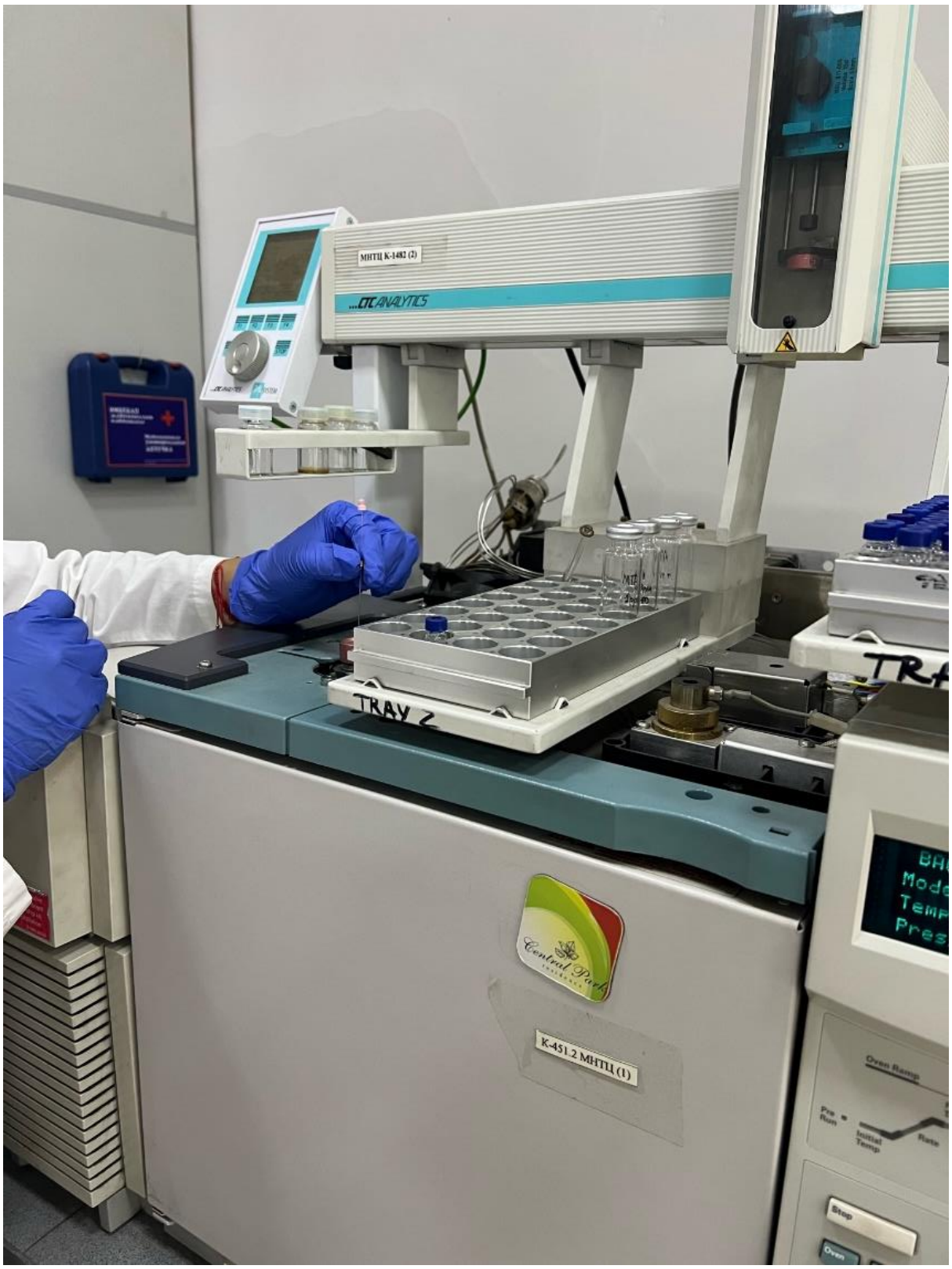
Brief information about the project

Name of the project	AP09057951 «Development of “green” methods for determination of pesticides in environmental objects based on Vacuum-assisted headspace solid phase microextraction»
Relevance	The development of "green" methods for detecting organic pollutants in environmental samples is a large area of scientific research in analytical chemistry. The importance of such studies is demonstrated by the fact that standard methods for determining organic substances in complex matrices rely on complex and labor-intensive sample preparation methods that necessitate the use of large volumes of toxic organic solvents. The development of "green" analytical methods is especially important for pesticides in use today, which are subject to ongoing regulatory oversight and monitoring. Headspace solid-phase microextraction (HS-SPME) is a promising organic solvent-free sample preparation technique that combines extraction, concentration, and purification in one step. The main issues with quantitative pesticide determination using HS-SPME are low analysis accuracy due to the matrix effect and low extraction efficiency of analytes with high boiling points and low Henry constants. To improve the sensitivity and reproducibility of pesticide detection in environmental objects, the proposed project will use a method that combines vacuum SPME (Vac-SPME) and cold fiber SPME. To improve analyte determination accuracy and control the matrix effect, the multiple Vac-SPME method will be used. The outcomes of research on the kinetics and processes of cold fiber SPME and Vac-SPME for different pesticide classes can serve as the foundation for the creation of accurate and affordable "green" techniques for the detection of additional analytes in different matrices. The cost, time, and labor costs of analysis will be decreased when the developed procedures are introduced into laboratories that determine the presence of pesticide residues in various items within the parameters of state regulations.
Purpose	Develop new accurate and cost-effective green methods for quantification of pesticide residues in environmental objects based on vacuum-assisted headspace solid-phase microextraction (Vac-HSSPME) and cold-fiber solid-phase microextraction (CF-SPME).
Objectives	Task 1. Development of a methodology for the determination of organic pesticides in water based on Vac-SPME Task 2. Development of a methodology for the determination of organic pesticides in soil based on Vac-SPME and CF-SPME Task 3. Development of a methodology for the determination of organic pesticides in grain samples based on Vac-SPME

