriam, in which, with the kind approval of the Brazilian Journal of Analytical Chemistry, you can



find two articles by Professors Elias A.G. Zagatto, and Marco A.Z. Arruda dedicated to Professors José L. F. Costa Lima and Miguel Valcárcel Cases.

We would like to take this opportunity to thank for taking the honorary patronage over the conference by the Rector of the Jagiellonian University prof. dr hab. Jacek Popiel, the scientific patronage by the European Chemical Society, Division of Analytical Chemistry and the Committee of Analytical Chemistry of the Polish Academy of Sciences, and the media patronage by Analityka journal.

We also thank the Ministry of Education and Science, Poland for co-financing the conference by the Programme “Doskonała Nauka”, the Gold Sponsor of the conference – company Shim-pol Shimadzu and other sponsors of the conference – companies Skalar, Kendrolab and Merck, and the City of Kraków.

My sincere thanks to my colleagues and PhD students from the Department of Analytical Chemistry for their dedication and hard work during organization of this meeting. I really appreciate the assistance of each of you.

Finally, I would like to thank all participants from Poland and from other countries, who decided to come, sometimes despite difficult situation in their region relating to Covid-19 pandemic, to maintain the tradition of the on-site conferences.

I wish you satisfaction with your presentations, fruitful discussions, making valuable scientific contacts, enjoyable time and the best impressions during the social events as well as feeling the warm and friendly environment of Flow Analysis conference.

Joanna Kozak

Conference Chairwoman



GENERAL

information

International Steering Committee

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Victor Cerdà (Spain)

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Petr Solich (Czech Republic)
Norio Teshima (Japan)
Marek Trojanowicz (Poland)
Marcin Wieczorek (Poland)
Elias A. G. Zagatto (Brazil)

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Paweł Nowak
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Michał Woźniakiewicz
Ewelina Kowa
Karolina Mermer
Marcelina Rusin

Conference secretariat

<https://flowanalysis2022.confer.uj.edu.pl>



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Jagiellonian University, Faculty of Chemistry,
Department of Analytical Chemistry
Gronostajowa Street 2
30-387 Krakow, Poland

Conference venue

Sheraton Grand Krakow Hotel

Powiśle Street 7, 31-101 Kraków

<https://www.sheratongrandkrakow.com.pl/>

The conference will be held in the Sheraton Grand Krakow Hotel. All oral and poster presentations, sponsors' exhibitions, as well as the Welcome Party and lunches will be organized there.

The Conference Office in the Hotel will be open from Sunday to Friday during conference session hours.

Patronage

Honorary Patronage

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Kraków

Conference presentations

The Conference will consist of the following types of contributions.

Symbol	Type of presentation	Time [min]	
		presentation + discussion	
OL	opening lecture	30	5
CL	closing lecture	30	5
PL	plenary lecture	30	5
KL	keynote lecture	20	5
IL	invited lecture	17	3
SL	lecture of gold sponsor	20	5
OP	oral presentation	12	3
YSP	oral presentation in the session of Young Scientists	8	2
P	poster presentation	60	

All multimedia presentations which are to be displayed with the use of conference computers should be prepared at least in Power Point 2013. Participants are kindly requested to pass on their presentations to service staff at least half an hour before the session at during which they will be presented in order to copy them onto the conference computer.

The maximum dimensions of the posters: 80 x 120 cm (width x height). The poster should be put up on the morning of the presentation day and removed when the poster session has finished.

Special Issue

Participants are invited to submit manuscripts based on Flow Analysis XV presentations for possible publication in *Analytica Chimica Acta*. The journal is organizing the special issue: **Advances in Flow Analysis. Selected papers from Flow Analysis XV, Krakow 2022.**

We invite participants to submit **research articles** and encourage to prepare review articles. Opening submission portal: 7-Jul-22. Deadline for manuscript submission: 7-Nov-22

Venue

Krakow is the one of the oldest and largest cities in Poland. It is located in the southern Poland, on the Vistula River. For centuries Krakow was the capital of Poland, the seat of kings, attractive for great scientists and artists from all around the world.

For many people, thanks to its rich history, Krakow nevertheless represents a synthesis of all things Polish, connecting tradition with modernity. In the special atmosphere of the beautiful and mysterious streets of the Old Town and Kazimierz district you will find everything you need to allow you to escape from everyday life. Galleries full of exhibitions, cafes, pubs and restaurants: all of this is an integral part of any visit to Krakow. And all this is merely a modest part of what we can offer travellers seeking exciting destinations on the world map.

Each place here has its own history, and numerous interesting legends and tales related to them still survive in the inhabitants' memories. The renaissance Royal Castle at Wawel, the gothic St Mary's Basilica, the historical trade pavilions of the Cloth Hall, the former separate Jewish city of Kazimierz, and even the Nowa Huta district, absorbed by Krakow together with its socialist-realist, industrial architecture, are all places which make a visit to Krakow extremely worthwhile.

Social programme

Tuesday, June 28, 2022 (17.30 - 19.30)

Guided tour around the old town of Krakow or around the Kazimierz - the old Jewish quarter

Wednesday, June 29, 2022 (7.20 - 17.30)

Visiting Wieliczka Salt Mine

The Wieliczka Salt Mine is now an official Polish Historic Monument and a UNESCO World Heritage Site. The tourist route is nearly 3 km long underground corridors, 800 stairs and a descent to depth 135 m underground.

Visiting Private Royal Apartments and Cathedral on the Wawel Hill.





OVERALL

programme

Sunday, June 26, 2022

17.00 – 19.00

Registration

19.00 – 21.00

Welcome party,

Sheraton Grand Hotel, Powiśle 7, Krakow

Monday, June 27, 2022

08.00 – 9.00

Registration

09.00 – 09.15

Opening ceremony

09.15 – 11.10

SESSION I

11.10 – 11.30

 Coffee break

11.30 – 12.50

SESSION II

12.50 – 14.00

 Lunch break

14.00 – 15.10

SESSION III

15.10 – 15.30

 Coffee break

15.30 – 16.40

SESSION OF YOUNG SCIENTISTS I

16.40 – 17.00

 Coffee break

17.00 – 18.10

SESSION OF YOUNG SCIENTISTS II

19.00 – 21.00

International Scientific Committee Meeting

Tuesday, June 28, 2022

09.00 – 11.10

SESSION IV

11.10 – 11.30

 Coffee break

11.30 – 13.00

SESSION V

13.00 – 14.00

 Lunch break

14.00 – 15.40

SESSION VI

15.40 – 16.00

 Coffee break

16.00 – 17.00

POSTER SESSION

17.30 – 19.30

Krakow Old Town / Kazimierz sightseeing

Meeting place: Sheraton Grand Hotel, Krakow

Wednesday, June 29, 2022

7.20 – 17.30

One-day excursion Visiting “Wieliczka” Salt Mine and Royal Private Apartments, and Cathedral on the Wawel Hill. Meeting place: Sheraton Grand Hotel, Krakow

Thursday, June 30, 2022

09.00 – 11.10

SESSION VII

11.10 – 11.30

 Coffee break

11.30 – 13.05

SESSION VIII

13.05 – 14.10

 Lunch break

14.10 – 15.20

SESSION IX

19.00 – 22.00

Conference banquet

Pałac pod Baranami, Rynek Główny 27, Kraków
(Palace under the Rams, The Main Square 27, Krakow).
Meeting place: Sheraton Grand Hotel, Krakow
at 18.40

Friday, July 1, 2022

09.00 – 11.00

SESSION X

11.15 – 11.35

 Coffee break

11.35 – 12.30

CLOSING SESSION

12.10 – 12.30

Closing remarks

12.30 – 13.30

 Lunch



DETAILED

programme

Sunday, June 26, 2022

17.00 – 19.00 Registration

19.00 – 21.00 Welcome party, Sheraton Grand Hotel, Powiśle 7, Krakow

Monday, June 27, 2022

08.00 – 9.00 Registration

09.00 – 09.15 Opening ceremony

09.15 – 11.10

SESSION I

Purnendu Dasgupta, Paweł Kościelniak

09.15 OL Marek Trojanowicz

Impact of nanotechnology on progress of flow methods in chemical analysis

09.50 PL1 Manuel Miró, Enrique J. Carrasco, Michal Michalec, María Rosende, David J. Cocovi-Solberg

3D printed micro-FIA (3D- μ FIA): the fourth generation of flow analysis

10.25 KL4 Norio Teshima, Hiroya Murakami, Yoshinori Inoue

Solid-phase extractants with hydrophilic groups useful for flow analysis

10.50 IL1 Jaroon Jakmunee, Pijika Mool-am-kha, Nuntaporn Moonrungssee, Surin Saipanya

A novel flow injection electrochemical sensor based on CNT-Ni(OH)₂-CB/Nafion nanocomposites for evaluating xanthine oxidase inhibitory activity

11.10 – 11.30



11.30 – 12.50

SESSION II

Jaroon Jakmunee, Marcela Segundo

11.30 KL2 Magdalena Flont, Patrycja Sokołowska, Elżbieta Jastrzębska, Zbigniew Brzózka

Development of a functional three-dimensional model of cells in a flow-through microsystem to study a dynamic process in vitro

11.55 IL2 Łukasz Tymecki

As simple as possible but no simpler...

12.15 IL3 Maliwan Amatatongchai, Nongyao Nontawong, Purim Jarujamrus, Duangjai Nacapricha, Peter A. Lieberzeit

Smart electrochemical sensors for simultaneous determination of dual carcinogen oxidative stress biomarkers through core-shell molecularly imprinted polymers

12.35 OP1 Marek Dębosz, József Kozma, Radosław Porada, Marcin Wieczorek, Justyna Paluch, Róbert E. Gyurcsányi, Jan Migdalski, Paweł Kościelniak

3D-printed manifold integrating solid contact ion-selective electrodes dedicated to multicomponent urine analysis

12.50 – 14.00



14.00 – 15.10

SESSION III

Purim Jarujamrus, Sławomir Kalinowski

14.00 PL2 Victor Cerdà

Portable analytical photometric and fluorometric systems based on the use of 3D printed devices

- 14.35 OP2 Jarda Ruzicka
Flow analysis: journey from the continuous to programmable flow
- 14.40 OP3 Petr Chocholouš, Jarda Ruzicka
Advances in flow injection are based on flow programming
- 14.55 OP4 Michał Michalec, Łukasz Tymecki
Microcontrollers for flow analysis - DIY automation
- 15.10 – 15.30 ☕ Coffee break
- 15.30 – 16.40** **SESSION OF YOUNG SCIENTISTS I**
Maliwan Amatatongchai, Łukasz Tymecki
- 15.30 YSP1 Justyna Głowacka
Flow analysis system for photometric determination of lactate dehydrogenase activity
- 15.40 YSP2 Justyna Skoczek
Multicommutated flow analysis system for fluorometric determination of lipolytic activity
- 15.50 YSP3 Karolina Mermer, Justyna Paluch, Justyna Kościelniak, Małgorzata Tomaszewska, Joanna Kozak
Simple automated flow system for determination of lithium in pharmaceutical samples and human blood serum
- 16.00 YSP4 Alejandra Vargas, Julián Morales, Víctor Cerdà, Laura Ferrer, Edwin Palacio
Paper sensor-based method using a portable 3D-printed platform and smartphone assisted colorimetric detection for ammonia and sulfide monitoring in anaerobic digesters and wastewater

- 16.10 YSP5 Francisca T. S. M. Ferreira, António O. S. S. Rangel, Raquel B. R. Mesquita
On-hand tool for CKD monitoring – design of a couple of μ PADs for ammonium and urea determination in saliva
- 16.20 YSP6 Juliana I. S. Aguiar, Susana O. Ribeiro, Andreia Leite, Maria Rangel, António O. S. S. Rangel, Raquel B. R. Mesquita
Development of a microfluidic paper-based analytical device (μ PAD) for iron determination in water
- 16.30 YSP7 Akarapong Prakobkij, Suchada Sukapanon, Suticha Chunta, Purim Jarujamrus
A fully high-throughput microfluidic paper-based analytical device fabricated by using an in-house computer-controlled X-Y knife plotter implemented with the deposition of reagents via technical pen-plotting for point-of-care testing

16.40 – 17.00



17.00 – 18.10

SESSION OF YOUNG SCIENTISTS II

Burkhard Horstkotte, Petr Kubáň

- 17.00 YSP8 Izabela Lewińska, Miguel M. Erenas, Luis Fermín Capitán-Vallvey
Thread-based microfluidic sensor for non-invasive lithium monitoring
- 17.10 YSP9 Celestine Vubangsi Gemuh, Burkhard Horstkotte, Petr Solich
Functionalization of commercial hydrophilic-lipophilic balanced copolymer for automatic magnetic dispersive micro-solid phase extraction of surface water contaminants

- 17.20 YSP10 Anna Saldan, Małgorzata Król, Michał Woźniakiewicz, Paweł Kościelniak
Application of microemulsion electrokinetic capillary chromatography for the analysis of dyes extracted from polyester fibers
- 17.30 YSP11 Natalia Kaczmarczyk, Julita Ciżewska, Natalia Treder, Natalia Miękus, Alina Plenis, Piotr Kowalski, Anna Roszkowska, Tomasz Bączek, Ilona Olędzka
The effects of imidazolium-based ionic liquids on the separation efficiency of biogenic amines during MEKC analysis
- 17.40 YSP12 Iwona Biel, Michał Woźniakiewicz, Jekaterina Mazina – Šinkar
Application of the capillary electrophoresis technique in real life
- 17.50 YSP13 Natalia Treder, Ilona Olędzka, Anna Roszkowska, Piotr Kowalski, Tomasz Bączek, Alina Plenis
Dynamic coating of phenyl-silica based stationary phases with ionic liquids used as mobile phase additives in the LC technique
- 18.00 YSP14 Ewelina Kowa, Anna Telk, Marcin Wieczorek
The benefits of coupling flow injection systems to ICP-MS in the analysis of biological samples
- 19.00 – 21.00 International Scientific Committee Meeting

Tuesday, June 28, 2022

09.00 – 11.10

SESSION IV

Zbigniew Brzózka, Manuel Miró

- 09.00 PL3 Petr Solich, Burkhard Horstkotte, Hana Sklenářová,
Petr Chocholouš, Dalibor Šatínský

Flow techniques for sample pretreatment

- 09.35 PL4 Yanlin Zhang, Robert W. Catrall, Spas D. Kolev

*Polymer inclusion beads – a new tool for online
separation and preconcentration in flow analysis.
Determination of Au(III) by bead injection analysis*

- 10.10 KL3 Petr Kubáň, Vera Dosedelova, Petra Itterheimova,
Frantisek Foret

*Analysis of non-invasive biological samples
by microcolumn separation techniques for medical
diagnostics*

- 10.35 IL4 Hana Sklenářová, Martina Háková, Maite Aguinaga
Martínez, Burkhard Horstkotte, Petr Pávek, Manuel Miró,
Petr Solich

*Automation in sample manipulation for long-term
monitoring*

- 10.55 OP5 David Jaime Cocovi-Solberg, Stephan Schnidrig, Manuel
Miró, Stephan Hann

Heartcutless coupling of bead injection and HPLC

11.10 – 11.30

 **Coffee break**

11.30 – 13.00

SESSION V

Norio Teshima, Raquel Mesquita

- 11.30 SL Paweł Stalica
Miniaturization and integration of Shimadzu analytical tools. From balances to HRAM-DIA/DDA-MS/MS
- 11.55 KL1 Jean-Luc Boudenne, Robert-Peillard Fabien, Coulomb Bruno
3D-printing contributions to design of extraction modules
- 12.20 IL5 Petr Chocholouš, Matej Gužiňák, Kristýna Pavelková, Dalibor Šatínský, Petr Solich
Second dimension in sequential injection chromatography
- 12.40 IL6 Burkhard Horstkotte, Sercan Yıldırım, Kateřina Fikarová, Ivana H. Šrámková, David J. Cocoví-Solberg, Hana Sklenářová, Petr Solich
Lab-in-syringe automated sample preparation for HPLC
- 13.00 – 14.00  Lunch break

14.00 – 15.40

SESSION VI

Jean-Luc Boudenne, Hana Sklenarova,

- 14.00 PL5 Elias A. G. Zagatto, Fabio R. P. Rocha
Revisiting chemical derivatization in flow analysis
- 14.35 PL6 Marcela A. Segundo
Bead injection and sample preparation: a fruitful marriage?
- 15.10 OP6 Paweł Knihnicki, Karolina Reizer, Jolanta Kochana
Flow system for D3 vitamin electrochemical determination in pharmaceutical formulations

15.25 OP7 Tânia C. F. Ribas, Raquel B. R. Mesquita, António O.S.S. Rangel

Use of dimethylglyoxime as a chromogenic agent in a flow-based system for the determination of Ni(II) in recreational water

15.40 – 16.00

 Coffee break

16.00 – 17.00

POSTER SESSION

Petr Chocholouš, Anna Telk

P1 Wanida Loetsaksrisakul, Natcha Kaewwonglom, Jantima Upan, Sam-ang Supharoek, Jaroon Jakmunee

Exploiting flow injection titration with CO₂ exclusion to determine free fatty acids at low concentrations

P2 Natcha Kaewwonglom, Somkid Pencharee, Suphakit Awiphan, Tinakorn Kanyanee, Jakarin Chawachate, Jeerayut Chaijaruwanich, Jaroon Jakmunee

A mono-segmented flow in hydrodynamic sequential injection system for online monitoring of ammonium in aquaculture

P3 Natcha Kaewwonglom, Miquel Oliver, David J. Cocovi-Solberg, Katharina Zirngibl, Dietmar Knopp, Jaroon Jakmunee, Manuel Miró

Kinetic flow-based method for plasmonic sensing of ultratraces of diclofenac in seawater

P4 Lea Kukoc-Modun, Maja Biocic, Josipa Dugeč, Njegomir Radić

Design of flow injection spectrophotometric system for penicillamine determination in pharmaceutical formulations

P5 Agnieszka Czajkowska, Dorota Korsak, Marta Fiedoruk-Pogrebniak, Robert Koncki, Kamil Strzelak

Flow analysis system for the control of microbial growth

- P6 Justyna Paluch, Karolina Mermer, Joanna Kozak
Determination of acid dissociation constant of selected biologically active compounds using a flow system with spectrophotometric detection
- P7 Marek Kozak, Joanna Kozak
Flow system for sulfur speciation analysis in bioethanol with ICP OES detection
- P8 Karolina Mermer, Justyna Paluch, Maciej Pitra, Joanna Kozak
Flow system for green and integrated sample pretreatment for the determination of antiviral drugs in surface waters
- P9 Karolina Mermer, Justyna Paluch, Daniel Kielbasa, Marek Kozak, Sławomir Kalinowski, Joanna Kozak
Automated flow system with a direct injection cell for simultaneous determination of Fe(II) and Fe(III) in beverages
- P10 Ana Machado, Adriano A. Bordalo, Raquel B. R. Mesquita
Sequential injection method for the biparametric determination of iodide and creatinine in urine
- P11 Maria J. M. Nunes, Raquel B. R. Mesquita, Tânia C. F. Ribas, António O. S. S. Rangel
Sequential injection method for the determination of macro and micro nutrients in soils
- P12 Andriy Vishnikin, Arina Skok, Yaroslav Bazel, Mohammed Al-Shwaiyat
Application of sequential injection lab-at-valve analysis to simultaneous spectrophotometric and/or kinetic determination of reducing agents, Si(IV) and P(V)
- P13 Alejandra Vargas, Víctor Cerdà, Gemma Turnes, Edwin Palacio
Determination of long-chain fatty acids in anaerobic digester supernatant and olive mill wastewater exploiting

- an in-syringe dispersive liquid-liquid microextraction and derivatization-free GC-MS method*
- P14 Petr Chocholouš, Jan Vinklársek, Eva Semerádová, Petr Solich
Determination of creatinine in urine using online solid-phase extraction
- P15 Sumonmarn Chaneam, Petcharat Sirisakwisut, Thanikarn Sukaram, Nunnicha Janthon, Benjaporn Theerawutthisart, Jitnapa Sirirak, Apisake Hongwitayakorn
Green analytical methods for quantitative analysis using natural reagent: Application of orchid flower extract as acid-base indicator and as specific reagent for copper ion analysis in flow-based system
- P16 Lucie Krizova, Nikola Mannova, Denisa Smela, Zuzana Bílková, Burkhard Horstkotte, Zuzana Svobodová
Lab-in-syringe for automated miniaturized preparation of magnetic immunosorbent for SARS-CoV-2 isolation
- P17 Sercan Yıldırım, Kateřina Fikarová, Burkhard Horstkotte, Petr Solich
Lab-in-syringe automated tandem implementation of protein precipitation and homogenous liquid-liquid microextraction for the determination of beta-blockers in serum
- P18 Mateusz Stawicki, Łukasz Tymecki
Thermoforming as a tool for fluidic systems fabrication - pros and cons
- P19 Purim Jarujamrus, Thanyanat Saiboh, Nutthaporn Malahom, Wipark Anutrasakda, Sanoe Chairam, Maliwan Amatatongchai
Naked eye quantification of formalin in food samples by using a microfluidic thread-based analytical device (μ TAD)
- P20 Marcin Wieczorek, Marek Dębosz, Ewelina Kowa, Justyna Paluch, Jan Migdalski, Bogusław Baś, Paweł Kościelniak

The use of 3D printing technique for the construction of miniature potentiometric cells and flow injection systems for multi-component water analysis

P21 Małgorzata Gołąb, Małgorzata Majewska, Martyna Przybyłowska, Michał Woźniakiewicz

Determination of tropane alkaloids using portable CE-C⁴D system

P22 Marcelina Rusin, Aneta Woźniakiewicz, Justyna Dobrowolska-Iwanek, Agnieszka Zielińska, Michał Woźniakiewicz

Development of human milk oligosaccharides analysis using capillary electrophoresis with mass spectrometry (CE-MS)

P23 Petra Itterheimová, Petr Kubáň, František Foret

Open-source arduino-based data acquisition devices

P24 Jelena Gorbatšova, Vyacheslav Bolkvadze, Merike Vaher, Evelin Halling, Jekaterina Mazina-Šinkar

Cannabinoids (Δ^9 -THC and CBD) quantitative analysis by in-situ portable drug analyzer Drug Hunter

P25 Martin Růžička, Mihkel Kaljurand, Jelena Gorbatšova, Merike Vaher, Evelin Halling, Martin Jaanus, Jekaterina Mazina-Šinkar

The development of handheld automatic drugmeter for quantitative analysis of γ -hydroxybutyric acid

P26 Aneta Woźniakiewicz, Małgorzata Gołąb, Michał Woźniakiewicz

Isolation and determination of selected hydrophobic components found in the nutmeg. Comparison of methods

P27 Alicja Chromiec, Klaudia Ordon, Renata Wietecha-Postuszny

Application of the SPME / CE-MS method for the determination of popular drugs in the blood

P28

Paweł Świt, Joanna Orzeł

Application of new methodological approaches for simultaneous determination of bisphenols in receipts using HPLC-DAD

17.30 – 19.30

Krakow Old Town / Kazimierz sightseeing

Wednesday, June 29, 2022

7.20 – 17.30

One-day excursion

Visiting “Wieliczka” Salt Mine and Royal Private Apartments, and Cathedral on the Wawel Hill

Thursday, June 30, 2022

09.00 – 11.10

SESSION VII

Peter C. Hauser, Spas Kolev

09.00

PL7 Wolfgang Frenzel

The role of flow injection analysis and related techniques in atmospheric research and air pollution surveillance

