REVIEW
of the official reviewer for dissertation work
Bektassov Marat Aidaruly on the theme «Development of comprehensive methods for organic compounds determination in water» presented for the degree of Doctor of Philosophy (PhD) in the specialty «8D05301 – Chemistry».

№	Criteria	Eligibility (one of the options must be checked)	Justification of the position of the official reviewer
1.	The topic of the thesis (as of the date of its approval) corresponds to the directions of development of science and/or state programs	1.1 Compliance with priority areas of science development or government programs:	
		1) The thesis was completed within the framework of a project or target program financed from the state budget (indicate the name and number of the project or program) 2) The thesis was completed within the framework of another state program (indicate the name of the program) 3) The dissertation corresponds to the priority direction of the development of science, approved by the Higher Scientific and Technical Commission under the Government of the Republic of Kazakhstan (indicate the direction)	This thesis was conducted within the framework of projects funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan: Grant project "Zhas Galym" for the scientific research of young researchers for 2025–2027: "Development of Methods for Quantitative Analysis of Biogenic and Anthropogenic Pollutants in the Environment and Identification of Their Sources", Project number AP25796583.
2.	Importance for science	The work **makes**/does not make a significant contribution to science, and its importance is well disclosed/not disclosed	Given the critical role of biogenic volatile organic compounds (BVOCs) in atmospheric chemistry and climate regulation, along with the persistent analytical challenges associated with their accurate quantification, there is an urgent need to develop robust and sensitive methods for BVOCs determination. This work is grounded in both fundamental scientific demands and practical challenges related to monitoring key atmospheric and marine constituents within the field of

			environmental analytical chemistry. The thesis contributes to the advancement of analytical methodologies for the determination of BVOCs in aqueous samples.
3.	The principle of independence	Self-reliance level: 1) **High**; 2) Medium; 3) Low; 4) No independence	The self-reliance level the author is evidenced by the development and validation of the analytical methodology, the identification and conceptualization of gaps in the existing literature, the design and execution of the experimental research, and the comprehensive management, processing, and interpretation of the generated data.
4.	The principle of inner unity	4.1 Justification of the relevance of the thesis: 1) **Justified;** 2) Partially justified; 3) Not justified.	The relevance of the thesis is justified through the development of a comprehensive and sustainable analytical framework, highlighting the scientific significance and timeliness of the research.
		4.2 The content of the thesis reflects the topic of the thesis: 1) **Reflects;** 2) Partially reflects; 3) Does not reflect	The content of the thesis reflects the topic of the thesis.
		4.3. The purpose and objectives correspond to the topic of the thesis: 1) **correspond;** 2) partially correspond; 3) do not correspond	The purpose and objectives correspond to the topic of the thesis.
		4.4 All sections and provisions of the thesis are logically interconnected: 1) **completely interconnected;** 2) the interconnection is partial; 3) there is no interconnection	All sections and provisions of the thesis are completely interconnected.

		4.5 The new solutions (principles, methods) proposed by the author are reasoned and evaluated in comparison with the known solutions: 1) **there is a critical analysis;** 2) partial analysis; 3) the analysis does not represent one's own opinions, but quotes from other authors	The solutions proposed by the author are substantiated by experimental research employing modern hyphenated analytical methods, conducted in accordance with the principles of green analytical chemistry. The findings are further supported by rigorous statistical analysis and lead to well-founded, evidence-based conclusions.
5.	Scientific novelty principle	5.1 Are the scientific results and provisions new? **1) completely new;** 2) partially new (25-75% are new); 3) not new (less than 25% are new)	The novelty of this research lies in the development and validation of innovative dynamic headspace extraction combined with gas chromatography-mass spectrometry (DHS–GC–MS) methodologies for the determination of BVOCs in aqueous matrices. A new method was developed for the quantification of 2-pentanone, dibromomethane, α-pinene, Δ3-carene, and nonanal in model water samples. Furthermore, a novel approach was introduced for the direct determination of dimethyl sulfide (DMS) and the indirect quantification of dimethylsulfoniopropionate (DMSP) in environmental waters. Thirdly, DMS emissions during microalgae aerosolization were studied, enabling the determination of DMS and the indirect quantification of DMSP in water.
		5.2 Are the dissertation findings new? **1) completely new;** 2) partially new (25-75% are new); 3) not new (less than 25% are new)	
		5.3 Technical, technological, economic or management decisions are new and reasonable: **1) completely new;** 2) partially new (25-75% are new); 3) not new (less than 25% are new)	
6.	The validity of the main findings	All main conclusions **are**/are not based on scientifically significant evidence or well-grounded (for qualitative research and areas of training in the arts and humanities)	The validity and reliability of the results are rigorously demonstrated. The methodological parameters were systematically evaluated, including accuracy, recovery, analyte response, limits of detection and quantification,

