# Hightech-based Micro/Nanostructured Sensors Devices and Microreactors in Real-time for Automated Process Analytical Multianalyte Platform Systems

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**PN-II-ID-PCE-2011-3-0538**

**Project Leader (PL): Prof. Dr. Jacobus (Koos) Frederick van Staden**

**PL's Project Laboratory: PATLAB, Bucharest of INCDEMC, Timisoara**

**PL's Host Institution for the project: National Institute of Research and Development for Electrochemistry and Condensed Matter (INCDEMC), Timisoara**

**Project duration in months: 60 months (2012-2016)**

**Project budget: 1.500.000RON**

## Project Summary

**The main purpose of the project is to develop efficient low cost dedicated automated multianalyte Platform (MP) systems with the integration of innovative research and technology of well designed characterised high-tech-based micro- and nanostructured sensors probes devices and very well functional catalytic micro reactors operational unit devices into a unit (flow-based or non-flow-based) that can easily be adopted and applied in real-time automated micro and nanoprocess analytical technology (PAT) systems as an industrialised end-product. A problem related analytical approach, considering complexity of every unique sample matrix with corresponding conditions, will be followed. The overall two phase strategic scenarios will include identification and efficient**

quantification of analytes that damage sustainable and healthy human life (groups of analyte parameters for example pesticides, toxic metals, organic and inorganic substances in food, beverages, water, soil.) and those that enhance excellent sustainable and healthy human life (vitamins, mineral, antioxidants) in food, pharmaceutical products. The main objectives are further to select and investigate the most probable suitable types of sensors devices and reactor conditions; in-depth characterize and optimize the sensor for individual and efficient group multianalyte detection; validate prototypes on real samples; implement the systems as MP for PAT Micro/Nano-systems. Analytes or analytes groupings will be tailored.

## Team Members

| Name | Role in the project |
|---|---|
| Jacobus Frederick van Staden, DSc, Prof., CSI | Director of Project |
| Raluca-Ioana van Staden, Dr habil., CSI | Principal Researcher |
| Ramona Georgescu, MSc | PhD student |
| Iuliana Moldoveanu, MSc | PhD student |
| Livia Alexandra Gugoasa, MSc | PhD student |
| Ionela Raluca Comnea, MSc | PhD student |
| Anita Elena Girbea, MSc | Young researcher |
| Radu Constantin Iacomin, MSc | Young researcher |
| Bogdan Calenic, PhD | Postdoc |
| Oana Elena Stoica, MSc | Technician |
| Sorina Oprina | Technician |
| Luciana Gherghina | Technician |

## Objectives

The main objective of the project is to develop efficient low cost dedicated automated multianalyte platforms with the integration of innovative research and technology of well designed characterised nanotech-based multi-parameter sensors probes (in micro and nano mode) and very well functional catalytic micro reactors operational unit devices into a unit (flow-based or non-flow-based) that can easily be adopted and applied in micro and nanoprocess analytical technology

**systems as an industrialised end-product. A problem related analytical approach, taking the complexity of every unique sample matrix with corresponding conditions into consideration, will be followed. The overall two phase strategic scenarios will include 1) identification and efficient quantification of analytes (processes) that damage sustainable and healthy human and animal life (groups of analyte parameters for example pesticides, toxic metals, organic and inorganic substances in food, beverages, water, soil, etc.) and 2) identification and quantification of analytes (processes) that enhance excellent sustainable and healthy human life (e.g. vitamins, mineral, antioxidants) in food, pharmaceutical products etc. Analytes or analytes groupings will be tailored to a selected specific dedicated number most needed but within the ability and feasibility of the project.**

**Specific objectives are:**

1. **Data retrieval and in-depth investigation, and analyte selection according to importance and urgent requirements.**
2. **In-depth strategic planning of the whole project from design to implementation.**
3. **Development with extensive characterization of electrochemical sensors including SECM.**
4. **Development with extensive characterization of optical devices for UV/Vis/fluorescence/chemiluminescence (with quantum-dots)/NIR/Raman/AFM.**
5. **Development with in-depth investigation of catalytic micro-/nanoreactors/nanostructured and nanoporous membranes unit devices.**
6. **Integration into micro- and nanoprocess analytical technology systems as key industrialised end-products.**