09.35

PL8 António O. S. S. Rangel, Raquel Mesquita

Targeting the determination of iron using various flow-based approaches

10.10

KL5 Orawon Chailapakul, Yosita Panraksa, Suchanat Boonkaew, Charles S. Henry

The capillary-driven microfluidic device for point-of-care testing: Applications for biomarkers analysis

- 10.35 IL7 Raquel B. R. Mesquita, António O. S. S. Rangel
Tackling sample complexity in microfluidic paper-based devices
- 10.55 OP8 Akhmad Sabarudin, Noval Dwi Wijaya, Setyawan Purnomo Sakti, Aulanni'am, Hani Susianti, Nur Samsu, Agus Naba, Ika Oktavia Wulandari, Yudit Oktanella, Dewi Anggraeni
Instrumentation-free approach to rapid detection of albumin-to-creatinine ratio using paper-based analytical devices
- 11.10 – 11.30 ☕ Coffee break
- 11.30 – 13.05** **SESSION VIII**
Wolfgang Frenzel, Akhmad Sabarudin
- 11.30 KL6 Duangjai Nacapricha, Paithoon Prasertying, Thita Ninlapath, Nanthatchaphon Jantawong, Thinnapong Wongpakdee, Thitaporn Sonsa-ard, Kanchana Uraisin, Phoontawee Saetear, Prapin Wilairat
Exploiting vertical flow in paper material for the fabrication of a unique microfluidic lid for reproducible addition of a liquid reagent
- 11.55 IL8 Purim Jarujamrus
Nitrogen-Doped Carbon Dots (N-CDs) as peroxidase mimic coupled with a laminated three-dimensional microfluidic paper-based analytical device (laminated 3D-μPAD) for smart sensing of total cholesterol from whole blood
- 12.15 IL9 Kamil Strzelak
Different approaches to nonenzymatic lactate determination in body fluids - the development of flow analysis system and paper-based analytical device

- 12.35 OP9 Joana L. A. Miranda, Raquel B. R. Mesquita, Edwin Palacio, José M. Estela, Víctor Cerdà, António O. S. S. Rangel

Chip-based spectrofluorimetric determination of iodine in a multi-syringe flow platform with and without in-line digestion

- 12:50 OP10 Phoonthawee Saetear, Nattinee Saechua, Kamonthip Sereenonchai

Sequential injection system for analysis of degree brix, orthophosphate and pH in raw sugarcane juice applicable to sugar industry

13.05 – 14.10  Lunch break

14.10 – 15.20

SESSION IX

Petr Solich, Marek Trojanowicz

- 14.10 KL7 Peter C. Hauser, Jasmine S. Furter, Marc-Aurèle Boillat

Purpose made capillary electrophoresis instrumentation

- 14.35 OP11 Michał Woźniakiewicz, Małgorzata Gołąb, Paweł M. Nowak, Paweł Kościelniak

An automated hydrodynamically mediated technique for preparation of calibration solutions via capillary electrophoresis system

- 14.50 OP12 Paweł M. Nowak, Maria Klag, Gabriela Kózka, Michał Woźniakiewicz

Development of a method for determining the acid dissociation constant by combining the capillary electrophoresis with microscale thermophoresis in the on line flow format (CE-MST)

- 15.05 OP13 Justyna Dobrowolska-Iwanek, Paweł Paško, Sviatoslav Grekh, Małgorzata Jamka-Kasprzyk, Anna Jurczak

Development of organic acids determination in saliva using isotachopheresis technique (ITP)

19.00 – 22.00

Conference banquet

Pałac pod Baranami, Rynek Główny 27, Kraków
(Palace under the Rams, The Main Square 27, Krakow)

Friday, July 1, 2022

09.00 – 11.00

SESSION X

Victor Cerda, Antonio Rangel

- | | | |
|-------|------|---|
| 09.00 | PL9 | <u>Paweł Kościelniak</u>
<i>White calibration methods in flow analysis</i> |
| 09.35 | KL8 | Kanokwan Kiwfo, Pei Meng Woi, Chalermpong Saenjurn, Siripat Suteerapataranon, Gary D. Christian, Ian D. McKelvie, Spas D. Kolev, <u>Kate Grudpan</u>
<i>Natural reagents for chemical analysis: consideration with United Nations Sustainable Development Goals</i> |
| 10.00 | OP14 | <u>Sumonmarn Chaneam</u> , Tananun Phongsutthisombat, Sirinya Panatta, Nicharee Yensuksiri, Peeraphon Thienwong, Apisake Hongwitayakorn, Duangjai Nacapricha
<i>Development of sequential injection analysis with cost-effective optical device based on LED colorimeter for simultaneous analysis of zinc and copper in dietary supplements</i> |
| 10.15 | OP15 | <u>Sławomir Kalinowski</u> , Bartosz Libeck
<i>Automatic flow system for determination of chemical oxygen demand (COD) with a two-beam photometric detector</i> |
| 10.30 | OP16 | <u>Ryoichi Ishimatsu</u>
<i>Spectroelectrochemical determination of the rate constant of protonation reactions for some radical anions</i> |

10.45	OP17	<u>Paweł Świt</u> , Joanna Orzeł, Sławomir Maślanka <i>Determination of EPA PAHs in simulated natural and artificial fires by HPLC-DAD-FLD with the application of the Multi-Component Integrated Calibration Method</i>
11.00	OP18	Chris Davies, <u>Agatha Grela</u> , Joanne Morrissey, Havovi Chichger, Lata Gautam <i>Quantification of diclazepam and its metabolites using GC-MS for chemometric purposes</i>
11.15 – 11.35		 Coffee break
11.35 – 12.30		CLOSING SESSION Joanna Kozak, Marcin Wieczorek
11.35	CL	<u>Purnendu Dasgupta</u> <i>Open tubular ion chromatography. The trials and tribulations to approach small</i>
12.10		Closing remarks
12.30 – 13.30		 Lunch





ABSTRACTS



**of opening & closing
lectures**



Marek Trojanowicz

Institute of Nuclear Chemistry and Technology
Laboratory of Nuclear Analytical Methods,
Warsaw, Poland

Marek Trojanowicz is Professor of Chemistry in the Institute of Nuclear Chemistry and Technology in Warsaw, Poland, and Professor Emeritus of the Department of Chemistry, University of Warsaw. His expertise includes the flow analysis, liquid chromatography and capillary electro-phoresis, a design of electrochemical sensors and biosensors, the application of ionizing radiation for water and waste treatment and application of chemical analysis in archaeometry. He was granted Visiting Professor positions in numerous universities all over the world including long-term appointments in University of Michigan (USA), University of California at Riverside (USA), University of Tasmania (Australia), Okayama University (Japan) and University of Sao Paulo (Brazil). He is author of 370 scientific publications, 2 monographic books and editor of 4 books in the field of the flow analysis and automation of analytical measurements. He is a member of the advisory boards of 10 international analytical journals. His current number of citations according to ISI Web of Knowledge is 7300, and Hirsch index is 46.

Impact of nanotechnology on progress of flow methods in chemical analysis

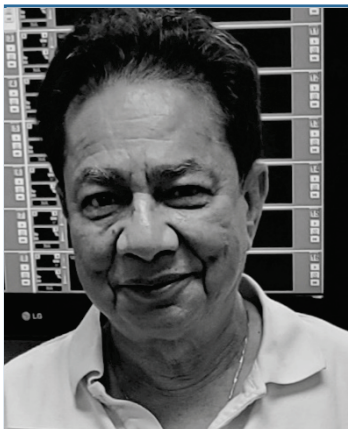
Marek Trojanowicz

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The history of modern analytical chemistry consists of numerous crucial events and development stages, which are influenced by the progress in various areas of science and technology. Most commonly it is effect of the progress in material science, electronics, mechanics, or informatics and computer science. Following the fundamental breakthrough of instrumentation of analytical procedures, a particular mark was made by a common introduction of electronics, and then microprocessors and computerization. Further development stages include miniaturization of instrumentation, automation and robotics, while first decades of 21st century can be characterized by an increasing contribution of the nanotechnology generally in chemical sciences, but also in the analytical chemistry.

The beginnings of contemporary nanochemistry should be probably dated back to second half of 19th century and developments in the colloidal sciences, and also investigation of the formation of monolayers on the solid surfaces or interphase boundaries. The properties of nanoparticles are affected by their size, chemical composition, crystal structure, chemistry of surface processes and the agglomeration state. A very intense studies on synthesis and properties of nanoparticles and nanostructures, is also reflected by increasing number of their applications in flow analysis.

The application of a planar nanostructures, such as SAMs or BLMs is employed in the immobilization of biomolecules/bioreceptors in biochemical methods, may improve the selectivity and sensitivity of analytical determinations. The application of liposomes involves the improvement of numerous immunochemical procedures. The carbonaceous and metallic nanoparticles are used as catalysts of various reactions, allow to develop a novel detection methods and can be efficiently employed for labelling of analytes. It is very advantageous to use them, especially after suitable functionalization, and also as magnetic nanoparticles, for improvement of separation processes in flow analytical procedures. The quantum dots with appropriate functionalization are also widely used for the development of novel selective and sensitive detection methods.



Purnendu K. (Sandy) Dasgupta

University of Texas at Arlington,
College of Science, Department of Chemistry,
Arlington, Texas, USA

Purnendu K. (Sandy) Dasgupta got his honors in Chemistry BSc degree, and MSc in Inorganic Chemistry from India and his PhD in Analytical Chemistry with a minor in Electrical Engineering) from Louisiana State University (LSU) in 1977 - while he was getting a PhD, he also earned a diploma as a TV mechanic. Sandy worked for a year as an instructor at LSU before joining the University of California at Davis to study inhalation toxicology of air pollutants. Sandy says he has never been quite sure what to do. In Bengali, his mother tongue, he is a published poet and a novelist. But after his father and grandfather he is a third generation University Professor. After 25 years at Texas Tech, he joined the University of Texas at Arlington where he is presently the Hamish Small Chair in Ion Analysis. He has authored 450+ papers/book chapters and was primary person to update the last edition of Christian's undergraduate text on Analytical Chemistry. More than 250 of his papers are in Analytical Chemistry and Environmental Science and Technology. He is a significant inventor, the majority of his 38 patents have been licensed. His work in chromatography been recognized by the ACS Award in Chromatography, Dal Nogare Award in Separation Sciences, the Giorgio Natta medal in Open tubular liquid chromatography. J Calvin Giddings Award in Chemical Education, Eastern Analytical Symposium "Fields of Analytical Chemistry" Award, etc. He is an IEEE fellow. His work on environmental perchlorate led to public awareness and eventual regulation. In 2018 he received the Texas Distinguished Scientist award from the Texas Academy of Science, first Asian American to be so honored. In the same year he received the Talanta Medal in Analytical Chemistry, the medal has been given since 1960, he is the fourth American to receive it. He received the ACS Award in chemical Instrumentation in 2018.

He says he likes to think of himself as a problem solver rather than a specialist. Above all, he likes building things: His website proclaims: We foster builders, not users.

Open tubular ion chromatography. The trials and tribulations to approach small

Purnendu K. Dasgupta

University of Texas at Arlington, Arlington, TX 76019-0065, USA

Hamish Small. Who invented ion chromatography in the early 70's, passed away in August, 2019. He was a prolific scientist and invented many other things, including hydrodynamic chromatography, indirect photometric detection in separations, thermal time of flight flowmetry, etc. I am fortunate to have known him personally. I am also fortunate that I occupy a chair named after him. This work is a tribute to Hamish Small.

Small is Greener. Small is higher performance. But getting to be small is not easy. There are also sacrifices to be made. If we make columns to be sufficiently small to get the highest performance, it is hard to make detectors that can see that performance or injectors and suppressors that can maintain that performance. There are four cornerstones to ion chromatography: Sample preparation and injection, Separation (column preparation), Eluent Suppression, and Detection. If you really get each of these things to be small, then connecting them all together and retaining that smallness becomes the hardest challenge. I started on a quest of open tubular ion chromatography more than two decades ago. In this presentation, I will describe where we presently are.

Acknowledgments

NASA MaTISSE grant 80NSSC19K0805, NSF CHE-2003324, Thermo Fisher Scientific, and University of Texas at Arlington.





ABSTRACTS



of plenary lectures



Manuel Miró

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Dr. Manuel Miró is Full Professor in Analytical Chemistry at the University of the Balearic Islands since 13th September 2017 at the age of 42. Since 2012 he is the Principal Investigator (IP) of the research group FI-TRACE (Flow Injection and Trace Analysis) at UIB. Since 2007 he is the Reviews Editor of *Analytica Chimica Acta* by Elsevier (IF:6.558, 2020) and IUPAC member (subcommittee VI) on Chemical and Biophysical Process in the Environment. Last year, he was appointed core member of the European Working Group on Sample Preparation from EuChemS-DAC (Division of Analytical Chemistry). He is also one of the four Associate Editors of the 3rd Edition of the *Encyclopaedia of Analytical Sciences*, Elsevier, since 2017. He has also been recently appointed Executive Editor of the journal *Analytical Science* by Springer.

He has published 210 peer-reviewed publications including 13 book chapters in International Publishers and one preface since 2000, from which 24 articles in *Analytical Chemistry* (IF: 6.986) from the American Chemical Society are worth mentioning. He has published 166 articles in Q1 journals and 45 articles in D1 (first decile) in the fields of analytical chemistry and environmental science. It should be added that he has been the corresponding author of 105 articles. His publication record shows a Hirsch (h) Index=40, a total number of citations of 5350, and an average citations/article of 26.

He has given 67 oral communications (52 as invited (Plenary or Keynote) and 6 as Opening Lecturer) in international conferences. He has collaborated with researchers from 65 different national, European and International universities. Over the last 10 years, Dr. Miró has supervised 10 PhD thesis at National and International Universities (1 co-supervised at Technical University of Denmark, 2 at the Universidade Federal de Bahia (Brazil), 1 at Chiang Mai University (Thailand) and 4 at UIB. He has participated in 33 national and international science research projects, with 17 projects as the Principal Investigator.

3D printed micro-FIA (3D- μ FIA): the fourth generation of flow analysis

Manuel Miró¹, Enrique J. Carrasco², Michal Michalec³, María Rosende¹, David J. Cocovi-Solberg⁴

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Additive manufacturing, so called 3D printing, is an emerging enabling technology that can facilitate the production of complex and unique 3D structures in various fields of analytical science, including the flow injection and the microfluidic arenas. In this invited lecture, the main 3D printing technologies (viz., stereolithography (SLA), light digital processing (LDP), photopolymer inkjet printing (PIP), and fused deposition modeling (FDM) that triggered the second dawn of FIA, Sequential Injection analysis (SIA) and Lab-on-valve (LOV) approaches by the incipient 3D- μ FIA concept as the fourth generation of flow analysis [1,2] are described in detail. This is followed by pinpointing the potential of 3D printing for the fabrication of cost-effective, versatile and fit-for purpose components of FIA setups, that is, rotary and hybrid valves and LOV stators, mixers and reactors of unique shapes, separation columns and detection systems as a result of the easiness for fast prototyping by iterative changes via CAD modelling, and the plethora of printable materials available at present. Special attention is given also to the role of 3D printing in manufacturing FIA/SIA/LOV platforms integrating on-line sample preparation and microextraction approaches [3,4] with the added advantage of in-situ printing of dialytic/gas diffusion membranes and sorptive phases by exploiting raw or composite materials as demonstrated by model applications in the environmental and bioanalytical fields.

References

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Acknowledgments

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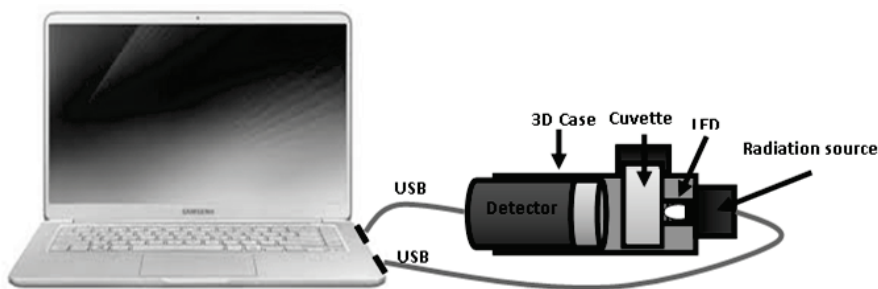
Dr. Víctor Cerdà studied Chemistry at the Barcelona University. He received his PhD degree in 1973 at the same institution. On 1982 he obtained the position of a Full Professor in the University of the Balearic Island, where he was head of the Chemistry Department, Vice-chancellor of Scientific Policy, Vice-president of the Spanish Society of Analytical Chemistry. Founder and President of the Association of Environmental Analytical Chemistry and of the Sciware Systems as a Spin Off company of this university. He has published 14 books, more than 610 research articles in SCI (E) journals, and more than 850 contributions in international conferences. Has been the leader of more than 40 research projects. Has organized more than 14 conferences.

Portable analytical photometric and fluorometric systems based on the use of 3D printed devices

Víctor Cerdà

Sciware Systems, 07193 Bunyola, Spain

The design of several 3D printed devices and some electronic boards, combined with the use of digital imaging detectors, has allowed to build different kinds of portable and cheap photometric systems. 3D printed devices are used to hold the detectors, usual and flow cells, and the light source. Detectors and light sources are powered through two USB of a notebook, avoiding the need of extra power sources. Several kind of image detectors have been tested: webcam [1], digital microscope [2, 3], and CCDs [4, 5]. They have been used for colorimetric, spectrophotometric and fluorimetric determinations of different kind of samples. Data treatment are manually managed with different software packages, like YouCam, ImageJ, Chemostat, and automatically using AutoAnalysis.



References

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Petr Solich

Charles University, Faculty of Pharmacy,
Department of Analytical Chemistry,
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Appointed as Professor of Analytical Chemistry at Faculty of Pharmacy, Charles University, CZ in 2007. Lecturer and examiner in courses of Analytical Chemistry, Instrumental methods of analysis, Special methods of instrumental analysis and Selected separation methods. Supervisor of > 80 Bc. and MSc. (Pharmacy and Medical Bioanalytics) diploma theses, supervisor of > 12 international Erasmus students, tutor of > 27 Ph.D. students (25 already successfully finished), mentor of > 7 post-doctoral research associates. Research activities are focused on Flow methods (Flow Injection Analysis, Sequential Injection Analysis, Sequential injection chromatography, HPLC, UHPLC) and application in pharmaceutical and environmental analysis; Automation of analytical procedures; Development and validation of novel LC methods; Application of novel trends in stationary phases in UHPLC (monolithic columns, sub-2-micron columns); Development of new separation methods for analysis of markers in biological materials; Environmental analysis of pharmaceuticals in soil and waste water. He was the principal investigator of many grants, currently is the guarantor of two huge projects of excellence - EFSA-CDN and STARSS (in total about 15 mil. EUR). For dozens of years cooperates with many prestigious universities worldwide. Until now has published over 280 impacted papers with more than 6 300 citations (without self-citations), h-index 42.

Flow techniques for sample pretreatment

Petr Solich, Burkhard Horstkotte, Hana Sklenářová, Petr Chocholouš, Dalibor Šatínský

Charles University, Faculty of Pharmacy, Department of Analytical Chemistry, Hradec Králové, Czech Republic

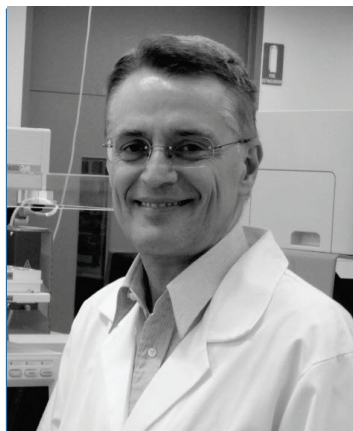
Recently, sample preparation has become of great interest to an increasing number of scientists and many attempts have been undertaken to automate such preparation procedures. The automation of the sample pretreatment step, in particular analyte extraction, enhances procedural repeatability, decreases the risk for human errors, and minimizes the need for manipulation with organic solvents and other reagents. To automate different extraction procedures, flow techniques as highly versatile tools, above all Sequential Injection Analysis (SIA), and applying the principles of flow methods on other platforms, have been the main approaches used for this task. In this field, various extraction methodologies have been developed based on liquid-liquid extraction in its basic, miniaturized form, and also in dispersive format. Modified SIA systems have been used for their applications in the field of environmental and biological samples analysis. Sample pretreatment using solid phase extraction is based on universal or selective sorbents including restrictive access materials, molecularly imprinted polymers, and nanomaterials. Extraction procedures have been automated in SIA systems with sorbents used in the form of preppacked micro-columns or taking advantage of the Bead-Injection approach, i.e. using a sorbent suspension as a renewable packing material in the miniaturized SIA-related Lab-On-Valve format. Lately, 3D printing technology was used to develop new flow system components including devices that allow easy and robust handling of polymer nanofibers as novel sorbents for solid phase extraction. Head-space extraction was also developed in the single-drop format for the analysis of volatile analytes using a second SIA-related approach Lab-in-syringe [1]. All these sample pretreatment methods can be used in combination with on-line determination using the same flow system or hyphenated with chromatographic systems. Outstanding examples will be discussed to demonstrate how flow techniques can improve the sample pretreatment and preparation step.

References

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Acknowledgments

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Spas D. Kolev

The University of Melbourne, Faculty of Science,
School of Chemistry,
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Spas Dimiitrov Kolev graduated from Sofia University “St. Kliment Ohridski” (Bulgaria) and completed his PhD degree at the Budapest University of Technology and Economics (Hungary). He is an Honorary Professor of Chemistry in the School of Chemistry and in the Department of Chemical Engineering of the University of Melbourne and Fellow of the Royal Australian Chemical Institute.

Most of his research is focused in the areas of flow analysis techniques, including paper-based microfluidics; chemical sensors, development of polymer inclusion membranes for separation or synthesis of metallic nanoparticles; and phytoremediation and phytomining. He has published 219 refereed journal articles, 7 book chapters and has co-edited a book entitled ‘Advances in Flow Injection Analysis and Related Techniques’ (Elsevier, 2008). He has delivered over 60 invited, keynote or plenary lectures at international conferences.

Spas Kolev is the Founding Editor-in-Chief and current Editor-in-Chief of the journal *Membranes* (Impact Factor 4.106) and member of the editorial or advisory boards of *Analytica Chimica Acta*, *Talanta*, *Sensors*, *Environmental Modeling and Assessment*, and *International Journal of Analytical Chemistry*.

He is the recipient of the Medal of the Japanese Association for Flow Injection Analysis (2010), Lloyd Smythe Medal (2009) and the Environmental Chemistry Medal (2017) (both from the Royal Australian Chemical Institute), Ronald Belcher Memorial Award (1988, *Talanta*), Max O'Connor Prize (2017, La Trobe University, Australia), and Grimwade Prize in Industrial Chemistry (2013, The University of Melbourne).

