REVIEWER REPORT

on the Dissertation Thesis of

Mgr. Kateřina Fikarová

The dissertation thesis (DT) entitled ***"Development of novel approaches to automated sample preparation for pharmaceutical and environmental analysis"*** submitted by the PhD candidate Mgr. Kateřina Fikarová was originated at the Faculty of Pharmacy, Charles University, in cooperation with the University of Balearic Islands and the University of Alicante. The DT reports recent developments in the field of automation of environmental and pharmaceutical sample processing techniques exploiting most advanced flow methodologies. Six novel methods in SIA format allowing automation of sample pre-treatment were devised. The techniques employ smart lab-in-syringe configurations facilitating coupling of efficient, mostly micro-extraction procedures, with modern instrumental methods, such as ICP-AAS or liquid chromatography. The determined analytes covered by the proposed flow methods, such as traces of heavy metals, mononitrophenols and alkyl phthalates leached from microplastics are of real environmental interest. Hence the topic of the DT is up-to-date, outlining a number of original ideas, thus demonstrating undoubtedly the potential of the candidate for pursuing independent and meaningful scientific research.

The DT designed as comments to six candidate´s publications involves 92 pages of text. As for the formal aspect of the DT, it is properly structured and presented in an understandable way with appropriate documentation by 30 diagrams and 2 tables. The Theoretical part is detailed, sometimes not quite easy to follow, because the candidate tends to present her ideas in the form of very long sentences, often extending to 4 lines of printed text. On the other hand, the proposed objectives of the DT are clearly defined; 185 references quoted show the current state-of-the-art in the field of study, i.e., flow-based approaches in the automation of analytical procedures. Here the candidate demonstrates awareness and understanding of the intended area of research and has shown deep knowledge in this branch of analytical science. The essential part of the DT entitled Supplementary material comprises six full-text reprints of papers based on original research and published after rigorous peer-reviewing in the first-rate analytical journals. The chapter Conclusions summarizes appropriately the most important outcomes of the DT. Additionally, the candidate has also presented her research in the form of 15 oral contributions or posters at several domestic or international conferences devoted to analytical chemistry. Finally, the candidate´s notable international experience must be highlighted.

Considering the significance of the results, high scientific quality, and novelty of the research encompassing international dimension, I do not hesitate to commend Mgr. Kateřina Fikarová to be awarded the PhD degree, subject to successful defence of her DT.

Comments and Queries

p. XI, Index of abbreviations: *APDC – ammonium dithiocarbamate* does not seem to be a correct explanation of this abbreviation. Moreover, the use of *FT* for flow techniques was not a good idea since FT is extensively used for Fourier transform in analytical publications.

p. 8, line 7 from the top: *" In the earlier FT, the sample was injected via injection septum by a rotary injection valve using continuously operating peristaltic pumps."* Is it true?

p. 9, line 4 from the top: *"Due to the high reproducibility of timing between injection and detection, the peak height can be used for quantification in the FT, which is in contrast to HPLC or capillary electrophoresis where the analyte migration or retention can slightly vary between samples."* Can you explain how a slight variation of retention or migration in HPLC or CE can influence the repeatability of the peak heights. Is there a big difference in the peak height repeatability when comparing FIA/SIA with HPLC/CE?

p. 13, line 14 from the bottom: "*In the pharmaceutical analysis, SIA is used mostly for quality control of drug formulations …*" Is there any pharmacopoeial method based on SIA?

p. 24, Table 1: Are really the LODs in ICP-MS and in GFAAS at *ppt* (parts per thousand) level?

p. 28, line 2 from the bottom: *"...the fibre is typically made of fused-silica coated with an organic polymer (e.g.,... PMDS), or metallic wire and is immersed ...*" Isn´t there something missing in the text?

p. 38 line 6 from the bottom: "*…by the addition of kosmotropic compounds…*" Is "kosmotropic" appropriate technical term?

p.67, line 1 from the bottom: "*Figure 28 illustrates the clean-up …*". The figure number does not match the figure description.

Please comment on the possibility/impossibility of the use of ultrasound radiation instead of magnetic stirring in the LIS technique.

Doc. RNDr. Miroslav Polášek, CSc. Hradec Králové, 21st August 2020