**Methodology**

**The following intermediate milestones (MS) are proposed:**

**MS1. Exploration and selection criteria (Month 6).**

**MS2. Strategic planning (Month 8).**

**MS3. Electrochemical sensors (Month 18).**

**MS4. Optical devices (Month 18).**

**MS5. Reactors (Month 26).**

**MS6  Integration (Month 36).**

**MS7. Management. (Throughout)**

**MS8. Dissemination (Months 24 – 36).**

**The scientific work and technological development are structured distributed into EIGHT(8) core workpackages (WP's):**

***WP1*** **- Coupled to intermediate milestone 1 of the project will be dedicated to explore various innovative pathways in terms of selection of most needed** ***groups of analytes*** **and proper sensor materials and detection devices for each of the** ***nanotech-based multi-parameter sensors devices*** **- electrochemical sensor probes and optical microscopic detection probes {UV/Vis, fluorescence and chemiluminescence (with quantum-dots), NIR, Raman, AFM} and (if needed) micro- and nanoreactors. WP1 is of paramount importance to identify potential breakthroughs that will be further investigated in the next WP's.**

***WP2*** **- Electrochemical sensors.**

***WP3*** **- Optical devices**

***WP4*** **- Micro-/nanoreactors/membranes (Coupled to intermediate milestones 2, 3, 4 and 5) will develop with extensive characterization**

**and in-depth investigation each of these identities simultaneously in parallel as separate identities.**

***WP5*** **- Coupled to intermediate milestone 6 will integrate the individual separate identities from WP2, WP3 and WP4 into micro- and nanoprocess analytical technology systems as key industrialised end-products.**

**WP6 - will be dedicated to the integration of the individual systems into a new integrated low cost, dedicated multianalyte platform unit. The idea is to develop a number of these multianalyte platform units for employment in different industrial process situations.**

***WP7*** **- will be used as management and risk assessment of the project**

***WP8*** **will form the dissemination and exploitation of results.**

**The workpackages (WP's) are divided into the following tasks (activities):**

**WP1:**

**1.1 Identification and information of most needed urgent analytes or groups of analytes.**

**1.2 Data retrieval of current known analytical methodology on selected analytes or groups of analytes.**

**1.3 In-depth investigation into new innovative pathways with careful strategic planning of proper new analytical methodology with proper sensor materials and detection devices for each of the *nanotech-based multi-parameter sensors devices* - electrochemical sensor probes and optical microscopic detection probes.**

**1.4 Identification of potential possible breakthroughs that will be further investigated in the next WP's.**

**WP2:**

**2.1 Identification and selection of proper suitable sensor material, and design of suitable sensor membrane/surface, sensor transducer, signal transport, amplification, processing for electrochemical sensors/SECM.**

**2.2 Construction and integration of different parts of the sensor probe into a suitable nano-structured sensing probe.**

**2.3 In-depth investigation and characterization of the functionality of the physical and chemical parameters of the sensing probe.**

**2.4 Optimization with constant in-depth evaluation of the electrochemical sensor in terms of concentration range, selectivity, response characteristics and detection limit for single, multi-parameter and grouping of analytes.**

**2.5 Validation of real-time electrochemical sensor device for the selected analyte material and selected application fields of food, beverages, water, soil, environmental, pharmaceutical and diagnostics.**

**<u>WP3:</u>**

**3.1 Identification and selection of proper suitable single and/or multiple optical microscopic detection probes units (UV/Vis, fluorescence and chemiluminescence (with quantum-dots), NIR, Raman, AFM).**

**3.2 Identification and selection of proper energy sources and filtering units for each optical device as needed.**

**3.3 Development with in-depth investigation and characterization of each individual type of optical device with careful strategic planning regarding the suitability as optical microscopic detection device.**

**3.4 Identification and selection of possible breakthroughs devices that is suitable as proper functional devices for further investigation for determination of single, multi-parameter and/or grouping of analytes as needed.**

**3.5. In-depth investigation of electromagnetic spectrums (UV/Vis, NIR, Raman) for suitable proper spectrochemical methods for qualitative identification (finger printing/pattern recognition capability) and possible quantitation for determination of single, multi-parameter and/or grouping of analytes as needed.**

**3.6 Identification and selection of individual or multiple wavelengths properties for proper suitable sensitive responses towards single, multi-parameter and/or grouping of analytes as needed.**

**3.7 Enhancement of response sensitivity and selectivity with extensive characterization using glowing tags like fluorescence, chemiluminescence, quantum-dots and non-glowing tags like SERS where needed.**

**3.8 Optimization with constant in-depth evaluation of the selected individual optical devices in terms of concentration range, selectivity, response characteristics and detection limit for single, multi-parameter and grouping of analytes.**

**3.9 Validation of real-time selected individual optical devices for the identification and quantification from real samples from selected application fields of food, beverages, water, soil, environmental, pharmaceutical and diagnostics.**