Polymer inclusion beads – a new tool for online separation and preconcentration in flow analysis. Determination of Au(III) by bead injection analysis

Yanlin Zhang¹, Robert W. Cattrall¹, Spas D. Kolev^{1,2,3}

¹School of Chemistry, The University of Melbourne, Melbourne, Australia

²Department of Chemical Engineering, The University of Melbourne, Melbourne, Australia

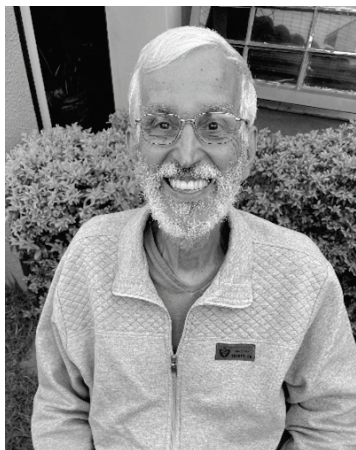
³Department of Applied Inorganic Chemistry, Faculty of Chemistry and Pharmacy, Sofia University “St.Kliment Ohridski”, Sofia, Bulgaria

Micro core-shell polymer inclusion beads (μ CSPIBs), composed of poly(vinyl chloride) (PVC) as the base polymer, Aliquat 336 (a mixture of quaternary alkylammonium chlorides) as the extractant/carrier and 1-tetradecanol as a plasticizer, were fabricated by a novel microfluidic technique and used for improving the selectivity and sensitivity of Au(III) determination in a bead injection analysis (BIA) system. The analytical reaction was based on the catalytic effect of Au(I) on the reduction of phosphomolybdic acid by thiourea.

The analytical procedure involved loading the separation column of the BIA system with μ CSPIBs where Au(III) was separated from the sample matrix as a result of its extraction in the form of $[\text{AuCl}_4]^-$ by Aliquat 336 into the μ CSPIBs. This was followed by stripping the extracted Au(III) from the μ CSPIBs into a receiving solution of thiourea where Au(III) was reduced to Au(I). The receiving solution was then mixed with a solution of phosphomolybdic acid which was reduced by thiourea to phosphomolybdenum blue and the latter was detected spectrophotometrically.

The BIA method was optimized and under optimal conditions it was characterized by a limit of detection of $8.6 \mu\text{g Au L}^{-1}$ and a sampling rate of 5 h^{-1} . Excellent selectivity in the presence of transition metal ions encountered in electronic waste was observed.

The newly developed BIA method was successfully applied to the determination of Au in aqua regia digests of electronic waste. These results were in good agreement with those obtained by inductively coupled plasma-optical emission spectrometry.



Elias A. G. Zagatto

University of Sao Paulo,
Centre for Nuclear Energy in Agriculture,
Division of Nuclear and Analytical Techniques,
Piracicaba SP, Brazil

Elias Ayres Guidetti Zagatto is Agronomical Engineer and M.Sc. in Nuclear Energy in Agriculture by the University of Sao Paulo (1971 and 1974) and Ph.D. in Analytical Chemistry by the State University of Campinas (1981). He participated in internships at the Technical University of Denmark (Lyngby DK, 1975-76) and at the Max-Planck Institute for Metal Research (Schwäbisch Gmünd DE, 1981) under supervision of J. Ruzicka and G. Tölg, respectively. He is currently Full Professor at the University of Sao Paulo, and a Titular Member of the Brazilian Academy of Sciences.

In 1975, he participated in the pioneering implementation of flow injection analysis to real analysis, nowadays recognized as an historic moment in the progress of Analytical Chemistry. Since then, his research has been focused on conceptual, methodological and applicative developments of flow-based strategies for large-scale analysis of agronomic, industrial, pharmaceutical and environmental samples.

The relevance of his research has been demonstrated by the numbers of published articles (192), meaning 6581 citations ($h = 44$, May 2022), books (4), book chapters [6], academic supervisions (64), patents (3), presentations at events, scientific collaborations, awards, etc.

Throughout his career, emphasis has been placed on valuing the ethical, moral and spiritual aspects, aiming to develop, among students, colleagues and collaborators, the feelings of team spirit, intellectual freedom, creativity and, above all, friendship.

Revisiting chemical derivatization in flow analysis

Elias A. G. Zagatto, Fábio R. P. Rocha

University of Sao Paulo, Center for Nuclear Energy in Agriculture, Piracicaba SP, Brazil

Chemical derivatization is usual for improving selectivity and sensitivity in flow analysis [1], and relevant aspects such as no need to reach steady state, kinetic discriminations, innovations for analyte detection, solid-phase reagents, and unstable reagents/products can be more efficiently exploited. In this context, several approaches have been proposed for sample insertion, addition of reagents and increase in sample residence time, as emphasized in this lecture.

Special attention is given to the sample-to-reagent volumetric fraction ratio [2], composition of the reagent carrier stream, influence of a chemically inert reagent carrier stream and available time for reaction development in flow-based spectrophotometric assays. In fact, a proper system design is fundamental for obtaining reliable results in compliance with the Green (and White) Analytical Chemistry.

These less conventional aspects for chemical derivatization in flow systems are recommended for specific situations where: (i) the sample-to-reagent volumetric fraction ratio cannot be increased at will; (ii) the nature and/or concentration of the buffer system can be modified to reduce the Schlieren spurious signals; (iii) the concentration of the buffer solution can be lowered by selecting a system with a proper pKa; (iv) the concentrations of organic solvents can be reduced.

The lecture ends up by emphasizing that, for implementing a traditional analytical procedure in a flow system, the chemical derivatization may require modifications in reagent nature and concentration, sample/reagent volumetric fractions and/or reagent carrier stream.

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Marcela Segundo holds a BSc in Microbiology and a PhD in Biotechnology. She is currently the Head of the Analytical Development Group at LAQV/REQUIMTE, Portuguese Government Associate Laboratory for Green Chemistry, Clean technologies and also member of the Executive Board of Faculty of Pharmacy, University of Porto, where she teaches in the Department of Chemical Sciences. Her current research is focused on automation of sample treatment and on development of high-throughput microchemical methods. Her scientific production (ORCID 0000-0003-2938-0214) comprises 14 book chapters, >140 peer-reviewed papers in international journals (h-index 32). In 2016 she received the FIA Award for Science attributed by the Japanese Association for Flow Injection Analysis. Since 2018, she is the Secretary of the Division of Analytical Chemistry of the European Chemical Society (EuChemS).

Bead injection and sample preparation: a fruitful marriage?

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The bead injection technique was proposed for automation of solid-phase chemistry with the possibility of renewal through microfluidic handling of beads. The beads can be carriers of different (bio)chemical moieties, providing a platform for implementation of different chemistries, focusing on analyte preconcentration, in-situ quantification and separative methods [1].

The present work aims to address the state-of-the-art regarding bead injection towards automatic sample treatment. Several features will be highlighted, including the reproducible manipulation of sorbents with different shapes and large size distribution through universal “handling” protocols. Applications concerning food, environmental and biological samples (saliva, plasma) will be discussed, focusing the problems posed by different matrices. Moreover, online hyphenation to separative techniques will be discussed, namely the issues posed by the compatibility of eluent with conventional HPLC procedures. The advantages of automatic sorbent renewal in solid-phase extraction procedures will be demonstrated, including the reduced eluent volume required and the improved sample throughput when compared to conventional batch procedures, featuring the accomplishment of Green Analytical Chemistry.

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Acknowledgments

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Wolfgang Frenzel has studied chemistry in Freiburg und Berlin (both in Germany). His Diploma and Ph.D. Thesis were done in the field of Analytical Chemistry with emphasis on electrochemistry and flow-through analysis. After a post-doctoral period at the Hahn-Meitner Institute in Berlin (where he dealt with trace element speciation in food and biomedical research) he became leader of the analytical research group at the Department of Environmental Engineering at the Technical University Berlin. Since 1986 he is permanently employed at TU Berlin as a research scientist. His activities at the TU Berlin encompass also teaching in Analytical Chemistry, Air Pollution Control and Atmospheric Research. Over the last 35 years he has supervised altogether about 90 PhD, Diploma, Master and Bachelor Theses. He is collaborating with scientists in several countries worldwide and since 2015 a deputy teacher at the German University in Bangladesh.

Scientifically his interests are devoted to the development and optimization of analytical methods for trace determination of environmentally relevant parameters in water, soil, and air. A particular focus of his work relates to the integration of sample preparation into analytical chemical methods.

Wolfgang Frenzel has published about 100 papers in peer reviewed journals and has written several book chapters. He is, among other positions, the managing editor of the analytical journal *Microchimica Acta* and editorial board member of the *Journal of Flow Injection Analysis* and *Archives of Environmental Protection*. For many years has the chairman of a biannual international conference on ion analysis (ICIA) and in 2014 he has initiated a biannual symposium on ultrafine particulate matter.

The role of flow injection analysis and related techniques in atmospheric research and air pollution surveillance

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Air quality is of utmost importance for human health. According to the World Health Organization (WHO) more than 90% of the global population live in areas where recommended limit values for one or more air constituent are exceeded and about 7 million premature deaths per annum are associated with insufficient quality of ambient and indoor air.

From an analytical point of view the determination of several trace gases and parameters referring to particulate matter is required. Among the huge variety of analytical methods applied to air pollution measurements flow injection analysis (FIA) and related techniques (e.g., SIA, MSFIA, LOV) have received considerable attention. This is connected (as is for many other applications of these flow systems) with the attractive features of flow methodology such as automation capabilities, high sample throughput, low consumption of sample and reagents, combination with a variety of detection principles, implementation of separation and preconcentration techniques, and the relatively easy adaptation of the overall flow set-up to a particular analytical task.

The lecture will initially present a brief overview of sampling techniques for gases and particles with particular emphasis on the respective features and limitation relevant to subsequent determination of different analytes. Procedures for trace gases generally rely on collection/trapping of the analyte gas in a liquid or on a solid sorbent. Prominent examples are the determination of SO₂, NO_x, O₃ and NH₃, but also of several organic gases and vapors. Flow methods for determination of selected compounds will be critically compared to alternative means of analyte determination in the liquid phase (i.e., chromatographic techniques, automated batch procedures, electrochemical and optical sensors). Another item addressed in the lecture are automated flow-through procedures combining sampling and analysis of gases (such as membrane- and non-membrane-based wet effluent denuders) offering unattended continuous monitoring capabilities.

In connection with the determination of the chemical composition of particulate matter, FIA and related techniques have also been applied. Examples of procedures will be given for the determination of water-soluble analytes (mainly salts), trace metals after digestion, and organic compounds after solvent extraction. A focus will be set on the use of FIA and related techniques for in-line sample preparation of solid particulate matter with subsequent atomic spectrometry and chromatographic separation methods.



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António O. S. S. Rangel (<https://orcid.org/0000-0002-6486-8947>) is Full Professor at the Universidade Católica Portuguesa, Escola Superior de Biotecnologia (ESB-UCP), Porto, Portugal. He has a degree in Chemistry and got his PhD degree in Biotechnology (Chemistry specialisation), in 1991. Presently, he is President of the Scientific Board of ESB-UCP, member of the Direction Board of the CBQF Research Center, coordinator of the PhD programme in Biotechnology, and member of the Editorial Advisory Board of Talanta. His research interests are mainly focused on the development of flow-based methods for the automation and miniaturization of (bio)chemical assays, with applications in the environmental, food, and biomedical sectors. He supervised 20 PhD thesis and is co-author of more than 190 peer-reviewed papers. His teaching activity involves general chemistry and analytical chemistry courses.

Targeting the determination of iron using various flow-based approaches

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Iron species are vital for most living organisms, participating in a plethora of biological processes. However, if present in excess, they can also display disruptive effects. Therefore, monitoring different forms of iron in water, soil, and biological fluids has become of particular interest in environmental and food analytical chemistry and also in toxicology, aiming for new methods to improve the limit of detection, use of low toxicity reagents, and reduce reagents consumption and effluents production. As flow analysis techniques can be quite useful for this purpose, our group has been involved in some studies to propose different approaches to target this determination in different samples. Some of these methods were focused on a new chromogenic reagent, others on tackling challenges posed by a particular sample, while other aimed for iron speciation.

In this scenario, a discussion of some of the flow-based methods will be discussed in this lecture, involving the determination of iron (III) or iron (II), iron complexes, and also the challenges associated with the particular type of sample, either water, soil, or biological matrices.

Acknowledgments

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Paweł Kościelniak

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Paweł Kościelniak was born in Krakow, Poland, in 1952. Since 1989 he worked at the Faculty of Chemistry, Jagiellonian University in Krakow as an assistant professor and in 2000 he was awarded the title of Professor. In the years 1996-2013 he also held the position of professor at the Institute of Forensic Sciences of Prof. Dr. Jan Sehn in Krakow.

In 1993, after a visiting professor stays at the Department of Applied Chemistry, Perkin Elmer Co. in Ueberlingen (Germany) and at the Flow Injection Analysis Research Centre in Shenyang (China), he became the head of the Flow Injection Techniques unit at the Faculty of Chemistry, JU. From 1997 to 2021 he held the position of head of the Department of Analytical Chemistry, and in the years 2002-2008 he was Vice-Dean of the Faculty of Chemistry, JU. In 2000 he founded Laboratory of Forensic Chemistry - first laboratory of this profile in Polish academic centers. Currently, he is the head of the Forensic and Clinical Analysis team.

Since 2007 he has been a member of the Committee on Analytical Chemistry of the Polish Academy of Sciences and he heads of the Forensic and Toxicological Analytical Unit of the Committee.

Prof. Kościelniak is an author and editor of 10 books, and of more than 350 scientific articles and book chapters. He supervised 28 Ph.D. theses. The main areas of his research interest are flow analysis, forensic chemistry and environmental analysis with special attention paid to such issues as analytical calibration, examination and elimination of interference effects, optimization and validation of analytical methods. He was the recipient of numerous academic awards and honors. Particularly, in 2013 he was honored with the FIA Award for Science by the Japanese Association for Flow Injection Analysis.

White calibration methods in flow analysis

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Recently, a new concept called "white analytical chemistry" (WAC) has emerged in the literature [1]. In addition to the green aspects (e.g., toxicity of reagents, amount of reagents and waste), WAC considers other key criteria that affect the quality of an analytical method, e.g., precision, accuracy (red), and e.g., cost and time efficiency (blue). Analogous to the RGB model principle that superimposed beams of green, red, and blue light produce the appearance of whiteness, a white analytical method is expected to exhibit consistency and synergy of all three attributes: environmental, analytical, and practical. The whiteness of an analytical method depends mainly on two steps: the sample processing procedure and the reciprocal treatment of the sample and standards, i.e., on the calibration method chosen.

The white calibration method procedure should consider: (a) the use of a single standard without the presence of additional reagents (green aspects), (b) the processes of adding external and internal standards to the sample and diluting the sample (red aspects), and (c) the possibility of realization in a miniaturized instrumental system operating automatically, efficiently, and rapidly (green, red and blue aspects). The latter conditions are provided by flow injection analysis (FIA) in particular using the gradient technique.

In the presentation, three already published calibration methods based on the FIA-gradient technique with features approaching those of the white calibration method will be presented. Based on experimental results, they will be compared with each other with special emphasis on analytical features. It will also be suggested such a combination of these methods that can further satisfy the WAC requirements to a greater extent. The chance of introducing such kind of calibration methods into laboratory practice will be subject to discussion.

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ABSTRACTS

of keynote lectures



Jean-Luc Boudenne

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Jean-Luc Boudenne holds a 3rd cycle doctorate in environmental analytical chemistry and an expert degree in Health and Environmental Chemistry of University of Provence, and a Master of Physical Chemistry of the University of Lille. Since 2012, he is Full Professor in environmental analytical chemistry at Aix-Marseille University, manager of the team “Transfer, Reactivity and Analysis of Micropollutants in the Environment” (TRAME) in the Laboratory of Environmental Chemistry (Marseille, France), and, since 2003, a member of the Expert Committee “Waters” at Anses (French National Agency for Environmental and Occupational Health Safety). He has been president and member of several working groups within European bodies (Anses, European Centre for Environment and Health-WHO, Gesamp-UNO), in the field of the analysis, occurrence and toxicity of disinfection by-products in various environments (seawater, drinking waters and swimming pool waters).

His research fields deal with the development of “conventional” and “alternative” analytical methods for on-site and/or real-time determination of water quality based on flow analysis systems (MSFIA, SIA, MPFS, ...), development of sample pre-treatment procedures (preconcentration of targeted analytes onto modified supports; photo-oxidation of non-absorbing compounds; derivative methods for metallic ion or functional group determination), and development of chromatographic methods combined with mass-spectrometric detections. His research also implies the study of chemical and physical disinfectants’ reactivity with natural and/or anthropogenic organic matter, and on their impacts on the formation of disinfection by-products.

He has published over 80 papers and chapter books in analytical chemistry and environmental sciences and is co-author of 7 international published patents.

3D-printing contributions to design of extraction modules

Jean-Luc Boudenne, Robert-Peillard Fabien, Coulomb Bruno

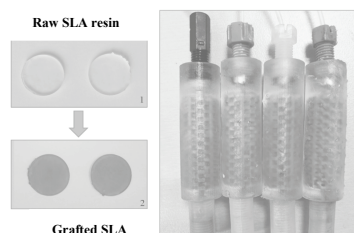
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In recent years, 3D printing has facilitated the creation of customized three-dimensional fluidic modules, such as detection cells and mixers, and several flow systems based on such 3D-printed modules have been reported previously for the determination of metals [1-3] or organic compounds [4]. However, many analytical methods require a preliminary extraction and/or pre-concentration step, traditionally performed by solid phase extraction (SPE) or stir-bar sorptive extraction (SBSE) procedures.

For these SPE or SBSE procedures, the use of 3D printing allows the fabrication of miniature devices with specific characteristics that can be adapted to different scenarios: the aim is to increase the efficiency of preconcentration while minimizing non-selective extraction on the printed material. To this end, different studies have been focused on the modification of 3D printed surfaces to incorporate smart materials or specific ligands to improve selectivity [5-7].

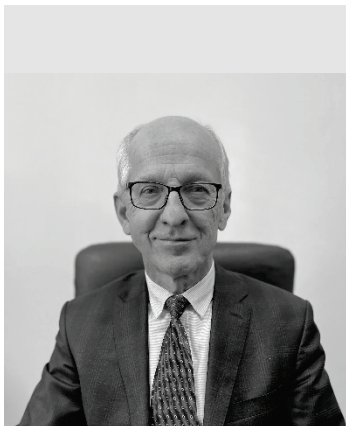
This talk will present some of the strategies that can be implemented in flow injection systems to manufacture sorbents for analyte extraction by 3D printing: porous materials, materials with a functionalized layer, chemically modified materials.

Pictures of 3D printed objects with SLA resin functionalized by dithizone carboxylate for mercury extraction during grafting steps (1: raw resin disks; 2: grafted resin disks; 3: grafted cuboids columns) [7].



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Prof. Zbigniew Brzózka is Head of the Chair of Medical Biotechnology, Warsaw University of Technology. For more than 40 years of scientific work, he created a research team in the field of chemical sensors and miniaturized analytical systems, successively membrane ion-selective electrodes (ISE), chemically modified field transistors (ISFETs, CHEMFETs), fiber optic optrod, enzymatic biosensors and more recently miniaturized analytical devices for comprehensive chemical analysis so-called "microTAS" and "Lab-on-Chip" systems. His current research is being laid on the development of microsystems for cell and tissue engineering (Cell-on-a-Chip) in *in vivo*-mimicking microenvironment where studies of cellular growth and responses to external factors are conducted for drug screening and toxicology applications. Developed Cell-on-a-chip microsystems will allow you to perform tests on cells taken from the patient and can be used in personalized medicine based on theranostic approach, where it is important to give the appropriate drug, at the optimal dose, to the right person at the right time.

He co-authored more than 190 scientific publications in international scientific journals (including 161 JCR publications, the total number of independent citations exceeds 3 300 and the Hirsch index is 35). He co-authored and a chief editor of several monographs published by Oficyna Wydawnicza PW: "Miniaturization in analytics" (2005) and "Microbioanalytics" (2009). In 2018, the latest monograph titled "Cardiac Cell Culture Technologies - Microfluidic and on-Chip Systems" was released by the prestigious Springer publishing house. He co-authored 25 patents and 3 patent applications.

Prof. Zbigniew Brzozka received a number of awards, i.e. Professor's Fellowship of the Foundation for Polish Science in science in the field of science in the period 2003-2006 and Prof. Kemula medal (Honour Award of Polish Society of Chemistry). He served as Dean of the Faculty of Chemistry, Warsaw University of Technology in 2008-2012 and 2012-2016.

Development of a functional three-dimensional model of cells in a flow-through microsystem to study a dynamic process in vitro

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Research work aimed at the development of modern oncology is constantly underway. Their inseparable element is the development and testing of new drugs and the study of the effectiveness of various combinations of anticancer therapies [1]. As a standard, in vitro cell cultures are used in the study of newly developed compounds with a potential anti-cancer character. Currently, one of the main goals of cellular engineering is to develop advanced, three-dimensional (3D) cellular and tissue models that mimic the physiology of tissues in vivo [2]. Thanks to the use of microsystems, it is possible to imitate the spatial growth of cells, the complex composition of the extracellular matrix or to control intercellular interactions in laboratory conditions. Miniaturized Lab-on-a-chip systems are one of the modern microfluidic technologies that enable the creation of advanced 2D and 3D cell cultures in laboratory conditions [3]. Flow-through cell culture has a significant advantage over conventional macroscopic cell culture techniques and can contribute to improving the state of knowledge in many areas of medicine, biology and chemistry.

Within this talk, two research projects will illustrate above approach:

- development of a flow model of a multilayer cell, made of normal and cancer cells of the ovary and breast (Cancer-on-a-chip cellular model), to investigate the effectiveness of selected anticancer therapies [4]
- development of a functional three-dimensional model of the pancreatic islet in a flow-through microsystem and its use to study the dynamic process of two-stage insulin secretion after glucose stimulation under flow-through conditions [5].

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Petr Kubáň obtained his Ph.D. in 2000 from Stockholm University, Sweden. Between 2001 and 2004 he was a postdoctoral fellow with Prof. Purnendu K. Dasgupta at Texas Tech University, USA, followed by a six-year academic career at Mendel University in Brno, Czech Republic. He then spent 2 years at Tallinn University of Technology, Estonia with Prof. Mihkel Kaljurand and currently works in the Bioanalytical Instrumentation group of the Institute of Analytical Chemistry of the Czech Academy of Sciences in Brno. His main research interests are microcolumn separation techniques, sample pretreatment and preconcentration, miniaturization, microfluidics, and the development of novel analytical instrumentation. He has been involved in clinical research during the past decade, dealing with novel approaches in diagnostics of various diseases in collaboration with the University Hospital in Brno. He is the coauthor of more than 90 scientific publications, with more than 2300 citations and a h-index of 29.

Analysis of non-invasive biological samples by microcolumn separation techniques for medical diagnostics

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Non-invasive biological fluids are becoming attractive in medical diagnostics, not only due to the latest Covid-19 pandemic and throat swab and saliva samples used in viral detection, but also in the context of other diseases. We have devoted the last decade to the development of various approaches to acquire such samples, including exhaled breath condensate (EBC), saliva, and sweat and developed many novel diagnostic approaches.

In this presentation, some of our recent results will be shown. We shall discuss the instrumentation that was developed in our laboratory and approaches for sampling EBC, saliva, and sweat, and their different applications, for instance, application of EBC in diagnosing pulmonary diseases.

We will also discuss the analytical potential of saliva, as another non-invasive sample, which can be used in diagnosis of gastroesophageal reflux disease. The analysis of various small molecules and bile acids in saliva will be presented and its clinical importance will be discussed.

Eventually we will also show how sweat analysis can be done in a simple manner for diagnosing cystic fibrosis and present some recent novel approaches called skin-wipe and skin-wash, that utilize simple cotton swabs or 3D printed devices.

