REVIEW
of the official reviewer
for the dissertation of Tolkyn S. Kurmanbayeva on the topic "Development of simple and accurate methods for on-site gas chromatographic determination of volatile organic compounds in environmental objects using solid-phase microextraction" 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 dissertation (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:	Development of accurate methods for on-site gas chromatographic determination of volatile organic compounds in environmental objects using solid-phase microextraction (Grant No. AP14871374). The dissertation was carried out within the framework of the state-funded research project AP14871374 (2022–2024), supported by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan. The research directly addresses the project's objectives through the development of accurate analytical methodologies for the on-site determination of volatile organic compounds in environmental samples. Furthermore, the dissertation is aligned with the national scientific priority concerning the rational use of water resources, flora and fauna, and ecology.
		√ 1) The dissertation 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 dissertation 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)	
2.	Importance for science	The work **makes** a significant contribution to science, and its importance is **well disclosed**	The dissertation makes a valuable contribution to analytical chemistry through the development and application of simple, reliable, and cost-effective methods combining solid-phase microextraction with portable gas chromatography for the on-site determination of volatile organic pollutants in air, water, and soil. Its scientific relevance lies particularly in advancing quantitative field analysis, addressing challenges related to calibration, matrix effects, and analytical performance, and demonstrating the applicability of the proposed methods under real environmental conditions. The importance of the research is clearly presented and well justified throughout the dissertation, particularly in relation to the need for rapid and reliable environmental monitoring and timely decision-making in pollution incidents.
3.	The principle of independence	Independence level: √ **1) High;** 2) Medium; 3) Low; 4) No independence	The dissertation demonstrates a high level of scientific independence, as reflected in the author's substantial involvement in the design, development, optimization, and validation of the proposed analytical methodologies. The research shows the author's ability to identify relevant scientific challenges, select appropriate experimental approaches, critically interpret the results, and formulate well-supported conclusions. Furthermore, the author's contributions to the scientific

			publications arising from the dissertation provide additional evidence of her capacity to conduct independent research and contribute to the advancement of analytical chemistry.
4.	The principle of inner unity	4.1 Justification of the relevance of the dissertation: √ **1) Justified;** 2) Partially justified; 3) Not justified.	The dissertation addresses a relevant challenge in environmental analytical chemistry: the need for rapid, reliable, and cost-effective determination of volatile organic pollutants directly at sampling sites. Its significance is particularly evident in environmental emergencies and remote locations, where delays associated with sample transportation and laboratory analysis may compromise timely decision-making. The research therefore has both scientific and practical relevance for environmental monitoring and pollution assessment.
		4.2 The content of the dissertation reflects the topic of the dissertation: √ **1) Reflects;** 2) Partially reflects; 3) Does not reflect	The dissertation fully reflects its stated topic, focusing on the development and application of solid-phase microextraction coupled with portable gas chromatography for the determination of volatile organic compounds in environmental samples. The experimental chapters address the analysis of BTEX in atmospheric air and transformation products of unsymmetrical dimethylhydrazine in water and soil, covering the different matrices and analytical challenges identified in the title.
		4.3. The purpose and objectives correspond to the topic of the dissertation: √ **1) correspond;** 2) partially correspond; 3) do not correspond	The overall purpose and specific objectives are clearly defined and consistent with the dissertation topic. Each objective addresses a particular analytical application, from the determination of BTEX in air to the adaptation and development of methods for rocket fuel transformation products in water and soil. The experimental work directly

			responds to these objectives, and the results demonstrate their successful achievement.
		4.4 All sections and provisions of the dissertation are logically interconnected: √ **1) completely interconnected;** 2) the interconnection is partial; 3) there is no interconnection	The dissertation presents a coherent and logically structured progression from the identification of existing analytical limitations to the development, optimization, and practical application of the proposed methodologies. The literature review establishes the scientific background, while the experimental chapters progressively address increasingly complex analytical matrices. The conclusions and methodological recommendations are directly supported by the experimental findings, ensuring consistency throughout the dissertation.
		4.5 The new solutions (principles, methods) proposed by the author are reasoned and evaluated in comparison with the known solutions: √ **1) critical analysis is presented;** 2) analysis is partial; 3) the analysis does not represent own opinions, but quotes from other authors 4) analysis is not available	The proposed analytical approaches are scientifically justified and critically evaluated against existing methodologies. Particular attention is given to extraction conditions, calibration strategies, matrix effects, detection limits, and analytical precision. Comparisons with conventional laboratory-based methods and previously reported SPME procedures support the assessment of their advantages and limitations. The application to real environmental samples further demonstrates their practical feasibility, while the author appropriately acknowledges the analytical constraints associated with portable instrumentation.
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 dissertation presents significant scientific novelty, particularly in the development and adaptation of SPME-based methodologies for quantitative on-site environmental analysis. The original contributions include the implementation of vacuum-assisted headspace solid-phase