			and reproducibility. The measurements were performed in triplicate to ensure statistical robustness. The analytical power of gas chromatography–mass spectrometry (GC–MS) enabled sensitive, selective, and precise identification and separation of BVOCs.
7.	The main provisions for the defense	It is necessary to answer the following questions for each provision separately: 7.1 Is the provition proven? 1) **proven**; 2) rather proven; 3) rather not proven; 4) not proven 7.2 Is it trivial? 1) yes; 2) **no** 7.3 Is it new? 1) **yes**; 2) no 7.4 Application level: 1) narrow; 2) medium; 3) **wide** 7.5 Is it proven in the article? 1) **yes**; 2) no	The **provision 1** is proven in the experimental part of the work in chapter 2. A DHS–GC–MS method was developed for the determination of 2-pentanone, dibromomethane, α-pinene, Δ3-carene, and nonanal. Optimized conditions included a 500 mL trapping gas at 30 °C and a 500 mL drying gas at 40 °C. The method is flexible, easily automated, and enables BVOC analysis in both freshwater and saltwater matrices; it can be expanded to aquatic environments with varying levels of dissolved organic matter (DOM). The **provision 2** is proven in the experimental part of the work in chapter 3. A DHS–GC–MS method was optimized, including incubation temperature, trapping time, desorption conditions, sorbent selection, and moisture control, and validated for the accurate quantification of DMS and the indirect estimation of DMSP in water samples. The alkaline conversion process for indirect DMSP determination was systematically evaluated. The method was successfully applied to real environmental samples, including raw wastewater, treated

			effluent, and microalgae-rich systems. It aligns with green analytical chemistry principles, achieving an AGREEprep score of 0.40. This approach provides a foundation for future studies on biogenic sulfur emissions, algal metabolism, wastewater dynamics, and climate-relevant aerosol formation. The **provision 3** is proven in the experimental part of the work in chapter 4. The production of DMS and DMSP by the freshwater microalga *Chrysochromulina parva* and the marine microalga *Chrysotila dentata* under varying aerosolization conditions was investigated. The DHS–GC–MS results suggest that the dispersal of these microalgal strains across geographic scales is unlikely. Nevertheless, their potential impact on climate is considerable, driven by DMS emissions, which may contribute to new particle formation and cloud condensation in the atmosphere. The method demonstrates significant utility for mechanistic studies of sulfur cycling and provides a foundation for future laboratory and mesocosm experiments aimed at linking biological activity with the emission of atmospheric precursors.
8.	The principle of reliability Reliability of sources and information provided	8.1 Choice of methodology - is justified or the methodology is described in sufficient detail 1) **yes;** 2) no	The thesis is defended within the framework of the educational programme 8D05301 – Chemistry. The optimization of experimental extraction parameters and the development of the DHS–GC–MS method are highly

			appropriate to the subject of the study, demonstrating sufficient depth, clarity, and scientific rigor.
		8.2 The results of the thesis were obtained using modern methods of scientific research and methods of processing and interpreting data using computer technologies: 1) **yes;** 2) no	DHS–GC–MS is a modern analytical method widely used in scientific research for the sensitive and selective detection of analytes. It generates complex datasets that require advanced data processing and interpretation, utilizing computer-based software for peak integration, identification, quantification, and statistical analysis, in accordance with current standards of scientific research.
		8.3 Theoretical conclusions, models, identified relationships and patterns have been proven and confirmed by experimental research (for areas of training in pedagogical sciences, the results have been proven on the basis of a pedagogical experiment): 1) **yes;** 2) no	Theoretical conclusions, models, and identified relationships and patterns were validated through experimental research. The thesis has advanced both the theoretical understanding and practical methodology of DHS analysis of BVOCs in water. The established relationships between trapping and drying parameters, analyte enrichment, and water retention in sorbent tubes provide valuable insights into the fundamental principles and methodological challenges of applying DHS to aqueous samples.
		8.4 Important statements are **confirmed** / partially confirmed / not confirmed by references to current and reliable scientific literature	Important statements are confirmed by references to current and reliable scientific literature.
		8.5 Used literature sources are **sufficient**/not sufficient for a literature review	The thesis includes 158 references, reflecting a comprehensive, well-rounded, and sufficient review of the

			relevant literature.
9	Practical value principle	9.1 The thesis has theoretical value: 1) **yes**; 2) no	This thesis strengthens the theoretical and practical foundations of dynamic headspace–based BVOC analysis in water, enabling trace-level determination in complex aqueous matrices.
		9.2 The thesis is of practical importance and there is a high probability of applying the results obtained in practice: 1) **yes**; 2) no	The developed methods provide a valuable tool for environmental monitoring, pollution mapping, and investigating the influence of BVOCs on cloud formation and climate.
		9.3 Are the practice suggestions new? 1) **completely new;** 2) partially new (25-75% are new); 3) not new (less than 25% are new)	The thesis represents a methodological advancement in the measurement of BVOCs. Through the systematic optimization of dynamic headspace sampling parameters, two novel analytical methods were developed for the quantification of BVOCs in water, including 2-pentanone, dibromomethane, α-pinene, Δ3-carene, nonanal, dimethyl sulfide, and dimethylsulfoniopropionate.
10.	The quality of writing and design	Academic writing quality: 1) **high;** 2) average; 3) below average; 4) low.	The overall quality of writing and presentation is high. The thesis is well structured and clearly articulated; however, it contains some minor typographical errors, and certain aspects would benefit from further clarification.
11.	Notes on a thesis	There is a recurring issue with the introduction and inconsistent use of abbreviations throughout the thesis. After an abbreviation has been defined, it should be used directly and consistently for the remainder of the text. P 9: ….(5977A)? There is no need to add this information. P 10:… (5977A? There is no need to add this information.	