Finally we will outline some sample preconcentration and pretreatment approaches to analyze trace amounts of different compounds in non-invasive samples that will conclude the variety of different approaches applied with microcolumn separation techniques for clinical diagnostics.

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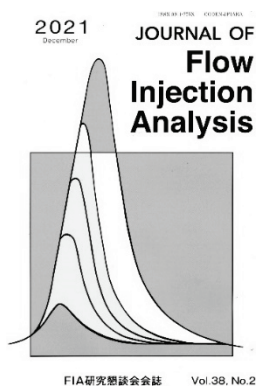


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Norio Teshima is a professor of Department of Applied Chemistry in Aichi Institute of Technology (AIT) that has ~6,000 students. In 1996, he received his Ph.D. in analytical chemistry under Prof. Takuji Kawashima of University of Tsukuba. His work focuses specifically on kinetic catalytic methods of analysis, environmental and biogas analysis, and sample pretreatment by solid-phase extraction. He looks forward to going a concert of his fave group, *Sakurazaka46* (see his Facebook and/or Twitter).

In 2018, he has been appointed to the chairman of the Japanese Association for Flow Injection Analysis (JAFIA). The JAFIA was founded by Professor Nobuhiko Ishibashi in Jan. 1984, since then it has been built by the 2nd Prof. Yosuke Ohkura, the 3rd Prof. Shoji Motomizu, the 4th Prof. Tadao Sakai and the 5th Prof. Toshihiko Imato, together with all members. Let him inform you all of the Journal of Flow Injection Analysis (JFIA) The successive JFIA editor-in-chiefs have been continuously maintaining and developing the JAFIA activities, namely Prof. Nobuhiko Ishibashi, Prof. Hiroko Wada, Prof. Takuji Kawashima, Prof. Tadao Sakai, Prof. Toshihiko Imato, Prof. Tsutomu Nagaoka and the current editor-in-chiefs, Prof. Hideji Tanaka. He would like to pay his respects to them. Also he asks you all to contribute to the *JFIA*.



Solid-phase extractants with hydrophilic groups useful for flow analysis

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Solid-phase extraction (SPE) is extensively used for the extraction and purification of a wide range of compounds. A variety of separation modes are used in SPE: reversed-phase (RP), ion-exchange, chelating, and so on. Among these modes, the RP-type SPE sorbent is widely used in pretreatment. But severe problems such as a low recovery rate and low degree of purification may occur on the SPE of relatively higher water-soluble compounds in aqueous solutions.

To solve these problems, we have developed RP-type sorbents [1,2] and hydrophilic interaction chromatography (HILIC)-type sorbents [3,4] for water-soluble compounds in which nucleobases and nucleosides were used as model water-soluble compounds. The proposed RP-type and HILIC-type sorbents have hydrophilic groups with different compositions and pore properties.

These functional sorbents can be introduced into flow analysis for water quality control [5] and urianalysis [6]. However, it is difficult to handle such fine particulate sorbents, and so we proposed binding methods of particulate sorbents to make several adsorption media; we call them molding-type solid-phase extraction media (M-SPEM) [7,8].

Also, we investigated the SPE characteristics of hydrous titania and alumina for inorganic anions and organic acids [9].

In this lecture, we introduce the above-mentioned recent studies.

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Professor Dr. Orawon Chailapakul is a professor of Department of Chemistry, Faculty of Science, Chulalongkorn University, Thailand. Dr. Chailapakul has been researching and focusing on the development of electrochemical sensors for the detection of various bio/chemical compounds. Dr. Chailapakul is the first researcher in Thailand who has utilized boron-doped diamond (BDD) as a sensor for organic and inorganic compounds and has also developed a novel device of lab-on-paper coupled with electrochemical detection and/or colorimetric detection for applications in point-of-care diagnosis (e.g. Human Papilloma Virus; HPV) and environmental monitoring (e.g. heavy metal).

The capillary-driven microfluidic device for point-of-care testing: Applications for biomarkers analysis

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Immunoassay has been popularly used in diagnostic applications due to its high specificity for each analysis. However, a conventional system is not so sensitive, various detections coupled with immunoassay have been developed for increasing the sensitivity. Electrochemical detection integrated with fast-flow/delayed paper-based platform can provide better sensitivity, and can reduce some drawbacks including time consuming, and technical complexity. In this work, employing the new geometry design platforms of the capillary-driven microfluidic device for biomarkers analysis will be presented. These proposed platforms are easy-to-use, able to reduce assay timing, and reagent consumptions which are applicable for on-site analysis without external instrument needed. The detection of hepatitis B, hepatitis C viruses, and lipoarabinomannan will be demonstrated by using this smart platform.

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Dr. Duangjai Nacapricha is an Associate Professor at the Department of Chemistry, Faculty of Science, Mahidol University, Bangkok Thailand. Dr. Duangjai Nacapricha has been one of the Thai research scientists in the community of flow-based analysis for many years. Her research interest has been development of flow-injection based systems for online gas separation and in-situ detection. Recently, she has expanded her research interest to cover microfluidic paper-based analytical devices and disposable analytical devices for field use. Integration of electrochemical sensors to the disposable devices are now one of the ongoing research.

Exploiting vertical flow in paper material for the fabrication of a unique microfluidic lid for reproducible addition of a liquid reagent

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The work exploited vertical flow through the paper thickness in a defined area of the two printed paper layers for reproducible addition of an acidifying reagent solution into a disposable reaction well. A unique three-layered paper-based microfluidic lid (μ Fluidic lid) was fabricated. The lid comprised of two paper layers that were printed with hydrophobic ink to form a circular zone. These paper layers were mounted using a foam-tape with a circular hole punched out that formed an airgap connecting between the two circular zones. Volume of the airgap was half of the size of the dispensing volume of the acid reagent (*viz.* 200 μ L). With the permeation of the acid reagent through the thin paper layers of the lid, the acid coalesced beneath the paper membrane and finally dripped into the sample solution under its own weight in a reproducible manner. The airgap inside the μ Fluidic lid acted as a reservoir that slowed down the coalescing of the acid reagent on the underside of the lower paper layer. Addition of the acid reagent into the reaction well converted sulfite in the sample solution into SO_2 gas that diffused through the PTFE membrane at the bottom of the microwell to the graphene-based gas sensor. This work also presents the concept of the *in-situ* gas generation with an *in-situ* voltammetric gas sensor for on-site and selective measurement of sulfite in wine. The method can be applied to all types of wine including wines containing CO_2 . With the limit of detection (3SD of intercept/slope) as low as $1.5 \text{ mg L}^{-1} \text{ SO}_2$, our method is suitable for analysis of high-quality wines which normally contain sulfite below the regulated limit of $10 \text{ mg L}^{-1} \text{ SO}_2$.

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Peter C. Hauser carried out his undergraduate studies in Switzerland and then obtained an MSc at the University of British Columbia (UBC) in Vancouver, Canada, under Prof. M. W. Blades working on ICP-OES (1985). This was followed by a PhD at La Trobe University (Melbourne, Australia) under Prof. R. W. Cattrall on Flow-Injection Analysis (1988). Following a postdoc at the Paul Scherer Institute (Switzerland) working on fuel cells and a lectureship at Auckland University (New Zealand) he took up his position at the University of Basel (Switzerland) in 1996. Research interests include miniaturization in analytical chemistry, flow-injection analysis, micro-separation techniques, electrochemical detection techniques for electrophoresis, optoelectronic detectors, automation in analytical chemistry, electrochemical gas sensors, miniaturized plasma spectrometers and detectors, clinical analysis, field and process analysis. This has always included different aspects of the use of electronics in analytical instrumentation.

Purpose made capillary electrophoresis instrumentation

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In contrast to chromatography the separation method of capillary electrophoresis (CE) does not require ultra-high-pressure manifolds. It is therefore possible to construct CE instruments relatively easily in the laboratory. This allows cost savings, but there are also applications of CE for which commercial solutions are not readily available. This includes field-portable and battery-powered instruments, automated on-site monitoring systems (process analysis), or laboratory instruments dedicated to special tasks.

While sample injection into the capillary may be done by manual syphoning this is not always easy to carry out, especially in the field, and it is preferable to automate this step as well as flushing operations by employing a flow-injection approach. The CE part may then be considered as a powerful multi-analyte detector in a flow-injection system. Pumping can be carried out by pneumatic pressurization and miniature valves allow the sequencing of operations with the help of a microcontroller. By making use of miniature components compact instruments can be assembled in a highly flexible and adaptable microfluidic breadboard approach. The most demanding parts of these instruments is the detector, but capacitively-coupled contactless-conductivity detectors (C⁴D) may be constructed in the lab if electronic expertise is available or be bought at relatively low cost. Also possible is the in-house building of absorbance or fluorescence detectors based on light-emitting diodes (LED) or laser diodes.

The presentation will highlight recent developments in our laboratory, in particular an instrument developed for coupling to a mass-spectrometer as detector.

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Kate Grudpan is an Emeritus Professor. He has experienced in analytical chemistry for over 40 years. He was named as a pioneer in some aspects including flow-based analysis and cost-effective analysis. He engaged in Ph.D. program in analytical chemistry to be launched for the first time in Thailand. He engaged in various national and international activities with analytical chemistry in Thailand to be internationally visualized and formed networks for multi-generations and multi-disciplinary aims. Apart from national organizations for which he served as committee members such as the National Research Council of Thailand and University Councils, he also served as a UN expert in a neighboring country, and as guest editors and member of editorial boards of various journals (publishers: Elsevier, Wiley, MDPI, etc.). He experienced in supervising 50 Ph.D. students, a number of M.S., B.S. students, and Post-docs. He has authored over 220 publications in international journals, and 5 book chapters by international publishers. He has received various recognitions such as the Outstanding Researcher of the year 1998, Outstanding Scientist of the Year 2001, Thailand Professor of the year 2011, JAFIA Scientific Award, Thailand Research Fund Distinguished Research Professor Award 2017, the Humboldtian fellow, and listed in the world's top 2% scientists on the University of Stanford list. Currently, his research interests cover flow-based analysis, cost-effective analysis, green analytical chemistry, and local wisdom based green innovation: local issue-global impact-sustainable world.

The initiative in using a natural reagent for chemical analysis has been supported not only by Thai colleagues but also by overseas scientists, including Prof. Gary Christian, Prof. Ian McKelvie, Prof. Spas Kolev, and Prof. Woi Pei Meng who are co-authors of the presentation.

Natural reagents for chemical analysis: consideration with United Nations Sustainable Development Goals

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Applying a more than 100 years old traditional knowledge of local villagers in a northern town of Thailand, simple guava leaf water extract was first reported as a natural reagent for iron determination using a flow injection system [1]. There have been further natural reagents for various analytes employing flow and non-flow-based analysis systems [2,3]. This presentation will briefly discuss such developments. Pros and Cons of using natural reagents will critically be discussed in connection to the approaches of green analytical chemistry (GAC), white analytical chemistry (WAC), and United Nations Sustainable Development Goals (UN-SDGs) [4,5].

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ABSTRACTS

of invited lectures

A novel flow injection electrochemical sensor based on CNT-Ni(OH)₂-CB/Nafion nanocomposites for evaluating xanthine oxidase inhibitory activity

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Xanthine oxidase (XOD) catalyzes the oxidation of xanthine (XA) to generate uric acid (UA) and reactive oxygen species (ROSs) that are associated with many diseases. Exploration of effective XOD inhibitors as potential medicines is essentially important. In this work, the newly designed electrochemical sensor has been fabricated to examine XOD inhibitory activity. The hybrid nanocomposites composed of carbon nanotubes/nickel oxide/carbon black/Nafion modified screen-printed graphene-carbon paste electrodes (CNT-Ni(OH)₂-CB/Nafion/SPGE) can obviously differentiate the electrochemical reactions of UA and XA. A voltammetric sensing platform can examine the XOD activity by simultaneously measuring the concentrations of UA and XA resulted from the XA-XOD reaction. Moreover, the sensing electrode was incorporated to flow injection amperometric system for screening XOD activity by high throughput determining UA. A linear range of 0.05 – 2000 μ M UA with a detection limit of 0.01 μ M (3S/N) was achieved. Besides, the developed method provides a sample throughput of 25 injections h⁻¹, excellent sensitivity (0.0189 μ A/ μ M), selectivity, repeatability (RSD 2.8%, n=7), and stability (RSD 1.2%, n=50). The proposed system was successfully applied to examine the inhibitory activity of some XOD inhibitors, including allopurinol, quercetin, gallic acid, kaempferol, ferulic acid, and caffeic acid.

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As simple as possible but no simpler...

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Until recently, the development of analytical chemistry was focused mainly on the miniaturization of components of devices for chemical analysis. At the same time, the desire to meet the requirements for the performance of analytical systems, their accuracy and precision of operation makes them excessively complex in construction and operation.

On the other hand, recent years have revealed that analytical tools to conduct screening tests are much more needed than universal, sophisticated devices to which, for example, in a lockdown situation, there is no access. Telemedicine without the possibility of remote diagnostics is an ordinary conversation and conservative treatment and not the equivalent of a stationary visit to a doctor's office.

Fortunately, many research teams develop ideas such as: constructing paper analytical systems with visual or optoelectronic detection, designing flow-through components and making them in the most economical methods of rapid prototyping, using a smartphone as a detection or control element.

In Author opinion, many of these simple systems, though they are called “*primitive*” by many, will be mass-produced and used in the future, even though they are not perfect.

Examples of such systems will be discussed during the lecture.

Smart electrochemical sensors for simultaneous determination of dual carcinogen oxidative stress biomarkers through core–shell molecularly imprinted polymers

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Molecularly imprinted polymer (MIP) based electrochemical sensors are attractive due to their high selectivity to the respective target analyte, they are cost-effective, straightforward to synthesize, and reveal excellent mechanical strength and long-term stability. This work explores use of core-shell MIP as novel dual-imprinted sensors for simultaneous determination of carcinogen oxidative stress biomarkers including nitrotyrosine (3-NT), 8-hydroxy-2'-deoxyguanosine (8-OHdG) and 4-nitroquinolin-N-oxide (4-NQO). Effective routes for applying nanoparticles's surface as reaction sites for constructing the core-shell MIP will be presented together with simple fabrication of compact electrochemical dual imprinted-sensors. Analysis based on dual MIP sensors to simultaneous quantification of 3-NT and 8-OHdG content in human urine and serum samples will also included in this presentation.

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Automation in sample manipulation for long-term monitoring

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Benefits of versatile manipulation of sample and reagents were applied in the past for the liberation of active substances using Franz diffusion cells in a sequential injection (SIA) systems. In this talk, an overview on recent development in this field will be given, kinetic profiles of different formulations were compared in the case of clotrimazole to describe profiles of drug liberation from commercial creams and a nanoformulated dispersion [1]. A similar approach was used for liberation testing of substances trapped in nanofiber membranes and controlled release profiles were obtained.

A novel flow-programming setup based on the SIA concept was proposed for on-line monitoring of cell membrane permeation [2]. The permeation kinetics of the marker rhodamine 123 was obtained in a fully automatic mode by sampling the apical and basolateral compartments of a 3D printed permeation module every 10 min. The effect of the P-glycoprotein transporter inhibitor on the efficiency of a marker transport across a cell monolayer was investigated in detail. Finally, a 3D printed module was developed and used for the automation of toxicity assays based on the release of *Metridia* luciferase upon interaction with toxic substances and thus changes in cell membrane permeability in real-time [3].

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Second dimension in sequential injection chromatography

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The second generation of *Sequential Injection Chromatography* [1] employing a 10 MPa stainless steel syringe pump and three valves utilizes chromatographic columns up to 100 mm in length or even the addition of the second dimension. This dimension can be online sample treatment and analytes preconcentration (solid-phase extraction) or second dimension chromatography to significantly increase the separation selectivity desired for the real-life sample analysis [2]. Chromatographic data, consisting of multiple peaks and utilizing spectral detection, are more complex than those from single analyte flow analysis. Thus, data handling, treatment, and evaluation are challenging to gain full information from the 2D analysis.

The 2D method sequence compilation and following optimization are also demanding but contribute to a better understanding of *Flow Programming* [3], separation processes, and hyphenation of both method stages.

The development of 2D methods for analyzing active substances contents in food supplements of natural origin will demonstrate the key features of *Flow Programming*, SIC hardware, and software.

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Lab-in-syringe automated sample preparation for HPLC

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An overview is given on recent research from our group on how to implement the most recent offspring of the flow technique family, Lab-In-Syringe (LIS) [1], for the preparation of samples to be analyzed via high performance liquid chromatography. LIS takes advantage of a modified Sequential Injection Analyzer to carry-out flow-batch operations inside the syringe void. Aided by a magnetic stirring bar and microprocessor control, the solution content can be stirred on demand with varying speed and time as required.

LIS shows high operational versatility including straightforward pressure change (moving the piston with the syringe head valve in a closed position), gas confinement (given the steadily sealed nature of a syringe), and possibility to the glass syringe as extraction, mixing, reaction, and even detection chamber.

LIS has, in particular, proven to be advantageous for the automation of liquid phase microextraction methodologies. Here, we overview recently developed LIS-automated sample preparation methodologies based on solid phase extraction, directly immersed single drop microextraction [2], and homogenous liquid-liquid extraction [3] for the enrichment of organic contaminants. In all cases, the extracted contaminants were subsequently analyzed by online coupled liquid chromatography with achieved matrix clean-up and preconcentration-related gain in sensitivity.

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Tackling sample complexity in microfluidic paper-based devices

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Nowadays, the interest in on-hand easy-to-use devices had an exponential growth and, if used correctly, it can be an effective aid in both food safety and healthcare. Fast, on hand analytical tools represent a vital strategy to identify undiagnosed health issues, even with asymptomatic, pre-symptomatic or unrecognized symptomatic diseases and to anticipate food degradation in shelflife. However, if a faster analysis results in a faster decision on the course of action, then it is crucial to have accurate and reliable tool.

To attain an immediate, on-hand response, the use of self-readable, easy-to-use devices is a natural choice namely the concept of microfluidic paper-based analytical devices (μ PADs). The portability and low consumption of both reagents and sample associated to this technique, make these devices ideally suited for unskilled operators and frequent analysis. However, following this strategy, as a faster alternative to conventional lab-based testing, rises sample handling challenges namely the reduced sample volume and matrix complexity. The downscaling of the analytical procedures results in a size limitation for in-line pretreatments as well as speciation and interferences minimization processes. A critical discussion of this issue will be addressed and a few examples will be shown.

Acknowledgments

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Nitrogen-Doped Carbon Dots (N-CDs) as peroxidase mimic coupled with a laminated three-dimensional microfluidic paper-based analytical device (laminated 3D- μ PAD) for smart sensing of total cholesterol from whole blood

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Nanomaterials mimicking peroxidase activity open a new avenue for the development of a colorimetric analysis technique. A great number of nanomaterial mimics have been developed to replace the use of natural enzymes for the determination of total cholesterol (TC). My talk will present a nontoxic and simple method for the synthesis of nitrogen-doped carbon dots (N-CDs) by a hydrothermal method via a 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)-diammonium salt (ABTS)-H₂O₂ chromogenic system for colorimetric TC analysis on laminated three-dimensional (3D)- μ PAD. The time-dependent density functional theory (TD-DFT) was utilized to better understand the effect of nitrogen doping on CDs and elucidate how the N-CDs reacted with H₂O₂. The laminated 3D- μ PAD was designed as a prototype device for semiquantitative detection of TC in the whole blood sample with zero-step of sample preparation and could be determined by the naked eye within 10 minutes by simple comparison using a color chart. Overall, the proposed colorimetric device serves as a low-cost, rapid, simple, sensitive, and selective alternative for TC detection in whole blood samples that is friendly to unskilled end users. And our developed device has the potential to be further developed into a self-monitoring device that may be used for point-of-care diagnostics by unskilled end users that can be measured other relevant biomarkers.

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Different approaches to nonenzymatic lactate determination in body fluids - the development of flow analysis system and paper-based analytical device

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The monitoring of lactate concentration is of key importance, among others in medicine. Lactate, which is the final product of anaerobic glycolysis occurring in all tissues of the human body, is a diagnostic marker of many different diseases such as hyperlactemia, hypoxia or sepsis [1]. Also, the production of lactate by cancerous cells takes place during the increased glycolytic activity under aerobic conditions [2]. There are many lactate determination methods based on enzymatic assays, however they are relatively expensive and complicated with laborious and time-consuming steps [3].

In this contribution, two analytical platforms for nonenzymatic lactate determination are presented. The analytical method used in the course of the research is based on the photochemical reaction between ferric ion and lactate leading to the formation of purple complex of ferrous ion with ferrozine [4]. The first approach concerns the development of mechanized multicommutated flow analysis system (MCFA) with flow-through optoelectronic cell integrating reaction chamber and detector. The second design is devoted to the paper-based analytical device (PAD) which is the downscaling effect of the flow analysis system. In both cases, the attention will be paid to design, optimization process and application of proposed systems. In the final stage, the analysis of selected body fluids will be used to assess the usefulness of the developed MCFA and PAD systems.

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ABSTRACT

of Gold Sponsor presentation



Miniaturization and integration of Shimadzu analytical tools. From balances to HRAM-DIA/DDA-MS/MS

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The issue of miniaturization in contemporary analytics is still relevant, as are the attempts to integrate a greater range of tools helpful in one complex analytical process.

The presentation will discuss the advantages of combining these various tools such as balances with an UHPLC both from one manufacturer, Quality by Design software integrated with control software and classic ionization techniques such as HESI/APCI combined with ambient MS and iMS techniques.



Figure 1. The modern Quality by Design software and the UHPLC workstation integrated with analytical balances from the same manufacturer allow for the integration of the analytical process and increase efficiency.

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ABSTRACTS



of oral presentations

3D-printed flow manifold integrating solid contact ion-selective electrodes dedicated to multicomponent urine analysis

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Urine is an example of a biological fluid, which is a great source of information about the condition of human organism. It contains many inorganic and organic cations as well as anions which play a significant role in a proper human organism functioning. The detection of any deviations from the normal concentration ranges of these components of urine might be a useful diagnostic tool for medical examiners indicating possible lines of treatment. In this respect, 3D printed devices integrating sensors for measuring multiple parameters may be one of the most viable approaches to ensure cost-effectiveness for widespread use. In this work, the 3D printed flow manifold, dedicated to simultaneous determination of K^+ , Na^+ and Ca^{2+} in the urine samples, is presented. Moreover, the lab-made gold electrodes with the octadecylamine-functionalized multi-walled carbon nanotubes (OD-MWCNTs) layer as a solid contact were used during measurements to show the possibility of improving metrological parameters, especially in comparison with coated wire electrodes. The applicability of the 3D printed flow manifold was investigated during the analysis of synthetic samples and two certified reference materials [1].

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Acknowledgments

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Advances in flow injection are based on flow programming

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In continuous flow format, sample zone travels downstream from injection point at constant flowrate through manifold and flow cell. Flow programming [1], based on combination of flow reversals, flow accelerations, and stop flow periods, is designed to transport sample zone upstream from injection point, into a holding coil, mix it with precisely metered volume of reagent, and bring it by flow reversal into flow cell. Computer controlled assay protocols comprise a series of flow reversals, accelerations, and stop flow periods tailored to accommodate a variety of reagent-based assays.