	Task 4. Preparation for the certification of the analysis methodology, testing of the developed methods on real samples of water, soil and grain crops
Expected and achieved results	The project developed four new techniques for the quantification of nitrogen-containing organic pesticide residues in water, soil and grain crop samples. The developed methods are based on the use of a “green” sample preparation method - solid-phase microextraction (SPME), which allows the extraction of target pesticides from various objects without the use of organic solvents and lengthy and expensive procedures for preliminary extraction and purification of extracts before analysis. To improve the efficiency of pesticide extraction, modifications were used for the first time - vacuum SPME and SPME with cooling of the sorbent during extraction. Lowering the pressure in the system made it possible to increase the efficiency of extraction of target pesticides from water by 4–17 times, from soil by 3–7 times, and from wheat samples by 1.2–6 times. The developed method for determining pesticides in water makes it possible to determine analytes in the concentration range of 0.5 – 25 μg/l with an accuracy of 107-117% and detection limits from 0.002 to 0.05 μg/l. The developed method for determining pesticides in soil provides quantitative determination of analytes in the concentration range of 25–200 ng/g with an accuracy of 69–109% and detection limits in the range from 0.1 to 4 ng/g. The developed method for wheat samples provides quantitative determination of analytes in the concentration range from 5 to 500 ng/g with an accuracy in the range of 70-131% and detection limits in the range of 0.1 – 3 ng/g. The developed methods provide a low-cost and green alternative to standard methods for the analysis of pesticides from complex matrices. Compared to other analytical techniques that use large volumes of organic solvents and disposable (disposable) cartridges and sorption tubes, the developed methods offer more than 3 times lower material costs and complete elimination of the use of organic solvents during sample preparation.
Research team members with their identifiers (Scopus Author ID, Researcher ID, ORCID, if available) and links to relevant profiles	Project leader, Ph.D. Dina Orazbayeva, since 2015 has been developing "green" methods for the determination of organic compounds in environmental objects. D. Orazbayeva has developed five new techniques for the determination of organic compounds in environmental objects based on solid-phase microextraction. Hirsch Index 5, Author ID in Scopus: 57189031525; Researcher ID Web of Science: O-5505-2017; ORCID ID: 0000-0002-3535-4576. Aray Zhakupbekova is working on her Ph.D. thesis on the development of new techniques for the determination of organic pollutants in soils based on vacuum solid-phase microextraction. She has seven publications in international journals with impact factor: Chemosphere, Trends in Environmental Analytical Chemistry and Journal of Dairy

	Science. Hirsch Index 5, Author ID in Scopus: 55347201400; Researcher ID Web of Science: P-5639-2017; ORCID ID: 0000-0002-1145-3457. Kapar Anel, a Ph.D.-candidate, performs a thesis on the development of numerical models of SPME. Hirsch Index 2, Author ID in Scopus: 57191839630; ORCID ID: 0000-0001-9828-5654. Bektassov Marat, Ph.D.-doctoral student of the 3rd year, is engaged in the development of equipment, materials and methods for "green" environmental monitoring. Hirsch Index 3, Author ID in Scopus: 57188593383; Researcher ID Web of Science: P-5639-2017. Alua Zhumadildinova Ph.D.-doctoral student of the 2nd year is working on her Ph.D. thesis on the development of methods for the determination of organic pollutants in environmental objects based on vacuum SPME. ORCID ID: 0000-0003-2520-7385. Dyussenkulova Balgyn is a 3rd year undergraduate student. ORCID ID: 0009-0004-0322-3696.
List of publications with links to them	1. Orazbayeva D., Muratuly A., Bektassov M., Zhakupbekova A., Kenessov B., Chromatographic determination of pesticides in soil: Current trends in analysis and sample preparation // Trends Environ. Anal. Chem. – 2022. – Vol. 35. – P. e00174. https://doi.org/10.1016/j.teac.2022.e00174 (IF=11.2, Q1 in "Chemistry, Analytical") 2. Zhakupbekova A., Baimatova N., Psillakis E., Kenessov B., Quantification of trace transformation products of rocket fuel unsymmetrical dimethylhydrazine in sand using vacuum-assisted headspace solid-phase microextraction // Environ. Sci. Pollut. Res. – 2022. – Vol. 35. – P. e00174. https://doi.org/10.1007/s11356-021-17844-1. (IF=5.8, Q1 in "Pollution") 3. Dyussenkulova B., Zhakupbekova A., Zhumadildinova A., Yusupova K., Kapar A., Orazbayeva D. Determination of nitrogen-containing pesticides in soil using vacuum-assisted headspace solid-phase microextraction // Chemical Bulletin of Kazakh National University. – 2023. – 110. – Is.4. –P. 12-22. – https://doi.org/10.15328/cb1340. (journal indexed in Web of Science Core Collection).
Patents	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TRAY 2
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K-451.2 MHTLL (1)
Oven Ramp
Pre Run
Initial Temp
Rate
Step
Oven