			microextraction under field conditions, the development of appropriate calibration strategies, and their application to the determination of volatile organic pollutants in different environmental matrices. Although the underlying analytical techniques are well established, their optimization, integration with portable gas chromatography, and experimental validation in real environmental scenarios constitute original and scientifically relevant contributions to analytical chemistry.
		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)	The dissertation provides original experimental findings concerning the analytical performance and practical applicability of SPME-based methods under field conditions. Particularly relevant are the results on extraction efficiency, matrix effects, calibration strategies, detection limits, and quantitative performance in different environmental matrices. Furthermore, the application of the developed methodologies to real atmospheric air samples and soils contaminated by rocket fuel provides new experimental evidence of their suitability for environmental monitoring. These findings represent a valuable contribution to the advancement of on-site analytical methodologies.
		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)	The dissertation proposes technically sound and partially novel solutions for the rapid determination of environmental pollutants under field conditions. The integration of SPME and vacuum-assisted headspace extraction with portable gas chromatography, together with the optimization of experimental conditions and calibration procedures, represents a practical improvement over conventional laboratory-based approaches for

			specific applications. These solutions are supported by experimental evidence and offer advantages in terms of analytical simplicity, reduced solvent consumption, and faster availability of results, particularly in remote locations and environmental emergencies.
6.	The validity of the main findings	All main conclusions **are** based on scientifically significant evidence	The main findings and conclusions of the dissertation are supported by sufficient experimental evidence. The developed analytical methodologies were systematically optimized and evaluated in terms of relevant analytical performance characteristics, including sensitivity, precision, recovery, and matrix effects. Their applicability was further demonstrated through the analysis of real environmental samples, with comparisons to laboratory-based methodologies where appropriate. The experimental results are consistent with the conclusions presented and provide a sound scientific basis for the proposed methodological recommendations.
7.	The main provisions for the defense	Provision 1: 7.1 Is the provision proven? √ **1) proven;** 2) rather proven; 3) rather not proven; 4) not proven 5) in the current wording, it is impossible to verify the proof of the provision 7.2 Is it trivial? 1) yes; √ **2) no** 3) in the current wording, it is impossible to verify whether the provision is trivial	Provision 1: 7.1: The provision is supported by experimental results demonstrating the suitability of the proposed SPME-GC-PID methodology for the quantitative determination of BTEX in atmospheric air. The extraction conditions and calibration strategy were systematically investigated, and the analytical performance was evaluated in terms of sensitivity, precision, and accuracy. The comparison with laboratory-based measurements further supports the reliability of the approach. The experimental evidence adequately justifies the proposed

		7.3 Is it new? √ **1) yes;** 2) no 3) in the current wording, it is impossible to verify the novelty of the provision 7.4 Application level: 1) narrow; √ **2) medium;** 3) wide 4) in the current wording, it is impossible to verify the application level of the provision 7.5 Is it proven in the article? √ **1) yes;** 2) no 3) in the current wording, it is impossible to verify the proof of the provision in the article Provision 2: 7.1 Is the provision proven? √ **1) proven;** 2) rather proven; 3) rather not proven; 4) not proven 5) in the current wording, it is impossible to verify the proof of the provision 7.2 Is it trivial? 1) yes; √ **2) no** 3) in the current wording, it is impossible to verify whether the provision is trivial 7.3 Is it new? √ **1) yes;** 2) no	analytical procedure and its applicability to on-site environmental monitoring. 7.2: The provision is not trivial, as it addresses the practical challenges of achieving reliable quantitative results under field conditions. Establishing appropriate extraction parameters and a suitable calibration strategy requires experimental optimization and careful consideration of the limitations of portable instrumentation. The proposed procedure is therefore supported by analytical reasoning rather than being a straightforward application of an established method. 7.3: Although SPME and portable gas chromatography are established analytical techniques, the provision introduces a specifically optimized procedure for on-site BTEX quantification, including defined extraction conditions and a calibration strategy adapted to field measurements. The originality lies in the development and experimental assessment of this particular analytical approach rather than in the underlying instrumental principles. 7.4: The proposed methodology has a medium application level, as it can be applied to the monitoring of BTEX in atmospheric air, particularly in remote locations or situations requiring rapid analytical results. Although its applicability is limited to a specific group of pollutants and defined analytical conditions, the approach has practical potential for environmental monitoring and field investigations.