Programmable Flow Injection (pFI) performs tasks that cannot be accomplished in continuous flow format:

- Automation of variety of reagent-based assays in the same manifold (lab-on-valve) without need for physical reconfiguration [2]
- Choice between monitoring at nonequilibrium (flow/gradient) or equilibrium (batch) conditions [3]
- Automated calibration by single standard solution [4]

Presentation is designed as a guide through recently published Tutorial: “Advances in Flow Injection Analysis” [5] and relevant literature.

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Microcontrollers for flow analysis - DIY automation

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Modern analytical systems typically require advanced drivers to control devices. To automate the operation of various sets of components, it is necessary to use appropriate hardware and programming tools that will orchestrate the work of devices of different manufacturers. Also, the control software should be able to write "scenarios" that will allow the system to make specific decisions, previously designed by the operator.

Currently, existing software solutions require support from hardware manufacturers and their integration in a heterogeneous environment is not always possible due to the lack of compatibility in software drivers. This limits the miniaturization and integration of such solutions.

Designing an integrated analytical system entails the need to integrate control and measurement systems. A convenient form of such integration is the use of a prototype microcontroller with open-source hardware and software. Such a device allows tight integration of components, and also requires some basic knowledge of communication protocols with peripherals.

In this communication, I will present a simple way to automate the operation of an exemplary flow analysis system for creatinine determination. Such a system consists of a programmable multi-position valve, and a syringe pump with an integrated optoelectronic detector, all three operated by a prototype Arduino microcontroller.

Acknowledgments

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Heartcutless coupling of bead injection and HPLC

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Bead injection stands as the greenest mechanization of SPE workflows due to the minute amount required of sorbent and sample, and the potential reuse of the later. More and more the original in-bead detection capabilities are relegated, and the BI is hyphenated to standalone liquid chromatographic separations for enhanced selectivity and sensitivity. This hyphenation is often carried out in heartcut mode, in which the analytes are eluted from the SPE and parked in the injection loop, from which they will be transferred to the analytic column. This approach suffers from peak broadening so unless the chemistries of the SPE and column are orthogonalized; there is also a decreased analytical signal since the analytes are not completely transferred to the column, and different analytes experience different enrichment factors in different cuts.

In the present contribution we present a new valve for hyphenating the BI and HPLC in a heartcutless manner, namely, what those skilled in the art call 'bead injection with the beads in the injection coil'. The prototype also allows the removal of the sorbent with positive pressure for a very reproducible bead unpacking. Enrichment factors obtained are higher than 300, SPE and HPLC columns' chemistries do not need to be orthogonalized and we report for the first time the manipulation of 5 µm sized sorbent particles.

A proof of concept application demonstrating the kinetic monitoring of a bioaccessibility test is also presented, including the unattended in-valve extraction, preconcentration and injection of different fractions into HPLC.

Acknowledgments

This work was supported by the Austrian Science Fund (grant number M02579). DJCS acknowledges greatly Hagen Preik-Steinhoff, Bert Manzke and Franco Cozzio from VICI AG International for the kind provision of the fluidic components: pump, valves, tubes, and connectors that were used throughout this research.

Flow system for D₃ vitamin electrochemical determination in pharmaceutical formulations

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Cholecalciferol (vitamin D₃), as an organic compound from the vitamin D group, is responsible for the proper maintenance of calcium and phosphate metabolism, it plays a role in cell proliferation, maturation and differentiation influences the regulation of immune processes. Recent studies have reported that level of vitamin D₃ in body fluids is related to the incidence and course of SARS-CoV-2 infection [1-3]. The multitude of pharmaceutical formulations (both drugs and dietary supplements) makes the pharmaceutical industry looking for simple, cheap, quick and repeatable methods of cholecalciferol determination in various, in terms of matrix, products.

The developed method of voltammetric determination of vitamin D₃ is based on the use of an unmodified glassy carbon electrode (GCE) in a supporting electrolyte solution of a simple composition (ethanol: water with the addition of LiClO₄, 9:1, v/v) and without special preparation of samples, thus complying with the above-mentioned requirements. Optimization of the measurement conditions allowed for the determination of cholecalciferol at the micromolar level in various pharmaceutical preparations (drugs and supplements), as well as in varied forms: in drops, soft and hard capsules. The use of a flow system has modernized and further simplified the entire procedure, allowed to improve the precision while maintaining other analytical parameters in comparison to stationary measurements. The content of the analyte in the tested pharmaceutical products was estimated with the use of three calibration methods: a series of standards, standard addition methods (SAM) and the Signal Increment Standard Addition Method (SISAM). On the basis of the performed measurements, the limit of detection and quantification was calculated, the values of which are respectively: $1.5 \cdot 10^{-7}$ and $5.1 \cdot 10^{-7}$ mol / L.

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Use of dimethylglyoxime as a chromogenic agent in a flow-based system for the determination of Ni(II) in recreational water

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Nickel occurs ubiquitously and naturally in the environment (soil, water, and air). Nickel is considered to be an attractive metal with several applications and is widely used in the industry. As a consequence, it can be easily released into the environment, reaching waters sources and the soil [1]. Most of the methods for its determination are based on atomic absorption or emission spectrometry. In this scenario, an alternative spectrophotometric-based method for the determination of nickel in recreational water was developed. The strategy was to devise a miniaturized and automatic system based on sequential injection analysis, intending to achieve an expeditious, real-time, and low-cost method. For the spectrophotometric determination, a reagent with associated selectivity for nickel determination was employed, the dimethylglyoxime. Additionally, considering the expected low levels of nickel in recreational waters, with a maximum concentration of $100 \mu\text{g L}^{-1}$, a liquid waveguide capillary flow cell (LWCC), with 100 cm optical pathlength, was used to improve the sensitivity and the limit of detection of the method.

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Instrumentation-free approach to rapid detection of albumin-to-creatinine ratio using paper-based analytical devices

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The paper-based analytical devices (PADs) are developed as the analytical platform for rapid detection of renal function through the determination of urinary albumin-to-creatinine ratio (UACR). The detection of UACR is based on the reaction propagation distance of albumin and creatinine in the PAD and does not require a camera/scanner, analytical software, and/or color reference chart. In this work, alkaline picrate is immobilized on the PADs and produces a yellow color along the propagation zone. With the presence of creatinine (Cr) in the sample, part of the propagation zone will change to an orange color at a certain distance according to the concentration of the analyte. The reagent for the albumin (Alb) is Bromothymol Blue (BTB), which results in a color change in the propagation zone from green to blue. Another reagent used for Alb is bromocresol purple (BCP) which produces a color change in the propagation zone from yellow to purple when reacting with albumin. The semiquantitative UACR is carried out by drawing a line/slope connecting the upper two zones of color change in propagation zones of the PADs, showing the UACR level in the reading scale. In comparison to the UACR gold standard, the proposed method resulted in good accuracy (87.5%).

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Chip-based spectrofluorimetric determination of iodine in a multi-syringe flow platform with and without in-line digestion

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Iodine is a component of the thyroid hormones essential for human growth and development [1]. In Europe, two-thirds of the countries reported inadequate iodine intakes [2,3]. In this work, a flow-based spectrofluorimetric method for iodine determination was developed. The system consisted of a multi-syringe module, used as a liquid driver, and a chip-based flow manifold for solutions handling with integrated spectrofluorimetric detection. Iodide was quantified from its catalytic effect on the redox reaction between Ce(IV) and As(III), based on the Sandell–Kolthoff reaction. An in-line oxidation process, aided by UV radiation, was implemented to eliminate interferences and release iodine from organoiodine compounds. The method developed was applied for the determination of iodine in salt, pharmaceuticals, supplement pills, and seaweed samples without off-line pre-treatment. The method allowed iodine to be determined within a range of 0.20–4.0 $\mu\text{mol/L}$, with or without the in-line UV digestion, with a limit of detection of 0.028 $\mu\text{mol/L}$ and 0.025 $\mu\text{mol/L}$, respectively [4].

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Sequential injection system for analysis of degree brix, orthophosphate and pH in raw sugarcane juice applicable to sugar industry

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This work presents, for the first time, a new sequential injection analysis (SIA) method to simultaneously analyze degree Brix, orthophosphate and pH in raw cane juice. These key parameters relate to price of harvested sugarcane and quality of cane juice for sugar production. The SIA system employed two detectors. First detector is a diode-array spectrophotometer, equipped with a regular flow cell, for measurements of degree Brix and orthophosphate. Quantitative of degree Brix ($^{\circ}\text{Bx}$; ca. % (w/w) sucrose) was based on manipulation of the schlieren effect at the interface between plugs of sample and water. Orthophosphate analysis was carried out based on the molybdenum blue method with significant reduction in consumption of the reagents. Compensation of the schlieren effect from sucrose for determination of orthophosphate was achieved by using a dual-wavelength spectrometric detection. Second detector is a pH-sensing device, called ion-selective field-effect transistors (ISFET). The ISFET is based on the current through the ISFET arising accordingly to the H^+ concentration in solution. Our developed SIA system provides linear calibration graphs fitting for purpose in analysis of sugarcane juice (pH: 0-14, $^{\circ}\text{Bx}$: 1.0-7.0 and P_2O_5 : 20-200 mg L^{-1}). Simultaneous analysis of sugarcane juice for pH, $^{\circ}\text{Bx}$ and P_2O_5 is carried out within 5 min (12 sample per h). Precision of SIA system is acceptable (RSD < 3%). Our SIA system gave quantitative results insignificantly different, as compared with conventional methods for analysis of pH, $^{\circ}\text{Bx}$ and P_2O_5 in sugarcane juice.

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An automated hydrodynamically mediated technique for preparation of calibration solutions via capillary electrophoresis system

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In recent years, there has been increasing interest in methods focusing on the automation of sample pre-treatment and analysis, as well as implementation of the rules of 'green chemistry'. Automation enables lessening reagents' consumption, hence decreasing the amount of chemical waste. Usually, it also shortens the time of the whole analysis process and limits the influence of the operator on basic validation parameters, such as accuracy, precision, repeatability, and linearity.

Now, we would like to present a novel procedure for preparing calibration solutions for capillary electrophoresis-based quantitative analysis [1]. Our approach, named the automated hydrodynamically mediated technique (AHMT), uses a capillary and a pressure system to deliver the expected amount of working solution and diluent directly to a sample vial. It should be emphasized that such automation does not entail any investment in new hardware or software, as the whole procedure can be achieved using a commercially available CE system. As a result, calibration solutions are prepared automatically inside the CE instrument, without any or with minimal manual operation. Two different modes were tested: forward and reverse, differing in the direction of hydrodynamic flow. The calibration curves obtained for a model mixture of analytes using AHMT were thoroughly compared to the standard procedure based on manual pipetting. The results were consistent, though the volume of obtained calibration solutions and the potential risk of random errors were significantly minimized by AHMT. Its effectiveness was further enhanced by the application of SCIEX® nanoVials, reducing the actual volume of calibration solutions down to 10 µL. The benefits of using such low volume may be critical in case of bioanalysis, where the application of expensive reagents in limited amounts, such as e.g. proteins is frequently appointed.

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Development of a method for determining the acid dissociation constant by combining the capillary electrophoresis with microscale thermophoresis in the on-line flow format (CE-MST)

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The speech will show the first successful application of the tandem technique, capillary electrophoresis-microscale thermophoresis (CE-MST), to determine the value of equilibrium constant, by connecting the CE and MST instruments on-line with a fused silica capillary. The model used was the equilibrium of the acid-base dissociation of fluorescein isothiocyanate, described by the value of the acidity constant (pKa). In order to identify the main shortcomings and propose an optimal strategy for further development of this methodology, a comprehensive evaluation of CE-MST and MST techniques was carried out against the 12 rules of "White Analytical Chemistry" [1]. In order to adapt the evaluation process to the specifics of the analysis of molecular interactions, the set of rules was modified, keeping their division into 4 red criteria (analytical), 4 green criteria (environmental friendliness and safety) and 4 blue criteria (practical and economic). The overall potential of both techniques was estimated in favor of the standard variant (MST). This result is not surprising as the CE-MST technique is currently still in an intermediate stage of development. Nevertheless, in the near future, it is planned to implement a strategy aimed at improving the certain criteria and increasing the overall quality of the CE-MST method.

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Development of organic acids determination in saliva using isotachophoresis technique (ITP)

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There are three sources of organic acids in saliva: i) the food that is eaten; significant increase in acid concentrations is observed immediately after eating a meal, which gradually decreases over time until it reaches the level before consumption, approx. two hours after the end of consumption ii) microorganisms inhabiting the plaque; research shows that some acids (e.g. lactic, acetic or formic acid) are formed as pyruvic acid fermentation products under the influence of microorganisms inhabiting the oral cavity environment; iii) as intrinsic saliva components[1]. The saliva microbiome can be modified not only as a result of diseases within the oral cavity, but also in the case of systemic diseases (e.g. diabetes, cancer or heumatoid arthritis)[2]. As part of this study, a method for the determination of selected organic acids in human saliva using capillary isotachophoresis was developed. An EA 202M analyzer (Villa Labeco, Spisska Nova Ves, Slovakia) equipped with two columns - preliminary separation (160 mm x 800 µm I.D.) and analytical (160 mm x 300 µm I.D.) and a conductometric detector was used for the analysis. The following solution was used as the leading one (pH = 4.5): hydrochloric acid solution (10 mM), including 0.1% methylhydroxyethylcellulose (M-HEC) and epsilon-aminocaproic acid solution (20 mM) solution. The terminating electrolyte contained 5 mM of caproic acid and 5 mM of histidine. The developed method allowed for the separation of 12 organic acids (including 4 short-chain fatty acids), of which 8 were identified in the saliva samples obtained from healthy children. The parameters determined during the validation of the method indicated that the obtained results are reliable. Thus, it may become a routine tool for the determination of organic acids in human saliva, which may turn out to be useful in the diagnosis of diseases of various etiological origins.

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Development of sequential injection analysis with cost-effective optical device based on LED colorimeter for simultaneous analysis of zinc and copper in dietary supplements

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The developed sequential injection analysis (SIA) system with cost-effective optical device for simultaneous determination of zinc (Zn^{2+}) and copper (Cu^{2+}) were proposed in this work. The detection device was in-house fabricated based on a concept of paired emitter detector diodes (PEDD) operated by two pieces of light emitting diodes (LED). Quantitative analysis of Zn^{2+} was based on the influence of Zn^{2+} on the oxidation rate of N-(1-Naphthyl)ethylenediamine dihydrochloride (NED) by hexacyanoferrate(III). Purple suspension product was occurred and monitored by the first PEDD. Cu^{2+} was determined by the reaction of 2-Carboxy-2'-hydroxy-5'-sulfoformazyl-benzene sodium salt (zincon). Purple-blue complex solution was monitored by the second PEDD. Two-channel recorder was used for monitoring of the two signals, one channel for Zn^{2+} and the other Cu^{2+} . Under the optimum condition, the linear range of Zn^{2+} and Cu^{2+} were 20 – 80 ppm and 1 – 5 ppm, with limits of detection of 15 ppm and 1 ppm, respectively. The proposed system provides satisfactorily good precision ($\text{RSD} < 5\%$). Throughput of 8 sample h^{-1} for two analytes (triplicate injections including cleaning step) was achieved. Finally, the developed system was applied to the analysis of dietary supplements and the results were compared with FAAS. Certified reference material was also used to accuracy evaluation. The proposed methods are automatic, simple, rapid and low cost. The system of SIA-PEDD could be further developed as a portable device for on-site application.

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Automatic flow system for determination of chemical oxygen demand (COD) with a two-beam photometric detector

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A system for automatic determination of chemical oxygen demand (COD) using the oxidation of organic compounds with the $K_2Cr_2O_7/H_2SO_4$ mixture has been developed. The system is designed to monitor COD in wastewater and surface water samples. The system uses a PEDD double-beam photometric detector. One vial contained the reaction mixture with the test sample and the other the reaction mixture with an addition of water. The reaction time and temperature for the different standard substances and COD ranges have been optimized. The measurement set was built in the form of a sequence analyzer using a syringe pump and a 6-channel electromagnetic valve. The analyzed samples are pre-filtered with a 100 μm metal mesh filter, and in the second stage they pass through a 0.45 μm filter. The sample filtering system has the option of automatic pre-filter cleaning with a back stream of water or air.

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Spectroelectrochemical determination of the rate constant of protonation reactions for some radical anions

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Using a conventional three-electrode system and electrochemical cell, we developed a simple spectroelectrochemical method (Fig. 1) that can be used to determine the rate constant of protonation reaction for several reduced species. Using the system, the absorbance change by the generation of the radical anion of 9,10-diphenylanthracene (DPA), biphenyl, and *p*-quaterphenyl was monitored. The molar extinction coefficient (ε) of the radical anions was calculated with the relation between the absorbance (Abs.) and charge (Q) during potential steps, as

$$\text{Abs.} = \frac{2\varepsilon Q}{SF} \times 10^3 \quad (1)$$

where S and F are the electrode area, and Faraday constant, respectively. The absorption spectrum of electrochemically generated dianion of *p*-quaterphenyl was also monitored. The absorbance change during the protonation at different concentration of ethanol (c_{EtOH}) for $\text{DPA}^{\cdot-}$, $\text{biphenyl}^{\cdot-}$, $\text{p-quaterphenyl}^{\cdot-}$, and $\text{p-quaterphenyl}^{2-}$ and the ε values were used to determine the first-order rate constant of the protonation reaction. For the analysis, the time-dependent amount of anions were simulated with a simulation software, Comsol Multiphysics, and fitted to the experimental curves to determine the first-order rate constant. We found that the first-order rate constant increased linearly against c_{EtOH}^2 for $\text{DPA}^{\cdot-}$, $\text{p-quaterphenyl}^{\cdot-}$, and $\text{p-quaterphenyl}^{2-}$, and against c_{EtOH}^3 for $\text{biphenyl}^{\cdot-}$.

Acknowledgments

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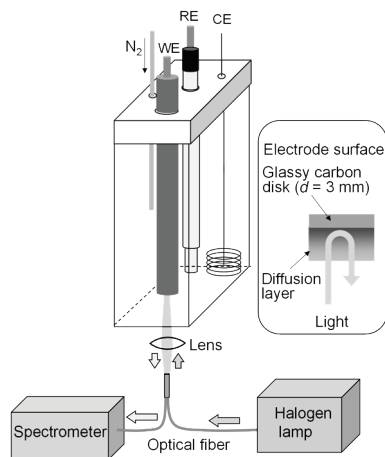


Fig. 1. Illustration of the spectroelectrochemical system used in this study.

Determination of EPA PAHs in simulated natural and artificial fires by HPLC-DAD-FLD with the application of the Multi-Component Integrated Calibration Method

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The modifications of the basic variant of the integrated calibration method (ICM) and in the version of the complementary dilution method (CDM) were adapted to multi-component analysis (MC) to obtain new calibration methodologies allowing to improve the accuracy of analytical results. Both approaches were developed conceptually, and their analytical performance was estimated based on polycyclic aromatic hydrocarbons (PAHs) determination in various air samples. To collect samples, synthetic and natural materials were combusted simulating fires (e.g., forest fires, landfills) or home users' combustion. An analytical method for determination indicated by the US Environmental Protection Agency (EPA) PAHs was developed using high-performance liquid chromatography (HPLC) with diode array (DAD) and fluorescence detection (FLD). A procedure of air samples collection (with XAD-2 sorbent) and extraction (with acetonitrile) before the chromatographic analysis was also developed. The method with fluorescence detection enables very good validation parameters for all considered PAHs: linear range 0-500 ng/mL, LOD<0.21 ng/mL, $R^2>0.999$ in most cases, and precision CV<3%. It has been demonstrated that the improved analytical results accuracy received in both proposed methods (MC-ICM and MC-ICM/CDM) was characterized by relative error (RE) smaller than 1.5%. Identified multiplicative interference effects occurring due to the coelution of other substances created during combustion were detected by analyzing the individual analytical results estimations. The results free from the multiplicative effect were calculated. Based on the research carried out for the combustion of selected materials, the formation of several main compounds (naphthalene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz[a]anthracene, and chrysene) was shown. According to RGB Additive Color Model for Analytical Method Evaluation, the MC-ICM/CDM method finally selected for all analysis is the White – method is a good candidate of choice for all applications.

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Acknowledgments

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Quantification of diclazepam and its metabolites using GC-MS for chemometric purposes

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There has been a rise in cases involving designer benzodiazepines. Given their similarity in structures and metabolic pathways, their analysis within biological samples is challenging and presents many problems for forensic and toxicological analysis and interpretation. This is particularly relevant for the designer benzodiazepine diclazepam. Diclazepam is a structural derivative of diazepam and has not been approved for human consumption. There is a concern regarding a lack of data for the expected concentrations of diclazepam's metabolites, particularly when analysing blood samples [1,2]. Additionally, diclazepam has been reported to have a metabolic half-life of approximately forty-two hours and metabolises into three active metabolites: delorazepam, lorazepam, and lormetazepam [3]. All three of these metabolites are available as individual benzodiazepine medications in their own right, complicating the toxicological interpretation of results of suspected diclazepam administration.

To aid the interpretation of these results we are developing a chemometric model which will identify the parent compound. We have gathered the analytical data for the chemometric model using a newly developed, optimised and validated GC-MS method which will be presented here.

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ABSTRACTS

**of oral presentations
in the session
of Young Scientists**

Flow analysis system for photometric determination of lactate dehydrogenase activity

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Lactate dehydrogenase (LDH, EC 1.1.1.27) belongs to the oxidoreductase groups widely distributed in the cells of all living organisms, where catalyses the equilibrium conversion of pyruvate to lactate, using NADH/NAD⁺ (a coenzyme system crucial for cellular respiration). Hence, its activity can be considered as a valuable clinical parameter in the diagnosis of diseases such as myocardial infarction [1], anemia [2] as well as various types of cancer [3].

A mechanized analytical strategy for lactate dehydrogenase activity determination will be presented. The proposed photometric method is based on the kinetic monitoring of colourful iron complexes formed in the course of their reduction by NADH. For such a bioanalytical method, a flow system based on Arduino-controlled microsolenoid pumps and valves including dedicated LED-based optoelectronic flow-through detector has been developed. Part of the presentation will be devoted to optimization of the analytical procedure and study on potential interferent effects. Finally, the practical usefulness of designed multicommutated flow analysis system will be demonstrated by the results of control serum samples analysis.

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Multicommutated flow analysis system for fluorometric determination of lipolytic activity

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Lipases are in simple words enzymes catalyzing the hydrolysis of ester bonds in several compounds commonly known as fats. But truly lipases are large and very diverse group of enzymes occurring extensively in living organisms from microbes to mammals [1]. Thus the examination of lipolytic activity is important in several fields from environmental investigations to clinical diagnostics.

In this work a straightforward Multicommutated Flow Analysis (MCFA) system for lipase activity determination is presented. The single-step detection method is based upon enzymatic hydrolysis of fluorescein esters of various carbon chain lengths. The developed MCFA system is coupled with the dedicated flow-through fluorescence detector made of integrated LEDs. In the course of this research several lipases isolated from different organisms and exhibiting different hydrolytic activity towards the fluorogenic esters were examined. Selectivity studies on the effects from some other enzyme classes were also performed. Finally, the potential utility of the developed MCFA system for controlling a microbial growth that gives the insight into bacterial enzyme excretion process will be announced.