**WP4:**

**4.1 Identification and selection of proper suitable micro- and nanoreactors/membranes for separation/filtration/reaction unit operations as needed for sampling preparation and processing.**

**4.2 Development and optimization with in-depth investigation and characterization of physical and chemical parameters of catalytic micro-/nanoreactors/nanostructured and nanoporous membranes unit devices in combination with selected individual nano-based sensors devices for optimal enhanced performance of single, multi-parameter and/or grouping of analytes as needed.**

**4.3 Validation of suitable proper reactors for quantification from real samples from selected application fields of food, beverages, water, soil, environmental, pharmaceutical and diagnostics.**

**WP5:**

**5.1 Integration of individual separate identities from WP2, WP3 and WP4 into micro- and nanoprocess analytical technology systems as key industrialised end-products.**

**5.2 Validation of micro- and nanoprocess analytical technology systems.**

**WP6:**

**6.1 Integration of individual systems in WP5 into a new integrated low cost, dedicated multianalyte platform unit.**

**6.2. Validation of this unit.**

**WP7:**

**7.1 Continuous progress evaluation and assessment of the project.**

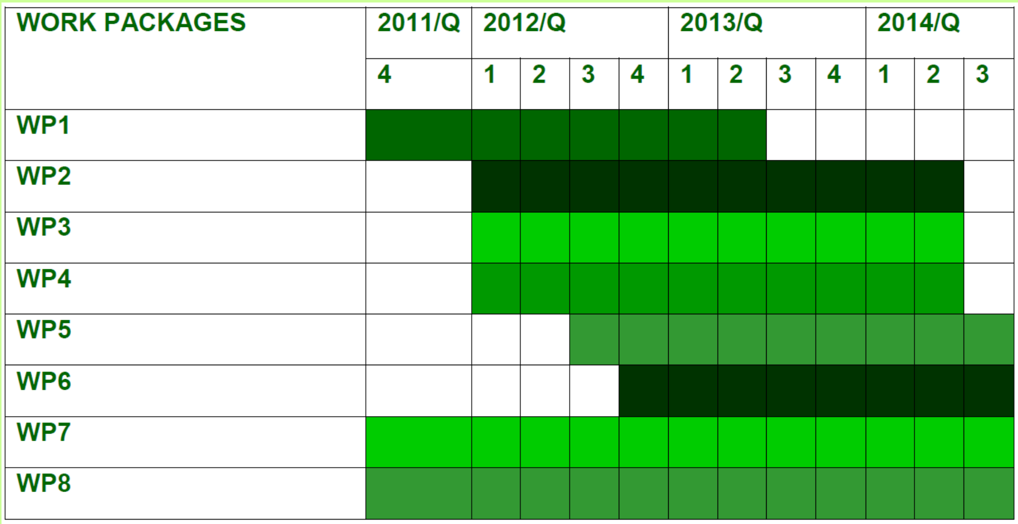
**7.2 Continuous proper coordination and project management.**

**WP8:**

**8.1 Dissemination with a website, flyers and of results through patents, published papers in ISI peer-reviewed journals, presentations at workshops, conferences, seminars.**

**8.2 Integration of knowledge with training/education of students/young researchers.**

**The Gantt chart of WP's of showing the description of the main Work Packages with the timescales associated with each WP.**




## Dissemination

## Papers published:

1. **Flow-injection analysis systems with different detection devices and other related techniques for the in vivo and in vitro determination of dopamine as neurotransmitter. A review.**
   **J.F. van Staden and R.I. Stefan-van Staden.**
   **Talanta, 102 (2012) 34-43. [http://dx.doi.org/10.1016/j.talanta.2012.05.017](http://dx.doi.org/10.1016/j.talanta.2012.05.017)**

2. **Influence of the physical immobilization of dsDNA on the carbon based matrices of electrochemical sensors**
   **LA Gugoasa, RI Stefan-van Staden, AA Ciucu, JF van Staden**
   **Current Pharmaceutical Analysis, 10(1), 20-29, 2014.**

3. **Graphene based dot microsensors for the assay of adenine, guanine and epinephrine**
   **JF van Staden, R Georgescu, RI Stefan-van Staden, I Calinescu**
   **J Electrochem Soc, 161(2), B3014-B3022, 2014.**

4. **Evaluation of amperometric dot microsensors for the analysis of serotonin in urine samples**
   **JF van Staden, R Georgescu, RI Stefan-van Staden, I Calinescu**
   **J Electrochem Soc., 161(4), B49-B54, 2014.**