		3) in the current wording, it is impossible to verify the novelty of the provision 7.4 Application level: 1) narrow; **√ 2) medium;** 3) wide 4) in the current wording, it is impossible to verify the application level of the provision 7.5 Is it proven in the article? **√ 1) yes;** 2) no 3) in the current wording, it is impossible to verify the proof of the provision in the article Provision 3: 7.1 Is the provision proven? **√ 1) proven;** 2) rather proven; 3) rather not proven; 4) not proven 5) in the current wording, it is impossible to verify the proof of the provision 7.2 Is it trivial? 1) yes; **√ 2) no** 3) in the current wording, it is impossible to verify whether the provision is trivial 7.3 Is it new? **√ 1) yes;** 2) no 3) in the current wording, it is impossible to verify the novelty of the provision 7.4 Application level:	7.5: The provision is supported by the experimental results published in Advances in Sample Preparation (2024), entitled "Simple and accurate on-site quantitation of BTEX in atmospheric air using solid-phase microextraction and gas chromatography with photoionization detection." The article presents the development, optimization, and analytical evaluation of the proposed methodology, providing experimental evidence supporting its applicability to quantitative on-site BTEX determination. Provision 2: 7.1: The provision is supported by experimental investigations of matrix effects in different water samples and aqueous soil extracts. The results demonstrate that, under the optimized extraction conditions, matrix effects are sufficiently limited to allow reliable quantification of the selected transformation products using an external calibration approach. The analytical performance and recovery studies provide additional support for this conclusion. 7.2: The provision is not trivial, since matrix effects are a major challenge in quantitative headspace solid-phase microextraction, particularly when analysing compounds with different physicochemical properties in complex environmental matrices. Demonstrating that these effects can be sufficiently minimized under specific experimental conditions requires systematic investigation and experimental verification.

		1) narrow; **√ 2) medium;** 3) wide 4) in the current wording, it is impossible to verify the application level of the provision 7.5 Is it proven in the article? **√ 1) yes;** 2) no 3) in the current wording, it is impossible to verify the proof of the provision in the article Provision 4: 7.1 Is the provision proven? 1) proven; 2) rather proven; 3) rather not proven; 4) not proven 5) in the current wording, it is impossible to verify the proof of the provision 7.2 Is it trivial? 1) yes; 2) no 3) in the current wording, it is impossible to verify whether the provision is trivial 7.3 Is it new? 1) yes; 2) no 3) in the current wording, it is impossible to verify the novelty of the provision 7.4 Application level: 1) narrow; 2) medium; 3) wide	7.3: The novelty of this provision lies in the experimental demonstration that matrix effects are limited for the selected UDMH transformation products under the specified Vac-HSSPME conditions. This finding provides new methodological knowledge regarding the suitability of simplified calibration strategies for their quantitative determination in water and aqueous soil extracts. 7.4: The findings have practical applicability to the determination of UDMH transformation products in environmental samples, particularly in areas affected by rocket fuel contamination. Although the conclusions are specific to the investigated compounds and extraction conditions, they facilitate quantitative analysis by reducing the need for complex matrix-dependent calibration procedures. 7.5: The provision is supported by the experimental results published in the International Journal of Environmental Analytical Chemistry (2025), which demonstrate the suitability of Vac-HSSPME-GC-NPD for the determination of UDMH transformation products in water. The evaluation of matrix effects and recovery studies using different water samples supports the conclusion that external standard calibration can provide reliable quantitative results under the investigated conditions. Provision 3: 7.1: The provision is supported by experimental results comparing the extraction of UDMH transformation products from soil using distilled