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Simple automated flow system for the determination of lithium in pharmaceutical samples and human blood serum

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Lithium is well-known for its use in industry - as a component of lithium-ion batteries or as a component of heat-resistant glass. Of particular importance is the beneficial impact it offers on human health. Lithium carbonate is an effective drug used especially in the treatment of bipolar disorder. Mood-improving supplements containing organic lithium salts are increasingly available on the market. Lithium presents a narrow therapeutic window and is characterised by high intra- and inter-individual variability regarding its pharmacokinetic. Therefore, to maximise effectiveness and minimise adverse drug events and toxicity, it is necessary to monitor the concentration of lithium in human blood serum [1]. For this reason, it is equally necessary to control the lithium content in pharmaceutical preparations. The aim of the study was to develop an automated flow system for the determination of lithium in pharmaceutical products, as well as in human serum. For this purpose, an automated flow system based using a set of syringe pumps integrated with multi-position valves was constructed in combination with a spectrophotometric detector. The principle of the method was based on the reaction of lithium with thoron forming a dark orange complex with lithium in an alkaline medium [2]. The scope of the study included the selection of appropriate analysis conditions such as analytical wavelength, concentrations and flow rates of reagents, reaction time, along with the degree of system automation. The developed method was then verified and its usefulness was proved by application to the determination of lithium concentration in pharmaceutical products and human serum. The use of the developed system significantly decreased the consumption of sample solution and reagent, and thus the production of waste, making the method compatible with the principles of Green Analytical Chemistry. Besides, the automation of the process enabled the reduction of time of the entire analysis, providing the opportunity to use it in practical terms.

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Paper sensor-based method using a portable 3D-printed platform and smartphone assisted colorimetric detection for ammonia and sulfide monitoring in anaerobic digesters and wastewater

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A simple and low-cost field-deployable method using paper sensors in combination with a 3D-printed portable digital image colorimeter to simultaneous determination of ammonia and sulfide in wastewater samples has been developed. Ammonia and hydrogen sulfide are harmful pollutants for the environment and important toxicants inhibiting anaerobic digestion, therefore, they are key monitoring parameters during wastewater treatment. The sample preparation system consisted of a microplate (sample reservoir) and sensor paper discs placed inside the microplate sealing system. The quantitative conversion of the ammonium ion to molecular ammonia and dissolved sulfide to hydrogen sulfide occurred by adding NaOH and HCl, respectively; followed by pervaporation and gas diffusion across a Teflon membrane into paper discs impregnated with bromothymol blue and reagents for the methylene blue assay. No preliminary sample treatment was required. The detection system consisted in a 3D-printed photographic lightbox with LED plates (powered by a battery) and a smartphone. A code has been developed in ImageJ for the fast colorimetric measurement of digital images. Optimization of factors affecting image capture and chemical determination was performed. Supernatant of anaerobic digesters and wastewater samples were analyzed with the proposed device and satisfactory precisions (RSDs < 10%) and recoveries (91–105%) were obtained.

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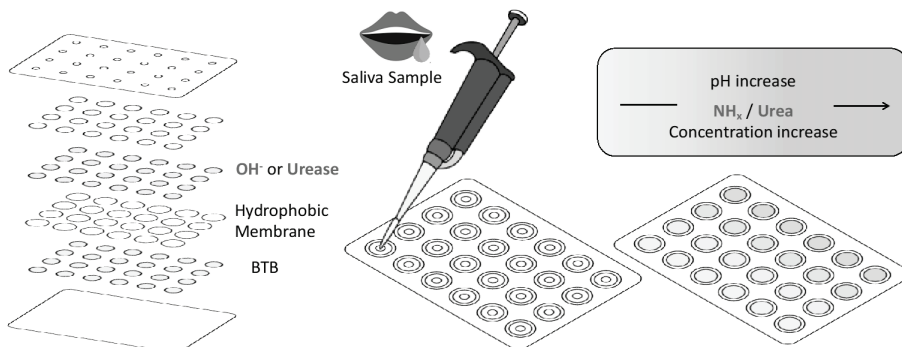
On-hand tool for CKD monitoring – design of a couple of μ PADs for ammonium and urea determination in saliva

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Chronic Kidney Disease (CKD), is a condition in which the kidneys malfunction, causing the accumulation of metabolic toxins. Urea is a nitrogenous product of the protein metabolism by the urea cycle, resulting from the toxic NH_x conversion. Since its primary form of secretion is the kidney, urea is one of the most used biomarkers for kidney failure. Microfluidic paper-based analytical devices (μ PADs) are a recent concept that has gained a lot of attention in the last few years as on-hand, point-of-care biosensors. These devices have gained a great interest since are capable of performing the analytical determinations in less time and with a more simpler, economic and user-friendly procedure.

In this work, a couple of μ PADs were developed for the determination of NH_x and urea in saliva samples. The determination was based on color change of bromotymol blue indicator (BTB) after the ammonia diffusion through an hydrophobic membrane [1].



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Francisca T. S. M. Ferreira would like to thank the PROM Programme – “International exchange scholarship for doctoral students and academic staff” for financing participation in the conference.

Development of a microfluidic paper-based analytical device (μ PAD) for iron determination in water

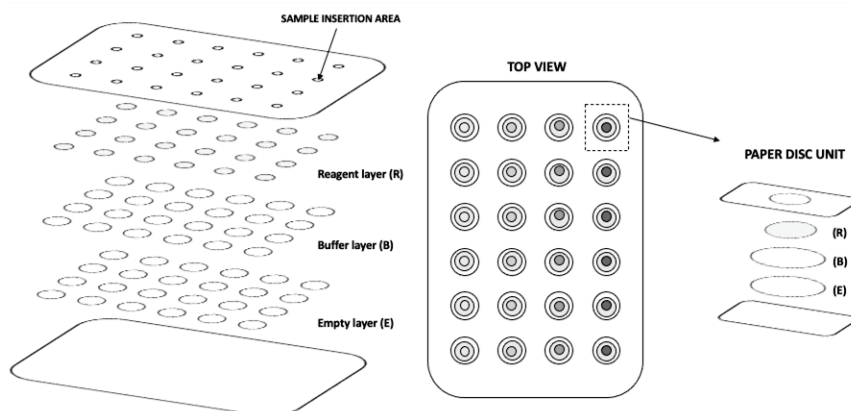
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Ingestion of waters with high iron concentration can cause several risks to human health. In this work, a new microfluidic paper-based analytical device (μ PAD) was developed to quantify iron in surface and waste waters using a new fluorescent 3-hydroxy-4-pyridione ligand. Under optimal conditions, a linear calibration curve was obtained in the concentration range of 0.25 – 2.00 mg/L with a limit detection of 0.11 mg/L. The proposed μ PAD method was successfully applied to the determination of iron in surface and waste water samples with no need for sample pre-treatment, and its accuracy evaluated by comparing to the AAS standard method (RD < 10%). The developed μ PAD is simple, economical, sensitive and present low analysis time. Each μ PAD uses low amounts of reagents, approximately 0.5 mg of ligand and 17 mg of sodium hydrogenocarbonate and sample consumption of 100 μ L.



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A fully high-throughput microfluidic paper-based analytical device fabricated by using an in-house computer-controlled X-Y knife plotter implemented with the deposition of reagents via technical pen-plotting for point-of-care testing

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Nowadays, a microfluidic paper-based analytical device (μ PAD) as a detection platform has gained attention due to their unique advantages of low cost, ultimate portability, ease of use, and disposal. The μ PADs have various techniques for fabrication, such as mask-guided patterning techniques, which have some limitations such as low reproducibility of the device, the use of hydrophobic reagent to create the channel, and low resistivity of solvents used in the detection system. To conquer these problems, an in-house computer-controlled X-Y knife plotter was used for μ PADs fabrication and a pen-plotting for reagent deposition using a technical pen. These provide less reagent consumption, simplicity, rapidity, reproducibility, and high device throughput. Furthermore, the paper device was laminated (LPAD) to increase mechanical strength and reduce sample evaporation during the analysis. Various prototypes of 2D and 3D devices by our proposed method were successfully fabricated. The fabricated 3D- laminated paper-based analytical device (LPAD) was then used for simultaneous glucose and total cholesterol (TC) determination in whole blood using the LF1 membrane as a sample zone for red blood cell separation. In addition, i1 Pro mini spectrophotometer was used to detect the appeared color on LPAD directly, and the obtained results were well agreed with the hospital method, which follows the clinical criterion. Finally, these enable the industry-scale mass production of simple chemical sensing devices with well-balanced cost and performance, and this concept contributes to the expansion of the applicability of any well-known target analyte in various applications.

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Thread-based microfluidic sensor for non-invasive lithium monitoring

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Lithium is a commonly prescribed remedy for some mental diseases, for example bipolar disorder. Patients undergoing lithium treatment must monitor its level regularly to ensure that it stays within a very narrow therapeutic range (0.4 – 1.2 mmol·L⁻¹ in serum) [1]. Gold standard methods for lithium determination in clinical laboratories include potentiometry with Li ion-selective electrodes, atomic absorption spectrometry or atomic emission spectrometry [2]. All of them require specialized equipment and trained staff, which creates a need for a simple, reliable and fast sensors for Li monitoring in biological fluids

In the presented research, a thread-based analytical device for salivary lithium determination was developed. Lithium ion recognition was achieved by incorporating chromoionophore-ionophore chemistry onto thread [3]. To boost the selectivity of the developed sensor, a highly efficient lithium extractant was added to the sensing cocktail, alongside its regular components.

The developed sensors allowed for lithium determination in a range from $8.8 \cdot 10^{-4}$ to 0.95 mol·L⁻¹ with remarkable precision of 0.3% and within a very short time (10 seconds). Sensors were validated with real saliva samples, analyzed without any pretreatment.

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Functionalization of commercial hydrophilic-lipophilic balanced copolymer for automatic magnetic dispersive micro-solid phase extraction of surface water contaminants

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In this research, we present an easy approach to magnetic dispersive solid phase microextraction and its automation for the enrichment of water contaminants. Here, SupelTM-Select HLB (Hydrophilic modified styrene polymer) beads were magnetized by introducing magnetite nanoparticles into the sorbent. The so-functionalized sorbent was employed in the development of an automated dispersive solid phase extraction methodology using the Lab-In-Syringe technique [1]. The methodology followed a previous study on using magnetic nanoparticles [2] by enabling automated exchange of the sorbent, i.e., developing an in-syringe bead injection concept. This involves, the aspiration of optimized volumes of sample and bead suspension into the syringe void and dispersion by the aid of a magnetic stirring bar placed inside the syringe. By automatic activation and deactivation of magnetic stirring process, the analytes were retained on the dispersed sorbent, which was, when the stirring stopped, captured on the magnetic stirring bar. Afterwards, the sample solution was discarded, and beads were washed in-syringe with water. Then, an optimized eluent was aspirated to strip the analytes from the anew dispersed sorbent. The extraction system was coupled online to a liquid chromatograph for the determination of model analytes mebendazole, bisphenol A, benzyl 4-hydroxybenzoate, diclofenac, and irgasan. Key parameters such as elution time, extraction time, sorbent suspension and eluent volume, as well as phase separation time were optimized. Limits of detection ranged from 1.2 µg/L to 6.5 µg/L with < 7 % RSD for all the analytes at 25 µg/L level were obtained. The method was linear over a tested range from 5 µg/L to 200 µg/L. The developed method was successfully implemented for the analysis of the chosen contaminants in surface water (lakes and rivers) with recoveries between 78.4 % and 105.6 %.

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Application of microemulsion electrokinetic capillary chromatography for the analysis of dyes extracted from polyester fibers

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One of the most important traces revealed at a crime scene is a microfiber. They provide information that may confirm or exclude the suspect's relationship with a place or a victim. Today, there is a significant increase in the popularity of polyester fiber. At the same time, there is a growing interest in disperse dyes used to color this type of fiber. Therefore, they are likely to be the subject of forensic analysis.

The aim of the study was to develop a method for the analysis of disperse dyes extracted from polyester fibers. Nine commercially available disperse dyes, which were used to dye three polyester fabrics, were tested. The developed method was also applied to the analysis of 15 real samples - polyester fibers collected from clothing. Extraction of dyes from 1 cm long threads was carried out in chlorobenzene at 100°C for 6 hours. The separation was performed using microemulsion electrokinetic capillary chromatography with DAD detection. Microemulsion (ME) based on a borate buffer with an organic phase of n-octane and butanol and a mixture of surfactants: SDS and sodium cholate was used. The addition of isopropanol, 2-hydroxypropyl- β -cyclodextrin and 2-hydroxypropyl- γ -cyclodextrin to ME resulted in notable improvement in resolution and selectivity. The content of additives was optimized by using the Doehlert matrix experimental design.

Values of the coefficient of variance obtained in the validation process, illustrating the repeatability and intermediate precision of the migration times fit in the range of 0.12-1.43% and 0.58-3.21%, respectively. The proposed method may be used in the discriminant analysis of fibers and is promisingly employable in forensic practice.

Acknowledgments

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The effects of imidazolium-based ionic liquids on the separation efficiency of biogenic amines during MEKC analysis

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Ionic liquids (ILs) such as imidazole can be used to prevent the sorption of analytes onto the quartz walls of the capillary. Coating the capillary wall with a cation layer increases its surface stability, consequently improving the repeatability of separation process. The aim of the study was to evaluate the effect of the addition of ILs to the base electrolyte (BGE) on the separation efficiency of the tested BAs using the micellar electrokinetic chromatography (MEKC) technique. Specifically, this study focuses on 12 ILs built from cations containing an imidazole ring with different alkyl substituents and anions, as well as one IL containing a pyridinium cation with tetrafluoroborate anion. ILs were used as a BGE component in four concentration ranges and analyzed for improving the electrophoretic separation of selected amines, such as homovanillic acid (HVA), vanillinmandelic acid (VMA), dihydroxyphenylglycol (DHPG), 3-methoxy-4-hydroxyphenyl glycol (MHPG), methanephine (M), normetanephine (NM), dihydroxyphenylacetic acid (DOPAC).

The results showed that the most effective ILs added to the BGE were those with a chloride anion (1-hexyl-3-methylimidazolium chloride [HMIM+Cl⁻] and 1-ethyl-3-methylimidazolium chloride [EMIM+Cl⁻]) [1]. Improved separation efficiency was also obtained for the BGE containing 1-hexyl-3-methylimidazolium hexafluorophosphate [HMIM+PF₆⁻]. Additionally, increasing the chain length in position 1 of the 3-methylimidazole ring of the liquid with the hexafluorophosphate anion helps to stabilize the separation conditions and affects the repeatability of the migration times. The results presented in this study revealed that alkylimidazole ILs containing anions, such as [Cl⁻], [BF₄⁻], or [MeSO₄⁻] dissolve very well in the BGE and the ability to stabilize and dynamically modify the capillary wall surface by ILs increases as the chain-length at the 1-position of the imidazole ring increases [1]. Overall, this study provides a critical evaluation of the impact of different types and concentrations of ILs on the performance of the MEKC method during the analysis of selected BAs.

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Application of the capillary electrophoresis technique in real life

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Today, it is imperative to give special attention to the use of an analytical instrumental method in everyday life for analysing samples. Requirements for the separation technics are challenging (low detection and quantification limits, fast analysis time, high accuracy, etc.). One of the separation techniques that deserves special attention is constantly evolving capillary electrophoresis (CE), which exactly meets the requirements. It is widely used in areas such as forensic chemistry (testing psychoactive substances, post-blast explosive residues), clinical applications (disease diagnosis, therapeutic drug monitoring, biopharmaceuticals, metabolic profiling of biological samples), and environmental pollution (airborne volatile substance).

In this presentation we would like to show an application of the CE methods such as: CE-C⁴D (capillary electrophoresis with dual capacitively coupled contactless conductivity detection) [1], [2], CE-FD (capillary electrophoresis coupled to a deep ultra-violet fluorescence detector) [3] in real life. Among the proposed devices, the Drug Hunter Analyzer (a transportable apparatus enabling the identification of psychoactive substances in a saliva sample using CE-FD) is particularly distinguished. The research and development of Drug Hunter is still ongoing, and the apparatus will be commercialized in the future.

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Dynamic coating of phenyl-silica based stationary phases with ionic liquids used as mobile phase additives in the LC technique

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Ionic liquids (ILs) belongs to huge group of compounds commonly called “green solvents” due to its low vapor pressure, thermal stability or non-flammability. Structurally, they form numerous combinations of organic cations with organic or inorganic anions, both of which may be involved in chemical processes during their application. Consequently, ILs can be interesting modifiers in the separation of analytes as a result of their addition to the mobile phase and then dynamic coating the surface of the stationary phases.

This study was aimed at the gain insight into the interactions occurring during chromatographic separation, where the phenyl, ether-linked phenyl and pentafluorophenyl based stationary phases were applied, and their performance was tested in the presence of thirteen different ILs. The changes in the column behavior were evaluated on the basis of chromatographic parameters, such as retention factor (*k*) peak area (*A*), number of theoretical plates (*NA*) and tailing factor (*Tf*) obtained after LC-FL analysis of four anthracycline antibiotics.

The findings of the studies proved that the presence of an aromatic ring in the structure of the ILs allowed for π - π interactions with phenyl stationary phases, and thus the strong analyte retention on phenyl column were significantly reduced. Moreover, by selecting the appropriate IL structure, it was also possible to improve other separation parameters.

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The benefits of coupling flow injection systems to ICP-MS in the analysis of biological samples

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Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a very sensitive and powerful technique, which finds application in inorganic analysis of biological samples like blood plasma, serum or urine. Unfortunately, such samples possess complex matrices that can be challenging in terms of occurrence of possible interferences or in reaching the acceptable level of total dissolved solids (TDS). Therefore, preparation of biological samples is very often time-consuming and associated with the need of large dilutions, matrix removal or pre-concentration. Coupling flow injection analysis (FIA) to ICP-MS offers reduction of that time and makes sample preparation easier or redundant. In the presentation, several examples of the use of FIA with ICP-MS technique, that can be found in the literature, have been presented.

The implementation of flow injection system by K. Kobayashi et al. in the analysis of blood samples via ICP-MS enabled simultaneous determination of selenium, copper and zinc without prior extraction [1]. Another example of FIA implementation to ICP-MS allows to determination of selenium in serum and cerebrospinal fluid in which the total sample volume was kept below 130 μL and where analysis time per sample was typically below 2 min due to the usage of dedicated flow injection system [2]. According to the authors, the proposed methods are faster than the determination of selenium with hydride generation.

Urine samples usually require dilution to avoid exceeding of the TDS limit caused by its major components: Na, Mg, Ca, K and Cl. Coupling ICP-MS to a flow injection system allows for on-line dilution and thanks to that reduction of sample volume, shorter preparation time, improved precision and elimination of potential contamination [3].

Moreover, recent trends in the use of FIA with ICP-MS and the advantages and disadvantages of combining these methods have been discussed.

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ABSTRACTS

of poster presentations

Exploiting flow injection titration with CO₂ exclusion to determine free fatty acids at low concentrations

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Flow injection (FI) titration is normally performed under steady-state condition, where peak width or peak area of FIagrams was employed in a logarithmic function calibration graph to quantify the analyte. In this work, we proposed a simpler FI- titration at non-steady state that provides a linear calibration graph plotting between peak height and analyte concentration. By using CO₂ trapping tube to exclude CO₂ from the hydroxide reagent, low concentration of the base can be used for titration of free fatty acid (FFA) at low concentrations. A two-line FI system using a simple mixing coil instead of a magnetic mixing chamber was constructed. After introducing a standard or sample into the carrier stream, the injected zone was propelled to merge with a reagent stream containing sodium hydroxide and phenolphthalein indicator. Neutralization reaction causes a decrease in pH of the solution, leading to a change of the indicator color from pink to light pink or colorless. The fading of pink color was monitored by a colorimeter and recorded as an FI-peak, of which peak height is linearly proportional to FFA concentration in the sample. Using palmitic acid as a standard FFA, a linear calibration range of 0.03-0.7 mM was obtained, with a detection limit of 0.01 mM. The low calibration range allows the dilution of oil samples to eliminate the influence of oil viscosity, which is normally occurred in the steady-state FI-titration. The developed method was applied to determine FFA in fresh and used edible oils, giving high sample throughput (43 injections h⁻¹) and low reagent consumption (2.8 mL injection⁻¹). Recoveries obtained from spiking standard FFA into the samples were found in the range of 92-110%. The contents of FFA determined by the proposed method agree well with those of the standard titration method.

A mono-segmented flow in hydrodynamic sequential injection system for online monitoring of ammonium in aquaculture

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Ammonium is an important water quality parameters to be monitored in the aquaculture. In this work, we proposed a hydrodynamic sequential injection (HSI) – monosegmented flow system for automated online monitoring of ammonium in aquaculture. Water sample was aspirated into the system to mix completely with reagent solutions in a monosegment sandwiched by two air plugs. Then, the colored product zone was detected by a homemade colorimeter that incorporated to the HSI manifold. Under the optimum condition; the proposed system provided linear ranges of 0.5 - 5.0 mg L⁻¹ with correlation coefficients of 0.9984. The limit of detection of 0.13 mg L⁻¹ was obtained. The developed system has advantages of low chemical consumption, high precision, compact and cost-effective instrument, automatic operation, and simple monitoring system. The application of the developed system to monitoring a fish pond will be presented.

Kinetic flow-based method for plasmonic sensing of ultratraces of diclofenac in seawater

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Plasmonic immunoassays developed by combining enzyme-linked immunosorbent assay (ELISA) and nanoparticles (NP) of noble metals are good substitutes of classic ELISA counterparts for naked-eye readout of biomolecules and small xenobiotics at trace levels. However, the dynamic aggregation phenomena of gold nanoparticles (AuNPs) complicate their detection in steady state assays characterized by poor repeatability on microplate readers. A competitive ELISA combined with the in-line generation of AuNPs in a stopped-flow based setup is reported in this communication to improve the robustness of the method by means of flow chemistry. Glucose oxidase enzyme was conjugated to a secondary antibody and yielded hydrogen peroxide that was injected in the system and acted as the reducing agent of the Au(III)/citrate reagent. The flow network was composed of two microsyringe pumps (one containing Au(III) and the other water as a carrier), a 24-position multiposition valve to sample automatically from several ELISA solutions, and a reaction coil. The enzyme-mediated surface plasmon resonance (SPR) of AuNPs generated in-situ was real-time monitored using a USB miniaturized photometer. The analyte specificity and high sensitivity of these immunoassays allowed the analysis of complex matrices without any pretreatment of the sample. In this work, the determination of diclofenac in seawater was proposed as a proof-of-concept using monoclonal antibodies against the analyte. The automatic fluidic approach has a reproducible timing in AuNPs nucleation and growth along with the unsupervised absorbance detection with a dynamic range for diclofenac spanning from 0.01 to 10 $\mu\text{g L}^{-1}$ and a LOD of 0.001 $\mu\text{g L}^{-1}$, that is one order lower than that of a conventional colorimetric ELISA. Repeatability and intermediate precision were <4% and <14%, respectively, as compared to RSDs as high as 30% as obtained with the batchwise plasmonic ELISA counterpart involving end-point detection.