		P 11: Publications – This part is repeated earlier. The same information was mentioned in a few lines above. P 14:… the sample [20]. The sorption step was skipped in the text. P 15: …application notes – The full stop is missing. P 17:… number S h – Correct the space. P 18:… P&T, - The article is missing P 20: Figure 1 caption. Add the corresponding reference [83]. P 20: Table 2. Explain the PAH, LOD and LOQ and n/d abbreviations. P 25: Name the type/material of cryo-trap. P 27:… sulfur compounds. These compounds are not investigated in this chapter. P 33: Figure 6. Add better figures (if possible). P 36: Table 4. Explain MTs, SQTs abbreviations. P 41: Caption of Figure 7: …. of the GC. The GC-MS system? P 42: (Section 3.6). There is no such section. P 43: No use of references. References are missing on this page. P 46: Figure 10. The information on the x-axis concentration is missing in the text. P 47:… drying gas flow rate and drying gas volume. Section 3.3.4 refers only to the drying gas flow rate, as drying gas volume is reported in 3.3.5. P 53: X-axis. Add the DMSP concentration units in parenthesis. P 55:…Table 7. Add a parenthesis to sample volume unit. Delete the % on the columns of RSD and Recovery. Explain the abbreviations of FDs, FPD, PFPD, FID. What is GC/MC-ICPMS? P 57: Explain the abbreviation of GSP. P 58: …detection limits between 0.01 and 0.05 μg L^{-1}. Clarify this, as different values are mentioned in Table 7. P 59: …recoveries ranging from 85% to 113%. Better clarify these values - where was this information reported? P 60: Annex B. It is missing. P 60: ….sulphur, sulphonic acid, sulphuric acid, sulphate. Throughout the thesis, these terms are written with "f" rather than "ph." P 61: In the article…Clarify in which article, [153] or [154]? P 61: pH0-14. Add space. P 62: Explain SiSS abbreviation. It is explained on page 63. P 63: Table 9: Explain FCM, - dash symbol. Also, correct GC-MS-DHS? Is Date column meaningful?

		P 64: Figure 19. Error bars? P 65: Section conclusion is missing. Overall, Section 4 is short. References: Correct the capitals on references [29, 89, 125, 129, 141, 153]. Also, correct the names of reference [120].	
12.	Scientific level of the doctoral student's articles on the topic of research (in case of defense of the dissertation in the form of a series of articles, the official reviewers comment on the scientific level of each article of the doctoral student on the topic of research)		The main results of this thesis were published in two international peer-reviewed journals: (a) Microchemical Journal (IF: 5.1): M. Bektassov et al., Microchemical Journal 214 (2025) 113989, https://doi.org/10.1016/j.microc.2025.113989, (b) npj Climate and Atmospheric Science (IF: 8.4): B. Rosati et al., npj Climate and Atmospheric Science 9, 32 (2026), https://doi.org/10.1038/s41612-025-01305-4. Furthermore, an abstract was presented at the European Aerosol Conference 2025 in Lecce, Italy.
13.	Decision of the official reviewer (pursuant to paragraph 28 of the present Model Regulations)		The dissertation of Mr. Bektassov is a rigorous and comprehensive scientific study addressing an interesting environmental research topic. It demonstrates strong theoretical grounding, appropriate methodology, and logically interpreted results. The findings are scientifically sound, contribute original knowledge to the field, and provide a solid basis for further research. The thesis meets all formal and substantive requirements of a doctoral dissertation. Considering its scientific novelty and practical relevance in Chemistry, it fully

			justifies awarding the Doctor of Philosophy (PhD) degree in Chemistry to Mr. Bektassov Marat Aidaruly.

In reviews, official reviewers indicate one of the following solutions:

1) to award the degree of Doctor of Philosophy (PhD);

2) send the thesis for revision (except for cases of thesis defense in the form of a series of articles);

3) refuse to award the degree of Doctor of Philosophy (PhD).

Copies of the reviews of the official reviewers are handed over to the doctoral student no later than 5 (five) working days before the defense of the thesis.

Official Reviewer:
Associate Professor Agapios Agapiou
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Nicosia, Cyprus

(place of work, academic title)

(signature)

Agapios Agapiou

(FULL NAME)