		4) in the current wording, it is impossible to verify the application level of the provision 7.5 Is it proven in the article? 1) yes; 2) no 3) in the current wording, it is impossible to verify the proof of the provision in the article	water, 1 M NaOH, and 1 M HCl. The results demonstrate that distilled water provides lower variability in extraction efficiency and reduced matrix effects, supporting its selection as the preferred extraction medium for the proposed analytical methodology. 7.2: The provision is not trivial, as the selection of an appropriate extraction medium is particularly challenging when dealing with compounds exhibiting different physicochemical properties. The influence of extraction conditions on analyte recovery and matrix effects requires systematic experimental evaluation, and the identification of distilled water as a suitable extraction medium represents a relevant methodological finding. 7.3: The provision presents an original experimental finding regarding the influence of extraction media on the determination of UDMH transformation products in soil. The demonstration that distilled water provides more favourable analytical conditions than acidic or alkaline solutions contributes new knowledge to the optimization of Vac-HSSPME-based methods for these contaminants. 7.4: The findings have practical relevance for the analysis of soils potentially contaminated with UDMH and its transformation products. The use of distilled water simplifies sample preparation, reduces reagent consumption, and facilitates field applications. Although the approach is specifically intended for the investigated contaminants, it provides a useful methodological basis for

			environmental monitoring in areas affected by rocket fuel contamination. 7.5: The provision is supported by the experimental results published in Microchemical Journal (2025), entitled “On-site gas chromatographic quantitation of transformation products of unsymmetrical dimethylhydrazine in soil using vacuum-assisted headspace solid-phase microextraction.” The study evaluates the influence of different extraction media on analytical performance and demonstrates the advantages of distilled water in reducing matrix effects. These findings support the methodological approach adopted for the determination of UDMH transformation products in soil.
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 details √ **1) yes;** 2) no	The selection of the analytical methodologies is scientifically justified and consistent with the research objectives. The dissertation provides detailed descriptions of the experimental procedures, including sampling, extraction conditions, chromatographic analysis, calibration, and method validation. This information is sufficient to understand the experimental approach and supports the reproducibility of the developed methods.
		8.2 The results of the dissertation were obtained using modern methods of scientific research and methods of processing and interpreting data using computer technologies: √ **1) yes;** 2) no	The research employs established and modern analytical techniques, including SPME, vacuum-assisted headspace microextraction, and portable gas chromatography with selective detection. Instrumental data processing and quantitative calibration procedures were applied, while finite element modelling using COMSOL Multiphysics was employed to investigate extraction kinetics. These approaches provide an appropriate

			methodological and computational basis for the research.
		8.3 Theoretical conclusions, models, identified relationships and patterns have been proven and confirmed by experimental research: **√ 1) yes;** 2) no	The main conclusions regarding extraction efficiency, matrix effects, calibration strategies, and analytical performance are supported by experimental investigations. The relationships identified during method optimization were evaluated under controlled conditions and, where applicable, verified through the analysis of real environmental samples. The experimental evidence is consistent with the conclusions and methodological recommendations presented in the dissertation.
		8.4 Important statements are **confirmed** by references to current and reliable scientific literature	The dissertation provides an appropriate scientific background supported by references to relevant peer-reviewed publications, established analytical methodologies, and internationally recognized standards. The literature is used to contextualize the research objectives, identify existing methodological limitations, and compare the developed approaches with previously reported methods. Overall, the principal scientific statements are adequately supported by the cited literature.
		8.5 Used literature sources are **sufficient** for a literature review	The dissertation includes 131 references covering environmental monitoring, portable gas chromatography, solid-phase microextraction, analytical method development, and the determination of volatile organic pollutants. The literature review provides an adequate overview of the current state of knowledge and identifies the

			main analytical challenges addressed by the research. The number, scope, and relevance of the cited sources are sufficient for the objectives of the dissertation.
9	Practical value principle	9.1 The dissertation has theoretical value: √ **1) yes;** 2) no	The dissertation contributes to the theoretical and methodological understanding of solid-phase microextraction for quantitative on-site analysis. The investigation of extraction kinetics, matrix effects, and calibration strategies provides useful scientific knowledge regarding the factors influencing analytical performance under field conditions. These findings contribute to the development of more reliable approaches for environmental analysis using portable instrumentation.
		9.2 The dissertation is of practical importance and there is a high probability of applying the results obtained in practice: √ **1) yes;** 2) no	The dissertation has clear practical significance, as demonstrated by the successful application of the developed methods to real environmental samples, including atmospheric air and soils contaminated with rocket fuel transformation products. The proposed procedures enable relatively rapid and cost-effective on-site measurements, particularly in remote locations where conventional laboratory analysis may be difficult or delayed. Their potential application to environmental monitoring and emergency response is therefore well justified, although routine implementation would require consideration of instrumental and operational limitations.
		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 practical recommendations are partially novel, as they introduce experimentally supported procedures for implementing SPME-based quantitative analysis under field conditions.

