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Jury Member Report – Doctor of Philosophy thesis.

Name of Candidate: Anastasiia Merdalimova

PhD Program: Physics

Title of Thesis: Optical Sensors Based on Hollow-Core Microstructured Optical Waveguides: 2-in-1 multispectral refractometry and raman spectroscopy

Supervisor: Professor Dmitry Gorin
Co-supervisor: Associate Professor Alexey Yashchenok

Name of the Reviewer: Andrei Sapelkin

I confirm the absence of any conflict of interest	**No conflict of interest**
(Alternatively, Reviewer can formulate a possible conflict)	**Date: 19/11/2023**

The purpose of this report is to obtain an independent review from the members of PhD defense Jury before the thesis defense. The members of PhD defense Jury are asked to submit signed copy of the report at least 30 days prior the thesis defense. The Reviewers are asked to bring a copy of the completed report to the thesis defense and to discuss the contents of each report with each other before the thesis defense.

If the reviewers have any queries about the thesis which they wish to raise in advance, please contact the Chair of the Jury.

Reviewer's Report

Reviewers report should contain the following items:

- Brief evaluation of the thesis quality and overall structure of the dissertation.
- The relevance of the topic of dissertation work to its actual content
- The relevance of the methods used in the dissertation
- The scientific significance of the results obtained and their compliance with the international level and current state of the art
- The relevance of the obtained results to applications (if applicable)
- The quality of publications

The summary of issues to be addressed before/during the thesis defense

The overall thesis structure and quality are appropriate for the PhD level examination, while the content is undoubtedly scientifically significant at international level, delivering state of the art methods in multispectral characterization of liquid analytes. The project certainly makes a sizable and original contribution to the field of development of optical sensors based on the hollow core fiber technology. The quality of the work carried out by the candidate is further supported by the number of peer-review publications provided by the author. The topic of the dissertation is commensurate with the content, while methodological approach is appropriate for the project objectives and challenges that have been addressed within this work.

At the same time, while overall dissertation is generally of good quality, it is my view that there is lack of details in places that needs to be addressed before the viva. I outline my recommendations below.

Abstract must contain a brief description of motivation for this work/problem statement, key methods/approaches used and summary of the outcomes. An Abstract is, essentially, a short version of the Conclusions (that is also currently far too generic – see below). In particular, while the need for early diagnostics is justifiable, it is not clear from the abstract why HC-MOW were selected as a platform for early diagnostics. My recommendation would be to explain in the abstract that a way towards early diagnostics proposed in this project is to measure certain (what?) parameters of extracellular vesicles at an early stage. Then explain why and how HC-MOW will enable such measurements. When stating comparison/referencing of HC-MOW against flat surfaces, specific information about the conditions is required (e.g. what was the droplet size on the flat surface and how does it compare to the volume in the waveguide, etc.). As an example, the current sentence "Initially, it was shown that compared to measuring a larger volume drop on a planar substrate, the use of HC-MOW allows to increase the intensity of the observed Raman scattering with a lower probe volume used, and different analytes provide different intensity improvement (fiber enhancement)." Can be changed to something more specific such as "First, we demonstrate that HC-MOW show markedly improved performance in Raman signal measurements when compared to equivalent volumes (?? ml) of analyte on a flat surface, resulting in amplitude enhancement in ??-?? range while maintaining S/N ratio (or improving it by a factor of ??)." This same approach whereby specific findings are stated must be extended to the rest of the abstract. Thus, if "significant enhancement" is mentioned, there must be quantitative evidence of that. If the Raman and refractometry improve early detection, there must be quantitative evidence by how much.

Chapter 3 is lacking description of the principles of the Raman effect, schematic of the system used for Raman measurements and principles of SERS: these are all essential parts of the methodology and must be included. I understand that manufacturing of the HC-MOW was not part of the project, hence I expect the detailed description (schematic, SEM, etc.) of the fibers and their origin used in this work to appear in the Methods section – this is currently lacking and must be included. There are also no details of how various solution were fed into the fiber (e.g. was it a pump, capillary effects, etc.) and no picture of the actual arrangement in the Raman set up. This information is essential for the Methods section and must be included. This is also something that I'll be exploring during the viva.

The summary of the Introduction is starts with "On the other hand…" that is not appropriate – it must start with something like "So far we demonstrated …" and then provide the summary of the review findings, the problem (problems) statement and a brief description of the approach proposed to address the problem (problems, issues). Some of that is present, but not explicitly so this is something that I would like to explore in some details during the defence.

A rather general problem across all chapters that summary sections are rather generic and not always contain the quantitively description of the findings. This should be addressed, and I will explore this further during the defence.

I found that conclusion is largely supported by the data and the corresponding discussions, but the candidate need to ensure consistency between the Abstract and Conclusions, both in terms of the style and the information: Abstract is, in essence, is a shortened version of the Conclusion.

Finally, there are several formatting issues that need to be fixed. These include starting chapters from a new page, making sure there are no empty spaces of (sometimes) half a page size (eg, see page 27, 102, etc.). Author must ensure that all figures are perfectly legible – some of them are not (e.g. see Fig 7-6). Captions must provide sufficient information to understand the content of a figure without reference to the text – some captions are far too abbreviated (e.g. see Fig 7-9). These must be provided. t has too short a caption: compare to e.g. Fig 6-7 that has a comprehensive caption). Some captions appear broken across pages from the figure (e.g. Fig 2-8) - this is not acceptable and needs to be corrected (e.g by reducing figure size). Spacing between captions and the main text must be appropriately adjusted (e.g it is far too small in Fig 2-8 caption). Line spacing seems to be varied across the document (see eg. start of Chapter 4 in page 62). English language needs to be addressed in few places as some sentences do not make sense (e.g. last sentence in caption to Fig 4-1 doesn't contain a verb) – please read through carefully or use a proof-reader service.

Provisional Recommendation

☐ *I recommend that the candidate should defend the thesis by means of a formal thesis defense*

☒ *I recommend that the candidate should defend the thesis by means of a formal thesis defense only after appropriate changes would be introduced in candidate's thesis according to the recommendations of the present report*

☐ *The thesis is not acceptable and I recommend that the candidate be exempt from the formal thesis defense*