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Design of flow injection spectrophotometric system for penicillamine determination in pharmaceutical formulations

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A new, sensitive, rapid, and precise flow injection spectrophotometric method for the determination of penicillamine (PEN) in pharmaceutical formulations has been developed and validated. Penicillamine is a synthetic amino acid that contains an additional SH group and can form non-toxic, water-soluble chelates with heavy metals, which are then excreted in the urine. PEN was the first chelator used for Wilson's disease, and it can also be used for lead, mercury and arsenic poisoning. The method is based on a fast one-step redox reaction wherein PEN reduces the Cu(II)-bathocuproine complex to the Cu(I)-bathocuproine complex, which exhibits absorption maxima at 483 nm. Therefore, a simple two stream single reagent manifold was used. By decreasing the inner volume of the flow cell (from 160 μL to 30 μL), the miniaturization of other parts of the manifold was enabled. Therefore the sensitivity of PEN determination was advanced, and sampling frequency increased. Optimization of experimental conditions and manifold parameters was carried out through the univariate method. The reagent, prepared in acetic buffer solution ($\text{pH} = 5$), was merged with a sample (200 μL) and injected into a carrier stream of distilled water at a confluence point, where streams combined. The flow rate of the carrier stream was 6 mL/min, and the reagent was pumped at the rate of 2 mL/min. The reaction coil, 30 cm long and 0.8 mm I.D. (volume 150 μL), was kept at room temperature. Since the flow rate through the reactor was 133 $\mu\text{L}/\text{sec}$, the incubation time was just over a second, yet sufficiently long for the reaction to reach a steady state. A linear calibration curve was established in a concentration range of PEN from 2×10^{-7} to 4×10^{-5} mol L^{-1} with regression equation $y = 8.48 \times 10^3 x - 0.0011$ ($R^2 = 0.9996$). The novel FI-method is sensitive enough to enable LOD below 8 ppb of PEN at a sampling rate of 180 s/hr without expensive instruments. In addition, method is precise (RSD 0.9%, $n = 100$) and accurate (recovery in the range from 98.7% to 101.6%). The proposed method can be applied for the determination of PEN in pharmaceutical formulations since usual excipients and other present active substances in pharmaceuticals do not interfere.

Flow analysis system for the control of microbial growth

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The monitoring of bacteria life cycle is of the utmost importance in medicine, food and environmental analysis as well as in agriculture. The significant information includes the data about period of nonreplication, which is the period of the bacteria adaption to new environment, pace of divisions and its lifetime [1]. Although, there are several methods for estimation of colony forming unit (CFU), many of the techniques are time-consuming, requiring properly trained research personnel to operate them [2]. Moreover, they are rather difficult to be applied as real-time/on-line operating monitors.

This contribution is devoted to the development of fully-mechanized turbidimetric multicommutated flow analysis (MCFA) systems for the monitoring microbial growth. The concept of the flow analysis system designs for such purposes will be presented, starting with a flow manifold for analysis microbiological McFarlands standards, through the systems allowing simulation and modelling of microbial growth, and ending with the MCFA-based monitor applied for the control of real microbiological cultures.

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Determination of acid dissociation constant of selected biologically active compounds using a flow system with spectrophotometric detection

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The acid dissociation constant (K_a) is one of the most important physicochemical parameters. This parameter characterises the acid-base properties of chemical compounds. It is particularly important in relation to biologically active substances, including drugs. It allows to determine the ionisation profile and pharmacokinetic properties of a drug, and to predict its behaviour in different compartments of the human organism [1]. As flow analysis is one of the most widely developed process improvement concepts in recent decades [2], it was agreed to employ the technique to construct a flow system for determining pK_a values. The study presents a novel and rapid method for determining the pK_a values of selected biologically active compounds (warfarin and coumarin derivatives) using pH gradient generation in continuous flow with spectrophotometric detection. The developed method is based on the formation of a pH gradient in a flow system by continuously varying the flow of combined streams of the acid and base forms of the Britton-Robinson buffer in the pH range 2-10. The principle of the method was to determine the pK_a value based on two defined relationships - the dependence of the dilution factor on the time of analysis and the pH on the dilution factor, and the time of analysis at the point of inflection of the curves of signals registered for the two forms (acidic and basic) of the tested compound. The method was verified using acid-base indicators and then applied to determine the pK_a value of warfarin and other coumarin derivatives. The ionic strength correction was introduced in the calculations. The pK_a values obtained were consistent with the literature values - the relative error (RE) did not exceed 4.3%. The values were determined with very good precision ($RSD < 1.5\%$). The developed method of determining the dissociation constant allows mechanization of the process, determination of the parameter in a short time with low consumption of reagents.

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Flow system for sulfur speciation analysis in bioethanol with ICP OES detection

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Sulfur is one of the most frequently determined elements in liquid fuels for motor vehicles and in biocomponents of fuels. It is caused by the negative influence of sulfur on the performance characteristics of liquid fuels as well as SO_x, resulting from the combustion of fuel, on the natural environment. Bioethanol is one of the components of liquid fuels for which sulfur, and sulfate ions are determined. In the presented studies, speciation analysis of sulfur was performed by direct determination of sulfur and indirect determination of sulfate ions. For this purpose, a flow system with ICP OES detection was developed, which enabled speciation analysis of sulfur within a single procedure. Sulfur was determined directly, while sulfate ions indirectly using the on-line precipitation reaction of barium sulfate, and the analytical signal of barium depending on the amount of precipitated barium sulfate. The flow conditions and concentrations of reagents for the precipitation of barium sulfate in the flow system were selected. The linear range of sulfur determination was tested for concentrations to 10.0 mg/kg, and for sulfate ions to 12.0 mg/kg. The quantification limits for sulfur 0.15 mg/kg and 0.18 mg/kg were determined for the lines S I 180.731 nm and S I 182.034 nm, respectively, and for sulfate ions – 0.50 mg/kg. The correctness of the determinations was proved on the basis of analysis of standard solutions with defined concentrations of analytes and a sample from interlaboratory studies. The method was applied to determine sulfur, and sulfate ions in bioethanol giving the results consistent with the reference method.

Flow system for green and integrated sample pretreatment for the determination of antiviral drugs in surface waters

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The widespread access to a variety of chemical agents, especially drugs, poses a significant danger to the environment. At the time of prevailing pandemic, it can be expected that the extensive use of antiviral agents may contribute to increased pollution of the environment, particularly surface waters [1]. The methods currently used to prepare water samples for the determination of these drugs are both complicated as well as characterised by many limitations, hence there is a growing need to develop novel and more straightforward solutions [2]. The aim of the study was to develop an automated system for integrated on-line sample pretreatment for the determination of selected antiviral drugs in surface waters combining solid phase extraction (SPE), flow analysis and UHPLC-MS/MS (Ultra-High Performance Liquid Chromatography - tandem Mass Spectrometry) technique. The presented approach, integrating various steps of on-line sample preparation for analysis, corresponds to the principles of the so-called Green Analytical Chemistry. The general scope of the research covers i.a. selection of the flow technique and conditions for SPE extraction, development of the procedure for the determination of selected antiviral drugs by UHPLC-MS/MS technique, validation of the developed method and verification of its usefulness by applying it for determination of antiviral drugs in surface waters. The presentation provides the results of preliminary studies, which included the development of procedure for water sample preparation and analyte preconcentration, as well as the results of testing and selection of separation conditions and instrument operation. The proposed integrated flow system for on-line sample preparation in combination with a sensitive UHPLC-MS/MS detection will enable the development of an innovative method for the determination of antiviral drugs in surface waters.

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Automated flow system with a direct injection cell for simultaneous determination of Fe(II) and Fe(III) in beverages

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Flow analysis is one of the most successfully developed concepts for the mechanisation of analytical processes in recent decades. The applications of flow analysis are characterised by a wide range of advantages, including the possibility of performing complex reactions under controlled conditions, the simplification of multi-step reactions, the reduction of reagent consumption and waste generation [1]. The speciation analysis of iron in groceries is an important issue due to the bioavailability of this element and its beneficial effect on human health. Additionally, in wines it influences their quality and stability. The study presents the development of a simple automated flow system with an original direct injection cell (DIC) and spectrophotometric detection for the simultaneous determination of Fe(II) and Fe(III). In the proposed method, the sample for analysis was prepared directly in a syringe. A mixture of 1,10-phenanthroline and 5-sulfosalicylic acid was added to the sample forming complexes with Fe(II) and Fe(III), respectively. Then the solution was introduced into the DIC and as a result, a signal for Fe(II) and Fe(III) was registered. Subsequently, EDTA solution was introduced into the direct injection cell to form a colorless complex with Fe(III), so the absorbance for Fe(II) complex with 1,10-phenanthroline was measured. Two-component calibration according to the 22 factorial design was used to determine Fe(II) and Fe(III) concentrations. The procedure was verified by the simultaneous determination of analytes in synthetic samples. Afterwards, the applicability of the method was investigated by performing an iron speciation analysis in samples of artesian waters, white wines and nettle juice. The results were consistent with those obtained with the ICP-OES reference method.

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Sequential injection method for the biparametric determination of iodide and creatinine in urine

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Iodine Deficiency Disorder is an ongoing worldwide recognized problem with over two billion individuals having insufficient iodine intake [1]. Urinary iodide concentration is the primary indicator of population iodine status and supplementation strategies evaluation [2]. Creatinine (Cr) is a recognized indicator of renal health, and an internal standard for normalization of other clinical biomarkers [3]. The aim of this work was to develop a simple, and fast method for iodine and creatinine monitoring in urine, without sample pre-treatment, easily to be deployed in clinical/analytical context to simultaneously assess the iodine nutritional status (by I or as I/Cr), and potentially kidney dysfunctions. The determination of iodine was based on the potentiometric detection of iodide using inline spiking of the samples. Creatinine determination relied on the routinely used Jaffe's colour reaction [4]. Both determinations were studied to be carried out in a flow approach, with linear working ranges covering the levels found in human urine, and combined in a single sequential injection (SI) manifold to attain a biparametric determination. The results were comparable to those obtained by reference methods, and recovery tests further confirmed the method accuracy. The developed SI method was effectively applied to urine samples, enabling 25 determinations for both parameters in one hour, requiring less than 2.5 mL of urine sample for 3 replicates.

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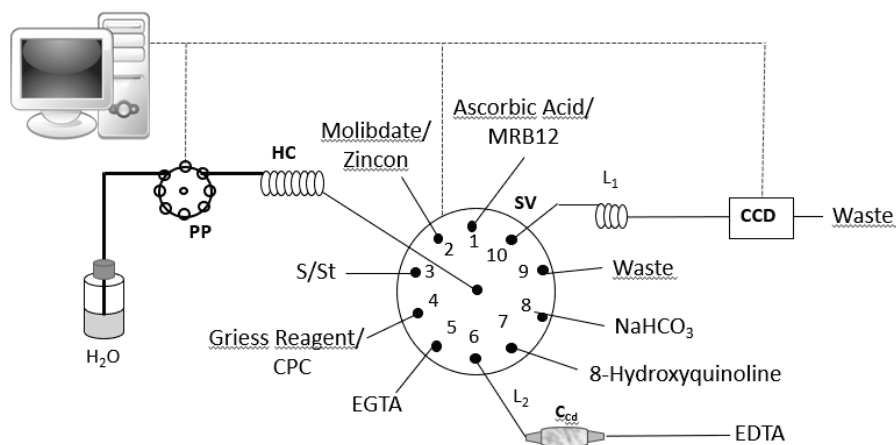
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Sequential injection method for the determination of macro and micro nutrients in soils

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The objective was to devise a single flow analysis manifold able to monitor macro and micro nutrients in soil leachates and extracts. Several analytes were targeted, namely nitrate, nitrite (1) and phosphate (2) as essential nutrients, followed by some metal ions such as calcium, magnesium (3), iron and manganese (4). The sequential injection systems for the individual determinations were already reported, and now a single manifold (Figure) for a multiparametric determination is proposed.



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Application of sequential injection lab-at-valve analysis to simultaneous spectrophotometric and/or kinetic determination of reducing agents, Si(IV) and P(V)

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The usual configuration of the FiaLAB® 3500 based SIA system was supplemented with an external mixing chamber (MC) in which the mixing of reagents and, if necessary, chemical reaction was carried out. The use of air bubbling instead of magnetic bar agitation simplified the manifold configuration. Conducting the mixing process in the RC guaranteed efficient rapid mixing of the reagents. After the completion of the reaction, the analytical signal was measured in the stop-flow mode. The program was written in such a way that the air bubbles could be completely separated. Thus, the problem of retaining bubbles in the flow cell was completely solved. For the simultaneous determination of ascorbic acid and methyl dopa in artificial mixtures and blood plasma, the SIA-LAV system was used. The use of the 18-molybdodiphosphate heteropoly complex made it easy to find, by varying the acidity, the conditions under which the reaction rates of various reducing agents with the reagent differ significantly. After that, simple and effective graphical kinetic methods were applied simultaneously to resolve the analytical signal (the sum of the kinetic curves) for each of the components of the mixture. For the analysis of mixture of analytes, mean centering of ratio kinetic profiles and, for the first time, kinetic profiles ratio difference methods have been proposed. Ionic association complexes formed between polymethine dye Astra Phloxine and 12-heteropoly molybdates of phosphorus(V) and silicon(IV) were used as analytical forms to develop SIA-LAV method for their simultaneous determination. Carrying out a chemical reaction and mixing in an external MC made it possible to adapt the mixing conditions to the requirements necessary for the complete formation of specific IA. The achieved sensitivity ($DL = 0.05 \mu M$) was an order of magnitude higher than when using the classical SIA approach [1]. A large excess of P(V) to Si(IV) and vice versa does not interfere with each other even without the use of masking agents.

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Determination of long-chain fatty acids in anaerobic digester supernatant and olive mill wastewater exploiting an in-syringe dispersive liquid-liquid microextraction and derivatization-free GC-MS method

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Long chain fatty acids (LCFA) are produced from the lipid hydrolysis of large amounts of fats, oils and greases (FOG) present in domestic and industrial wastewater. In biological wastewater treatment processes, such as anaerobic digestion, LCFA have been identified as the main inhibitory factor of microbial activity. Therefore, LCFA monitoring is important because it determines the performance of the depuration process and the biogas production. Gas chromatography with mass spectrometry (GC-MS) stands out among the analytical techniques for LCFA [1]. However, reported sample preparation methods involve derivatization and a subsequent extraction process. These procedures are time and solvent consuming and increase waste generation. To overcome these problems, the dispersive liquid-liquid microextraction (DLLME) technique is an interesting alternative to extract and preconcentrate the analytes [2]. DLLME procedure was performed automatically in a Lab In a Syringe using a magnetic stirring assisted (MSA) system for a fast and homogeneous extraction [3]. The aim of this work was to develop an automated extraction method, without derivatization, for the preconcentration of LCFA before analysis by GC-MS. The method was applied to quantify five LCFA (C14-C18) in supernatants of anaerobic biodigesters and in olive mill wastewaters. The proposed method is simpler, faster and environmental friendlier than other referenced manual procedures. This is the first reported automated method for extraction, preconcentration and individual quantification of LCFA with no need of derivatization.

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Determination of creatinine in urine using online solid-phase extraction

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Creatinine in human urine was determined by a method based on *Flow Programming* [1] using a Sequential Injection Analysis manifold equipped with a *hybrid switching/injection valve*. The combination of online solid-phase extraction using strong cation-exchanger sorbent and direct UV spectrophotometric detection at 235/270 nm provided the necessary method selectivity. The column was packed by means of *Bead Injection* [2] in a channel of the hybrid valve and regularly renewed when the recovery significantly dropped. The method compiled sorption of creatinine at pH 2.4, washing out of urine matrix with 50% acetonitrile, and elution of creatinine with 1% ammonium hydroxide. The eluent/matrix wash/sample/standard zones sequence was created in the pump holding coil and then flushed into the column. The sample volume was automatically set to 10 µl or 20 µl based on the signal of the 100× times diluted urine. A single run duration was <3 min. Method relative standard deviation was <5.0 % (n=6). A calibration range was linear 0.02-0.40 µg creatinine ($R^2 = 0.9959$). The standard addition method used three different volumes of one working standard solution for quantification. The method range covered 1.0-20.0 mmol/L of creatinine in urine. The accuracy of the method was compared and statistically tested with the results measured by a routine enzymatic assay.

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Green analytical methods for quantitative analysis using natural reagent: Application of orchid flower extract as acid-base indicator and as specific reagent for copper ion analysis in flow-based system

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In Thailand, orchid flower is one of the most ubiquitous plant used in traditional culture. However, according to the COVID-19 pandemic since 2020, orchid cut-flower industry was also affected and could not be exported. Therefore, the vast majority of disposed orchid need to be utilized. Green analytical chemistry methods employing crude aqueous extracted from orchid flower for analytical purpose were presented. In the orchid extract, there are many anthocyanin compounds which commonly known as acid-base indicator. Firstly, acid-base titration using the orchid extract as end point indicator was demonstrated. The results showed no significantly different from standard AOAC as well as potentiometric titration. Next, the orchid extract was also utilized as a reagent in flow injection analysis (FIA) incorporating a gas diffusion unit (GD) for determination of ammonia nitrogen. As a result of diffusion process in the GD, the aqueous orchid acceptor became more alkaline and its color changed from purple to green. This concept was applied to chemical fertilizer samples and waste water samples from agricultural fields. The results of our method agreed well with the reference OPA method. Finally, the orchid extract has been utilized as a specific reagent in sequential injection analysis (SIA) in order to potentially determine copper ion. The color change of anthocyanin-copper complex was measured. The analysis results of water sample and dietary supplements showed no significant difference between our method and FAAS. These indicated that orchid extract was acceptable to be used in the field of analytical chemistry which were economical, simple, accurate and reliable.

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Lab-in-syringe for automated miniaturized preparation of magnetic immunosorbent for SARS-CoV-2 isolation

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Immunomagnetic separation is a method of choice when isolation of a specific analyte or structure is required. In this approach, affinity reagents, e.g., antibodies or aptamers, are non-/covalently bound to the surface of preactivated magnetic beads. In the research lab, the most widely used technique for magnetic immunosorbent preparation is a batch approach that requires a multitude of manual steps such as repetitive liquid uptake and pouring of the reagents, switching the tube position between the magnetic separator and rotator or vortex, etc. The main goal of our work was to miniaturize and automatize the procedure to obtain a reliable and reproducible method for the preparation of the required small amount of magnetic immunosorbent (200 – 500 µg) using the Lab-In-Syringe (LIS) platform. The LIS system was composed of a computer-controlled automated syringe pump featuring a 12-port multiposition valve head and a 1 ml glass syringe. In-syringe mixing of all solutions including bead suspension and recollection was enabled by an integrated magnetic stirring bar and external stirring induction system for the creation of a rotating magnetic field. Solutions were aspirated via PTFE tubing or introduced to the system via an open-port interface. A variety of system and operational parameters were studied. This way, we developed a synthesis method that was applied to the preparation of anti-spike immunosorbent that can isolate the SARS-CoV2. Evaluation of the magnetic immunosorbent was carried out at the University of Pardubice on saliva samples from patients diagnosed with SARS-CoV2 that were analyzed by PCR method in a BSL3 lab. The automated system provides significant advantages over the manual batch procedure including a shorter processing time, higher precision, and low batch-to-batch variability.

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Lab-in-syringe automated tandem implementation of protein precipitation and homogenous liquid-liquid microextraction for the determination of beta-blockers in serum

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Beta-blockers are considered very effective agents in treating various cardiovascular diseases, including hypertension, cardioprotection after myocardial infarction, arrhythmia, angina pectoris, etc. Apart from their medical use, they might be abused in sports, requiring vehicle control and bodily movements. Therefore, the determination of beta-blockers in biological matrices is vital for therapeutic drug monitoring, doping control, and toxicological screening [1]. In this study, we developed a Lab-In-Syringe automated method for the determination of six beta-blockers in serum samples based on the tandem implementation of protein precipitation and salting-out liquid-liquid extraction. The double-step sample preparation procedure is based on precipitation of serum proteins with acetonitrile followed by phase separation with the aspiration of the salting-out agent [2]. For the first time, a 1 mL glass syringe was employed in Lab-In-Syringe to reliably handle the frequently encountered small sample volumes in bioanalysis (e.g., 100-200 µL). For this, the stirring system was re-engineered to enable homogenous void content mixing at high stirring rates (2000-3000 rpm) with the aid of a very small magnetic stirring bar, a driver ring, and an adjustable ring elevator produced by 3D printing [3]. Parameters influencing the extraction performance, including type and concentration of the salting-out agent, stirring rate, pH, and stirring time after the salt solution aspiration, were optimized by univariant studies. In addition, applicability to online coupled HPLC-UV and LC-MS was demonstrated.

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Thermoforming as a tool for fluidic systems fabrication - pros and cons

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The thermoforming technique is all about warming up of thermoplastic to the softening point and making the casting thanks to the use of a matrix of the appropriate shape (negative or positive). Such a method of producing plastic elements is widely used in many areas of life and in chemical analysis it has not gained much attention in the context of producing functional elements so far.

Mechanization of analytical chemistry procedures can be accomplished in many different ways. One useful way is to use a network of channels to direct, merge, and divide the sample and reagents streams to open up the stoichiometry of the reaction, which is the basis of the analytical determination. The methods of producing flow channels have been of interest to scientists for years, because the quality of the fluidic network determines the repeatability and reproducibility of the flow system

This communicate presents an innovative, yet simple and economically attractive approach to the fabrication of a channel network, applicable to chemical analysis where the miniaturization of the analytical system is not a top design priority.

The concept that will be presented allows for the production of channel systems of any topography, including elements such as a straight channels, reactor, loops, T-joints, Y-joints, etc.

The low cost-consuming nature of the manufacturing process by the proposed methodology makes the analytical systems disposable, e.g. due to the analysis of potentially infectious samples.

Naked eye quantification of formalin in food samples by using a microfluidic thread-based analytical device (μ TAD)

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The use of formalin (FA) to maintain food freshness and storage time is illegal. FA intake causes chronic and acute effects on human health. Therefore, this work presents the development of a novel naked-eye quantification of FA in food samples by using a microfluidic thread-based analytical device (μ TAD). This proposed method was based on a sulfite assay where FA reacted with excess Na_2SO_3 to generate NaOH that was quantified on μ TAD using acid-base titration. The μ TAD was fabricated from an untreated cotton thread (20 cm) treated with phenolphthalein solution. Next, two treated threads were coated with low and high concentrations of KHP as a primary standard solution on each thread. Both threads were tied together with a central knot before being attached to the wood box platform before the analysis. For sample preparation, the 430 μL of a sample solution containing FA was added into a microcentrifuge tube that contained 70 μL of Na_2SO_3 . The mixture was kept at room temperature for 5 minutes to ultimately generate NaOH. After loading the mixed solution at the central knot, the resulting length of the color change (colorless to pink color) on the μ TAD can be analyzed by the naked eye compared with the vernier caliper and ImageJ software, which was linear with the FA concentration. In addition, greater selectivity towards FA compared with potential interference ions was also observed. Furthermore, the developed μ TAD can be applied to analyze food samples, which shows results in agreement with those obtained by the conventional method.

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The use of 3D printing technique for the construction of miniature potentiometric cells and flow injection systems for multi-component water analysis

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Ion-selective electrodes are characterized by a sensitivity, selectivity and linearity range comparable to advanced analytical techniques. At the same time they are very cheap elements, both in construction and in operation. The use of potentiometric detection in flow analysis is still not popular. It may be caused, on one hand, by problems with the use of classic ion-selective electrodes with an internal electrolyte solution, and on the other hand, by difficulties in their implementation to work in a non-equilibrium flow mode. The construction of original, miniaturized "coated wire" or "solid contact" ion-selective electrodes and dedicated flow measurement cells, made with the stereolithographic 3D printing technique, have been presented. Such systems allow for quickly, simultaneous SEM measurement for several analytes present in the injected sample segment [1,2].

The proposed systems were tested in the analysis of natural water samples, simultaneously determining potassium, sodium, calcium and chloride. The developed analytical procedures were optimized and validated. The advantages and disadvantages of the proposed manifolds were also discussed, considering as an effective analytical tool in the monitoring of environmental waters.