5. **Detection of folic acid from orange juice using amperometric dot microsensors based on graphite and graphene**
   **R. Georgescu, J.F. van Staden, R.I. Stefan-van Staden, C. Boscornea**
   **Rev Roum Chim, 60(5-6), 461-466, 2015**

6. **Comparison of fluorescein and potassium permanganate in fast low cost screening methods for determination of folic acid in pharmaceutical tablets**
   **R. Georgescu, J.F. van Staden**
   **Rev Roum Chim, 60(5-6), 563-569, 2015**

7. **Analytical continuous flow systems. Where two worlds collide! From gravimetry and test tubes to flow sytems to FIA to SIA to PAT and from Orsat to control room to PAT to TAP**
   **J.F. van Staden**
   **Rev Roum Chim, 60(5-6), 403-414, 2015**

8. **Application of phthalocyanines in flow-and sequential-injection analysis and microfluidics systems: A review**
   **J. F. van Staden**
   **Talanta, 139, 75-78, 2015**

**Papers submitted to ISI journals:**

1. **Single-walled carbon nanotubes for enantioanalysis of malic acid in wines**
   **RI Stefan-van Staden, IR Comnea, JF van Staden, I Moldoveanu**
   **Submitted to ISI journal**

2. **Screening test for cancer biomarkers using FT-IR**
   **RI Stefan-van Staden, I. Moldoveanu, JF van Staden**
   **Submitted to ISI journal**

## Papers presented at conferences:

1. Amperometric Dot-Sensors based on Zinc Porphyrins for the Determination of Sildenafil Citrate
   Jacobus Frederick van Staden, Raluca-Ioana Stefan-van Staden
   IMCS 2012 The 14th International Meeting on Chemical Sensors, May 20 – 23, 2012 Nuremberg, Germany (Oral presentation)

2. Nanotechnology and Multianalyte Platform Flow Systems
   Jacobus Frederick van Staden, Raluca-Ioana Stefan-van Staden
   4th EuCheMS Chemistry Congress (EuCheMS 2012) 26-30 August 2012, Prague, Czech Republic (Oral presentation)

3. Simultaneous neurotransmitters analysis using microelectrodes based on porphyrins
   Raluca-Ioana Stefan-van Staden, iuliana Moldoveanu, Jacobus Frederick van Staden
   4th EuCheMS Chemistry Congress (EuCheMS 2012) 26-30 August 2012, Prague, Czech Republic (Oral presentation)

4. Nanotechnology in the Flow Domain of Process Analysis
   Jacobus Frederick van Staden
   1st International Conference on Analytical Chemistry. Analytical Chemistry for a Better Life, Targoviste, Romania, September 18-21, 2012 (Invited Key note)

5. New amperometric microsensors for the analysis of serotonin in urine samples
   Jacobus Frederick van Staden, Ramona Georgescu, Raluca-Ioana Stefan-van Staden
   223rd ECS Meeting, May 12-16, 2013, Toronto, Canada (Oral presentation)

6. Combined DOT sensors for food, pharmaceutical and biological analysis
   Jacobus Frederick van Staden, Raluca-Ioana van Staden
   245th ACS Meeting, April 7-11, 2013, New Orleans, USA (Oral presentation)

7. Influence of the physical immobilization of dsDNA on the carbon based matrices of electrochemical sensors
   LA Gugoasa, RI Stefan-van Staden, AA Ciucu, JF van Staden
   1st International Conference on Analytical Chemistry. Analytical Chemistry for a Better Life, Targoviste, Romania, September 18-21, 2012. (poster)

**8. New amperometric microsensors for the analysis of serotonin in urine samples**
**Ramona Georgescu, Jacobus Frederick van Staden, Iuliana Moldoveanu, Ioan Calinescu, Raluca-Ioana Stefan-van Staden**
**1st International Conference on Analytical Chemistry. Analytical Chemistry for a Better Life, Targoviste, Romania, September 18-21, 2012. (poster)**

**9. Graphene based sensors for the assay of adenine, guanine and epinephrine**
**Ramona Georgescu, Jacobus Frederick van Staden, Raluca-Ioana Stefan-van Staden**
**1st International Conference on Analytical Chemistry. Analytical Chemistry for a Better Life, Targoviste, Romania, September 18-21, 2012. (poster)**

**10. Novel smart food production and packaging systems (invited lecture)**
**JF van Staden**
**SUSChem, Brussels, October 6, 2015**

**11. Improved advanced materials and intelligent control systems for a wide set of applications (invited lecture)**
**JF van Staden**
**SUSChem, Brussels, October 6, 2015**