			Particularly relevant are the proposed calibration strategies, the use of vacuum-assisted extraction with portable gas chromatography, and the simplified sample preparation procedures for contaminated soils. Although these recommendations build upon established analytical principles, their adaptation and validation for specific environmental applications represent original and practically useful contributions.
10.	The quality of writing and design	Academic writing quality: √ **1) high;** 2) average; 3) below average; 4) low.	The dissertation is well structured and generally written in clear, appropriate scientific language. The objectives, experimental procedures, results, and conclusions are presented in a coherent manner, supported by relevant tables, figures, and references. The overall organization facilitates understanding of the research and its main contributions. Although some minor linguistic and editorial improvements could be made, these do not affect the scientific quality of the work.
11.	Comments to the dissertation	The dissertation represents a valuable and original contribution to analytical chemistry, supported by systematic experimental research and three publications in international peer-reviewed scientific journals. The work demonstrates scientific independence, methodological rigor, and a coherent presentation of the results and conclusions. The contributions of previous researchers are appropriately acknowledged through scientific references, and the author's own contributions to the published research are clearly identified. Based on the examination of the dissertation, no apparent concerns regarding academic integrity have been identified. Overall, the dissertation meets the expected scientific standards for the award of a PhD degree.	
12	The scholarly quality of the doctoral candidate's articles on the research topic (in	The dissertation is based on three peer-reviewed articles published in international journals, all directly related to its research objectives and contributing to the development of analytical methodologies for on-site determination of volatile organic compounds in environmental samples. 1. Kurmanbayeva et al. (2024), Advances in Sample Preparation. This article presents an analytical method for on-site BTEX determination in atmospheric air using SPME and portable GC-PID. The study includes appropriate optimisation, calibration and analytical performance evaluation,	

	the case of a dissertation defended in the form of a series of articles, the official reviewers provide comments on the scholarly quality of each of the candidate's articles on the research topic)	together with a comparison of on-site and laboratory measurements. It represents a scientifically sound and practically relevant contribution to environmental monitoring. 2. Kurmanbayeva et al. (2025), International Journal of Environmental Analytical Chemistry. This article investigates the determination of UDMH transformation products in water using vacuum-assisted HS-SPME and portable GC-NPD. The evaluation of detection limits, recoveries and matrix effects supports the applicability of the proposed methodology. Although its novelty is moderate and its sensitivity is lower than that of GC-MS, the work provides a useful contribution to field-based environmental analysis. 3. Kurmanbayeva et al. (2025), Microchemical Journal. This article extends the proposed analytical approach to UDMH transformation products in soil, focusing on sample preparation and matrix effects. The optimisation of extraction conditions improves analytical performance in this complex matrix. The study is methodologically sound and provides a relevant contribution to the analysis of contaminated soils. In general, the three articles demonstrate a coherent research progression and good scientific quality. Although their novelty lies mainly in adapting and optimising established techniques, they provide original and practically relevant contributions to on-site environmental analysis. All three publications are directly related to the dissertation topic and merit consideration in its scientific evaluation.
13	Decision of the official reviewer	1) √ **to award the degree of Doctor of Philosophy (PhD);** 2) to return the dissertation for revision if the dissertation does not comply with paragraphs 4 and/or 6 of the Rules and/or only partially complies with the principles (except for the principle of academic integrity) specified in paragraph 5 of the Rules, and/or if it contains deficiencies requiring correction. In the case of a dissertation submitted in the form of a series of articles, the dissertation shall not be returned for revision; 3) to refuse to award the degree of Doctor of Philosophy (PhD) or Doctor by Profile in the event of a violation of the principle of academic integrity or non-compliance with the principles of scientific novelty, internal consistency, and reliability.

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