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Determination of tropane alkaloids using portable CE-C⁴D system

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A majority of methods for determination of tropane alkaloids use chromatographic methods LC-MS or GC-MS [1] techniques due to their high sensitivity. However, these methods are quite expensive in terms of equipment and required chemicals. Thus, the aim of our research was to develop a simple, cost-effective, and fast method for determination of atropine and scopolamine by capillary electrophoresis using a miniaturized system with contactless conductometric detection (CE-C⁴D). Furthermore, the compatibility of the developed system with the deep eutectic solvents (DES)-based extraction procedure was tested.

During the study two methods for the determination tropane alkaloids were developed using the CE-C⁴D system. The first involved extraction with methanol : water solution (7:3, v/v) and BGE consisted of 0.5 M acetic acid (pH 2.5) with 0.25% (v/w) β -cyclodextrin [2]. While the second exploited DES-based sample extraction method (menthol : acetic acid, 1:1 molar ratio) and non-aqueous capillary electrophoresis technique (NACE) due to immiscibility of the tested DES with the water-based background electrolyte. In this case, BGE_{NACE} consisted of 7.5 mM sodium cholate, 1 mM 18-crown-6, and 15 mM acetic acid prepared in methanol. For both methods, the CE separation parameters were as follows: capillary length 29.9/18.4 cm (total/effective), the voltage of 11 kV (positive polarity) and siphoning injection (7 cm elevation, 30 s). The total separation time was less than 6 min in both cases. Both extraction methods used ultrasonic energy to facilitate and accelerate the extraction process. The developed methods were compared and discussed using RGB model [3].

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Development of human milk oligosaccharides analysis using capillary electrophoresis with mass spectrometry (CE-MS)

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Breast milk is a complete food in terms of composition, containing nutrients essential for the proper development of an infant. The benefits of breastfeeding are related to bioactive components present in breast milk, including one of the most abundant compounds – oligosaccharides. Human milk oligosaccharides (HMOs) are complex glycans which are sialylated or fucosylated. Abundance of particular HMO is influenced by several factors, such as maternal genetics, stage of lactation, mother's age and diet, and also environmental factors. HMOs perform many functions in the infant's organism, those most well documented relate to supporting the immune system [1]. Human milk is a multi-component mixture, therefore requires proper preparation and choosing the appropriate separation technique for qualitative and quantitative characterization of HMOs [2].

The research focused on the analysis of eight selected human milk oligosaccharides standards including sialylated, fucosylated and neutral ones (of which three pairs are isomers) using capillary electrophoresis coupled with mass spectrometry (CE-MS). The main aim of this research was to optimize the conditions of the CE-MS method consisted in the selection of the most effective separation buffer. In order to achieve this goal, twelve various separation buffers were tested, including formic and acetic acids (100, 200 mM) and their ammonium salts (10, 20 mM) as well as the addition of organic agents, e.g. methanol, acetonitrile (10, 20%). The best results were obtained with 10 mM ammonium formate, offering the total analysis time below 11 min. In the next stage, a procedure was developed for the preparation of a human milk sample for analysis, with special emphasis on the purifying the sample from fats and proteins to limit the matrix effect.

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Open-source arduino-based data acquisition devices

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Data acquisition (DAQ) device is an important part of any analytical instrument as there is the need of converting the measured analog signal into a digital form of binary code followed by processing by computing technology. The conversion is provided by the analog-to-digital converter (ADC), whose characteristics determine the properties (such as voltage range, resolution, data sampling frequency or noise) of the DAQ device.

There is a plenty of commercial DAQ devices available, however those might not have the needed properties or might be too expensive for the intended purpose. Therefore, in our work we focused on a construction of a simple and inexpensive DAQ device, which would be appropriate for analytical chemistry, including sensitive methods like separation techniques and which any analytical chemist would be able to build without a deeper knowledge of programming. As a result, we constructed three devices using Arduino Nano™ microcontrollers in combination with various cheap ADC modules (ADS 1115, MCP 3424 and LTC 2400) with a resolution in a bit range from 16 to 24. Schematics and corresponding source codes needed for construction were published as open-access in our recent article [1].

The best performance was obtained from the DAQ device using a 24-bit ADC module (LTC 2400). The usefulness in analytical chemistry was then demonstrated by performing an analysis of inorganic cations and anions in clinical skin wipe and tap water samples by capillary electrophoresis with contactless conductivity detection. Our developed DAQ device showed comparable performance to a commercial 24-bit DAQ device, while the purchase price of our device was only a fraction of cost of the commercial one.

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Cannabinoids (Δ^9 -THC and CBD) quantitative analysis by *in-situ* portable drug analyzer Drug Hunter

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Nowadays, marijuana is the most commonly used illegal drug. Δ -9-tetrahydrocannabinol (THC) and cannabidiol (CBD) are the primary cannabinoids found in cannabis. THC is known as the main intoxicating cannabinoid in marijuana. Meanwhile, CBD does not possess psychoactive effects and is legal to use for different disorders treatment. Nevertheless, it is necessary to separate THC and CBD to avoid false-positive test results since both can be present in the oral fluid sample after smoking marijuana. Moreover, traces of THC can be found in oral fluid after use for up to several weeks. Therefore, the accurate quantification of THC, indicating the recent marijuana use, is important for safe roadways and workplaces.

The method for quantitative analysis of cannabinoids by nonaqueous capillary electrophoresis coupled to deep UV fluorescence detection in the oral fluid (OF) was previously published by our group [1]. The current research aims to develop a more rapid, robust, and sensitive method for quantitative analysis of THC and CBD in oral fluid samples using a portable *in-situ* drug analyzer Drug Hunter. The new method is based on capillary zone electrophoresis (CE) with organic modifiers coupled to native fluorescence detection in the deep UV range. Presently, Drug Hunter is being optimized to analyze cannabinoids in the OF samples within 8 min, including sample collection and pretreatment procedures. Baseline separation of THC and CBD is achieved using two background electrolyte systems: 25 mM NaOH, 9.5 mM H₃PO₄, 60% methanol or 25 mM NaOH, 8.5 mM H₃PO₄, 40% methanol.

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The development of handheld automatic drugmeter for quantitative analysis of γ -hydroxybutyric acid

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In the recent years, capillary electrophoresis (CE) has grown interest as a perspective technique for development of low-cost, small size, portable analytical instruments for a wide range of in filed applications. One of possible usage of portable CE is detection and quantitative analysis of drugs of abuse employed in a rapid testing of a suspect person, replacing currently used immunochemistry-based test kits that suffer from difficult quantitation. One of drugs that is poorly monitored by contemporary rapid tests is γ -hydroxybutyric acid (GHB), misused as recreational as well as “date rape” drug.

A method for quantitative analysis of GHB using CE was previously described by our group [1]. The aim of this research is to develop a handheld drugmeter to quantify GHB in drinks and saliva by police officers or medical personnel. The instrument was developed with special focus on (1) simplicity of operation for a non-chemist personnel with minimal training (automation of analytical measurement and result interpretation), (2) instrument robustness for portable use, (3) low-cost construction by sourcing from off-the-shelf components and 3D printed parts.

To this date, a prototype instrument was constructed and successfully applied on analysis of GHB spiked saliva samples. Detection limit of 2 ppm GHB in saliva sample was achieved using our in-house built contactless conductivity detector. The instrument will be subject of further iterative design process with final goal to be subject of roadside test trials by local police force.

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Isolation and determination of selected hydrophobic components found in the nutmeg. Comparison of methods

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Nutmeg is a popular spice that is also occasionally used for recreational purposes by adolescents experimenting with psychoactive substances [1]. The preparation of plant sample, such as powdered nutmeg, is recognized as the challenging analytical procedure. It is complex at several stages: the analyte isolation from the sample, the purification of the extract from the remaining matrix components, and finally interfacing the sample solution with the analytical method.

The aim of this research was to compare two methods of isolation and determination of selected hydrophobic components present in nutmeg (i.e. myristicin, elemicin, methyleugenol). Nutmeg samples, purchased from various producers, were analyzed by: (i) the MME/MEEKC method, using ultrasound-assisted micellar extraction and analysis of extracts using the microemulsion electrokinetic capillary chromatography technique; and (ii) the MAE/GC-MS method, which uses microwave-assisted extraction with organic solvents and analysis of the extracts by gas chromatography coupled with mass spectrometry. In the frame of the research, validation parameters such as: linearity, repeatability, recovery, limit of detection and limit of quantification were determined for both methods. The time and effort involved in preparing samples for analysis, as well as the consumption of energy and reagents needed to carry out the entire process, were also taken into account. Furthermore, the methods were compared with the use of the RGB model, which allows one to emphasize the strengths and weaknesses of the compared methods, taking into account their analytical performance, environmental impact, and practical aspects [2]. In terms of time consumption, both methods are similar, while the use of environmentally harmful chemical reagents is significantly limited in the case of the MME/MEEKC method.

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Acknowledgments

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Application of the SPME / CE-MS method for the determination of popular drugs in the blood

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According to European drug report 2021 [1] seizures of narcotic substances in the European Union have increased steadily over the last decade. Cocaine, MDMA and mephedrone remain among most popular drugs. In addition to the increased supply of certain substances, an increase in the purity of stimulants available on the market has also been noted. There are also concerns about the more and more frequent cases of acute poisoning, e.g. cocaine or MDMA.

It is important to develop appropriate methods for the determination of these substances in biological material. Samples of this type (e.g. blood) have a complicated composition, therefore it is necessary to select the appropriate procedure for their preparation. The solid phase microextraction (SPME) method allows the extraction of analytes from small amounts of sample, which is especially important for toxicological and forensic analyzes. The great advantage of this method is also the reduction of the amount of used reagents [2].

The aim of the research was to combine the developed solid phase microextraction method [3] with capillary electrophoresis coupled with a mass spectrometer in order to determine selected drugs and metabolites in human blood. An important stage of the research was the selection of a separation buffer that will allow effective separation of analytes. Buffers based on formic acid and acetic acid with additives of acetonitrile and methanol and buffers based on ammonium formate were tested.

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Application of new methodological approaches for simultaneous determination of bisphenols in receipts using HPLC-DAD

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Bisphenols (BPs) compounds affect the level of hormones in the human body and contribute to the development of certain diseases, such as cancer. Concerning health aspects, it is essential to use appropriate methods like the H-point standard addition method (HPSAM) and integrated calibration method (ICM), facilitating the most accurate detection and determination of these compounds with the elimination of possible interferences. The study's main aim was to demonstrate how the HPSAM and ICM methods can be modified and adapted to the simultaneous determination of three bisphenols compounds (A, F, and S) in thermal papers - receipts samples from local markets. A new separation method was developed based on high-performance liquid chromatography (HPLC) with diode array detection (DAD). The time of a single analysis, enabling three analytes to be separated, was less than 10 min. The HPSAM and ICM approaches have been expanded to apply more measurement conditions (selected wavelengths based on DAD maps) than in the basic variants. Moreover, signal increment and generalized versions with linear and parabolic approximations were considered based on different experimental points. Based on estimations of analytical results obtained with various modifications of the method mentioned above, it was confirmed to improve the accuracy ($RE < 5\%$) and precision ($CV < 5\%$). The applied methods allow systematic errors due to the coelution of other substances to be compensated. The analytical efficiency of both methods was confirmed based on the analysis of receipt samples compared to the results obtained with the reference method, which was molecular fluorescence spectroscopy. Both methodological approaches can be successfully applied to the analysis of other analytical systems using various detection techniques, proven based on the RGB Additive Color Model for analytical method evaluation.

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Acknowledgments

The work was the result of the research project Freedom of research – 2021, financed by the Research Excellence Initiative of the University of Silesia in Katowice, Poland.





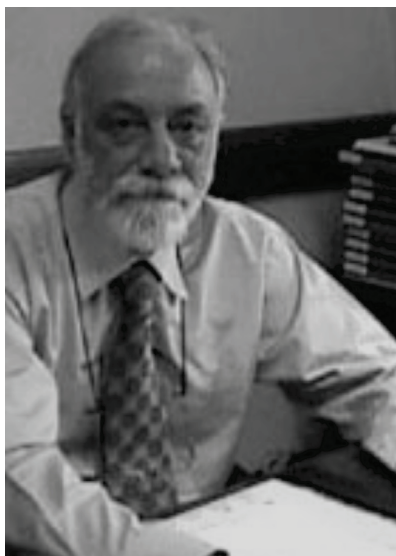
IN MEMORIAM

*Professor José Luís Fontes da Costa Lima (1945 – 2022)
organizer of conference Flow Analysis X, 2006*

*Professor Miguel Valcárcel Cases (1946 – 2022)
organizer of conference Flow Analysis VI, 1994*

outsanding experts in the field of analytical chemistry

José Luís Fontes da Costa Lima – an example to be followed



I am very sad to communicate the death of the Emeritus Professor José Luís Fontes da Costa Lima, a leading chemist who made significant contributions to the development of Analytical Chemistry in Brazil.

His parents, José Coimbra da Costa Lima and Maria de Belém Rodrigues Fontes da Costa Lima, lived in the parish of Cedofeita, Porto (Portugal), where he was born on December 5, 1945. He married Maria Assunção da Costa Lima and both were graced with two children (José Pedro and Rui) and five grandchildren.

He graduated in Chemistry at the Faculty of Sciences of the University of Porto (FF-UP), in 1970. After that, he spent 9 years at the service of Portugal in Africa. In 1985, he concluded the doctoral program in Chemistry, specialty Analytical Chemistry, being approved with distinction and praise.

At the beginning of his scientific career, Prof. Costa Lima experienced important moments when, together with colleagues from Barcelona, proposed a flow-through sandwich PVC matrix membrane electrode for flow injection analysis¹ and, in collaboration with renowned scientists from Cardiff, proposed an ion-selective electrode without an inner reference solution².

In 1986, he started working at FF-UP and decisively contributed to the formation and consolidation of a research group that today stands out in the context of analytical, bioanalytical and electroanalytical chemistry, especially in the pharmaceutical, biological, and agro-environmental areas.

In 1996, he was appointed Full Professor, and after his compulsory retirement in 2016, he became Emeritus Professor.

Regarding Luso-Brazilian scientific interactions, Prof. Costa Lima was one of the pioneers. In 1989, during an international congress on FIA-HPLC held in Córdoba he was introduced to some researchers from CENA/USP, and invited me to spend a 15-day period in Portugal, where we prepared a research project on multisite detection in flow analysis³. This was the initial seed of a fruitful scientific interaction that lasted for a quarter of a century.

This interaction resulted in important scientific contributions involving conceptual, methodological and/or applicative developments in flow analysis, including multi-site detection, multi-commutation, multi-pumping flow analysis, in-line separation/concentration exploiting fluidized beds, single-interface flow analysis, nanomaterial applications, among others⁴. In addition, other Brazilian (e.g. IQ/USP, IQ/Unicamp, Embrapa, UNESP, UFPB, UFPE, UEB), Portuguese (Catholic University of Porto) and Spanish institutions were involved. Apart from the excellent research results, the outstanding number of scholarships, scientific visits and student participations in a different scientific environment was a positive factor towards the improvement of the formation of students and technicians.

In Brazil, Prof. Costa Lima actively participated in the organization of the VII International Conference on Flow Analysis (1997), and collaborated in several Annual Meetings of the Brazilian Chemical Society (RA-SBQ) and National Meetings on Analytical Chemistry (ENQA). In 2000, he had a prominent role in the XXIII RA-SBQ, as the theme of the meeting was “The Brazil-Portugal chemical bond”. In 2003, he attended the XII ENQA giving the brilliant conference “Development of flow analysis in the context of the Luso-Brazilian interaction”⁵.

He encouraged more than 30 Brazilian collaborators to undertake medium-term internships at the University of Porto, and to participate in scientific events in Portugal, especially the Luso-Galician Congresses, the Euroanalysis XI (Lisbon, 2000), and the X International Conference on Flow Analysis

(Porto, 2006). On the other hand, more than 20 Portuguese researchers carried out scientific missions in Brazil. This resulted in 89 published articles with the participation of both Portuguese and Brazilian researchers.

In 2005, he was sworn in as a member of the Brazilian Academy of Sciences.

The relevance of the scientific work of Prof. Costa Lima can be assessed in terms of its publications (Web of Science: 454 scientific articles, 9293 citations, $h = 52$), communications at scientific events and training of human resources, especially graduate students, which he affectionately called “scientific children”⁵.

He also dedicated himself to academic-scientific management activities, participating in the organization of several congresses, workshops and similar. During the last years as an active scientist, he was the Director of the FF-UP, carrying out an outstanding work.

In memoriam

A big party took place on December 5, 2015 to celebrate his retirement. I had the pleasure of being present, offering him an award from the University of S. Paulo, which recognized his efforts in favor of Brazil.

As an Emeritus Professor, he continued to work at FF-UP, giving international conferences, strengthening the scientific relations with Brazil, etc.

Throughout his career, he was member of editorial boards of several journals (e.g. *Microchim. Acta*, *Farmaco Sci.*, *Am. J. Enol. Vitic.*, *Port. Electrochimica Acta* and *Rev. Port. Quim.*); member of organizing committees in 26 international meetings; member of 06 scientific societies; coordinator of the European M.Sc. program in Environmental Analytical Chemistry; integrant of the Chemical Center of the University of Porto - REQUIMTE; collaborator with several research foundations; etc. He was worldwide recognized, as demonstrated by the important awards.

Some personal characteristics of Prof. Costa Lima, such as leadership, creativity, spirit of investigation, tenacity, ease of integration into a teamwork and, above all, friendship and companionship should be highlighted. As for friendship, I received from Prof. Costa Lima an extraordinary example: when serious health problems affected me, he surprised me: I received a visit from this friend who crossed the Atlantic Ocean to comfort me.

He did several society volunteer activities, promoting social aid, participating in charity parties, etc. An interesting activity is that, when young, he was the table tennis coach of the Portuguese team.

He was always ready to host visiting friends. I was often at his home, and confirmed that he was an exemplary host, and also an excellent father, grandfather, husband and son-in-law. There, I could see that important moral values are passed from generation to generation in his family.

Costa Lima had a special dedication to Brazil and his collaborators lost a very good friend and example to be followed. I am sure that he will be very happy in this new dimension of his life, undoubtedly full of Peace, Joy and Happiness.

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Elias Ayres Guidetti Zagatto

The logo for the Brazilian Journal of Analytical Chemistry (BrJAC) is displayed in a large, bold, black-outlined font. The letters 'BrJAC' are stylized, with the 'B' and 'r' connected, and the 'J' and 'A' also connected.

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Miguel Valcárcel Cases



Who has not seen or heard about books such as Analytical Chemistry: A Modern Approach to Analytical Science, Principles of Analytical Chemistry: A Textbook, or Foundations of Analytical Chemistry: A Teaching-Learning

Approach, or about flow injection analysis-FIA, and sequential injection analysis-SIA? These are, in fact, some contributions from Prof. Miguel Valcárcel Cases, at the University of Córdoba-Spain, who leave us on 9th January 2022 at the age of 75.

Prof. Valcárcel was Dean of the Faculty of Sciences at the University of Córdoba, Vice-Rector for academic guidance and teaching and Vice-Rector for quality, as well as the first Director of the Andalusian Institute of Fine Chemistry and Nanochemistry since 1994.

Born in Barcelona (Spain), Prof. Valcárcel was a graduate of the University of Seville where also obtained his Ph.D., and was an assistant teacher until 1975. He was an associate professor of Analytical Chemistry at the Faculty of Science of Palma de Mallorca in 1975, an institution where he was also Dean and full professor at the University of Cordoba in the year 1976.

He was also President of the Analytical Division of the European Federation of Chemical Societies and was a member for 4 years of the High-Level Expert Group of the European Union's Growth Program.

Valcárcel received the Spanish national Enrique Moles prize for Chemical Science and Technology (2005), the Maimónides prize for scientific-technical research from the Junta de Andalucía (1992), and the Solvay Research Prize in Chemical Sciences from the CEOE Foundation (1997). He has the Robert Boyle Medal from the Royal Society of Chemistry (UK, 2004), the Enrich Planquette Prize from the Austrian Chemical Society (1996), the Gold Medal from the University of Warsaw (2000), and the Medal from the Portuguese Chemical Society (2000). He also received the distinction of Cordoba citizen of the year 2006 in the education/research section, and the Averroes

de Oro-Ciudad de Córdoba medal in 2006 for his scientific trajectory. He was also awarded the title of Doctor Honoris Causa by the University of Valencia (2010) and the European DAC-EuChMS (Division of Analytical Chemistry of the European Association for Chemical and Molecular Sciences) award in recognition of his scientific and teaching career (2015).

He was the author of ca. 700 papers, published 9 scientific books, and co-authored 15 chapters of multi-author books.

Owner of a unique vitality and a very accurate vision of Analytical Chemistry, Prof. Valcárcel contributed to the formation of dozens of students, of which he was extremely proud, and some of them are today Full professors spread all over the world.

The Brazilian Journal of Analytical Chemistry mourns his death, and through this simple tribute, recognizes his great contribution to Analytical Chemistry and science around the world.

Marco Aurélio Zezzi Arruda



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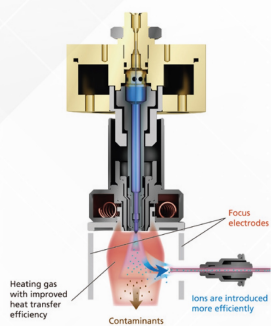


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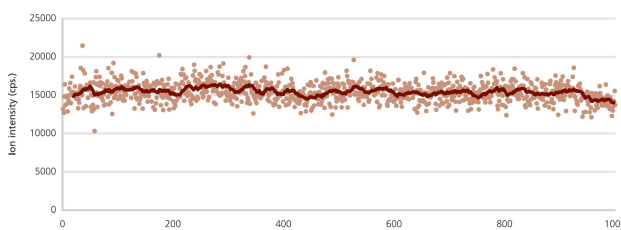
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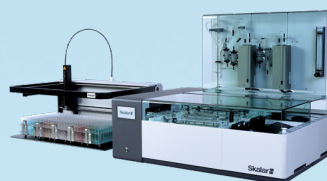
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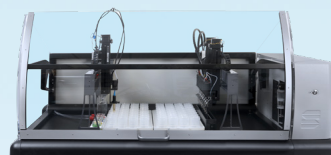
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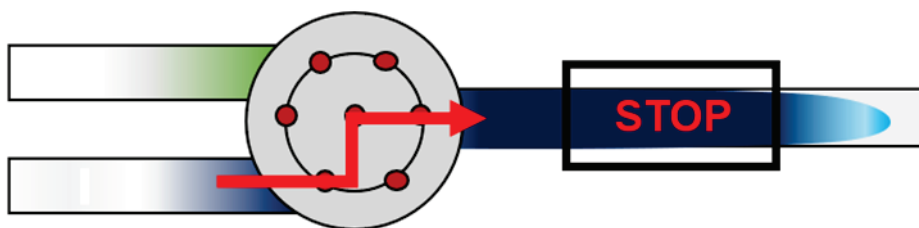


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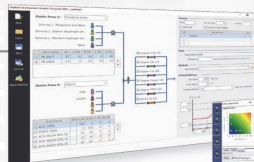
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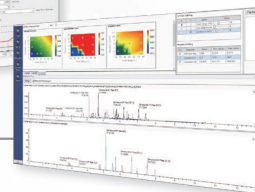


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