

FAST-TRACK BTX DETECTION IN WATERWAYS: PORTABLE MIMS FOR ON-SITE ENVIRONMENTAL MONITORING

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The detection of Benzene, Toluene, and Xylenes (BTX) in water is critical for assessing pollution in aquatic environments, as these compounds pose significant environmental and public health risks. Traditional methods for BTX monitoring often require lab-based analysis, causing delays in obtaining actionable data. This study introduces an innovative use of portable Membrane Inlet Mass Spectrometry (MIMS) for the rapid, on-site detection of BTX. It provides a swift, efficient, and dependable method for real-time monitoring of environmental contaminants in waterways.

Introduction

In a world facing increasing environmental challenges due to both natural disasters and human-induced activities, pollution has become a significant global issue. The contamination of water, air, and soil by harmful substances poses a threat to all living organisms and disrupts ecosystems. Rapid detection and intervention are crucial to prevent further damage, particularly in the wake of disasters and industrial accidents. Portable environmental monitoring tools, such as membrane inlet mass spectrometry (MIMS), offer significant advantages in this regard. MIMS technology allows for real-time, in-field analysis of volatile organic compounds (VOCs) and other pollutants, without the need for extensive sample preparation.

Since its introduction in the 1960s, MIMS has proven to be highly effective for monitoring environmental pollutants in water, soil, and air, providing a reliable and portable solution for environmental monitoring. Its application can significantly improve our response to environmental threats, offering high sensitivity, rapid results, and the ability to detect contaminants at the site of an incident.

Experimental section

Table 1 List of the BTX compounds used for validation of the MIMS method.

Compound	CAS number	Molecular weight (g/mol ⁻¹)	Target Ion (m/z)	MRL (I, II class of water) µg L ⁻¹	MRL (drinking water) µg L ⁻¹
Benzene	71-43-2	78.11	78, 77	500	2
Toluene	108-88-3	92.14	91, 92	500	700
Xylene	1330-20-7	106.168	105, 106	50	50

The details of the compounds of interest are presented in Table 1, including their molecular weights, target ions, and maximum residue levels (MRLs) allowed in drinking and irrigation water. The MRL limits are expressed in micrograms per litre of water (mgL⁻¹) as determined by the regulations of the Republic of Serbia where class I and II can be used for irrigation.

Our testing, calibration, and validation of the MIMS portable analytical system for water-quality monitoring were carried out in accordance with the relevant guidance documents. Following the guidelines for the validation and verification of quantitative and qualitative test methods and the analytical quality control requirements, we clearly demonstrate that BTX from water can be successfully quantified with a high level of confidence in the results.

Experimental set up

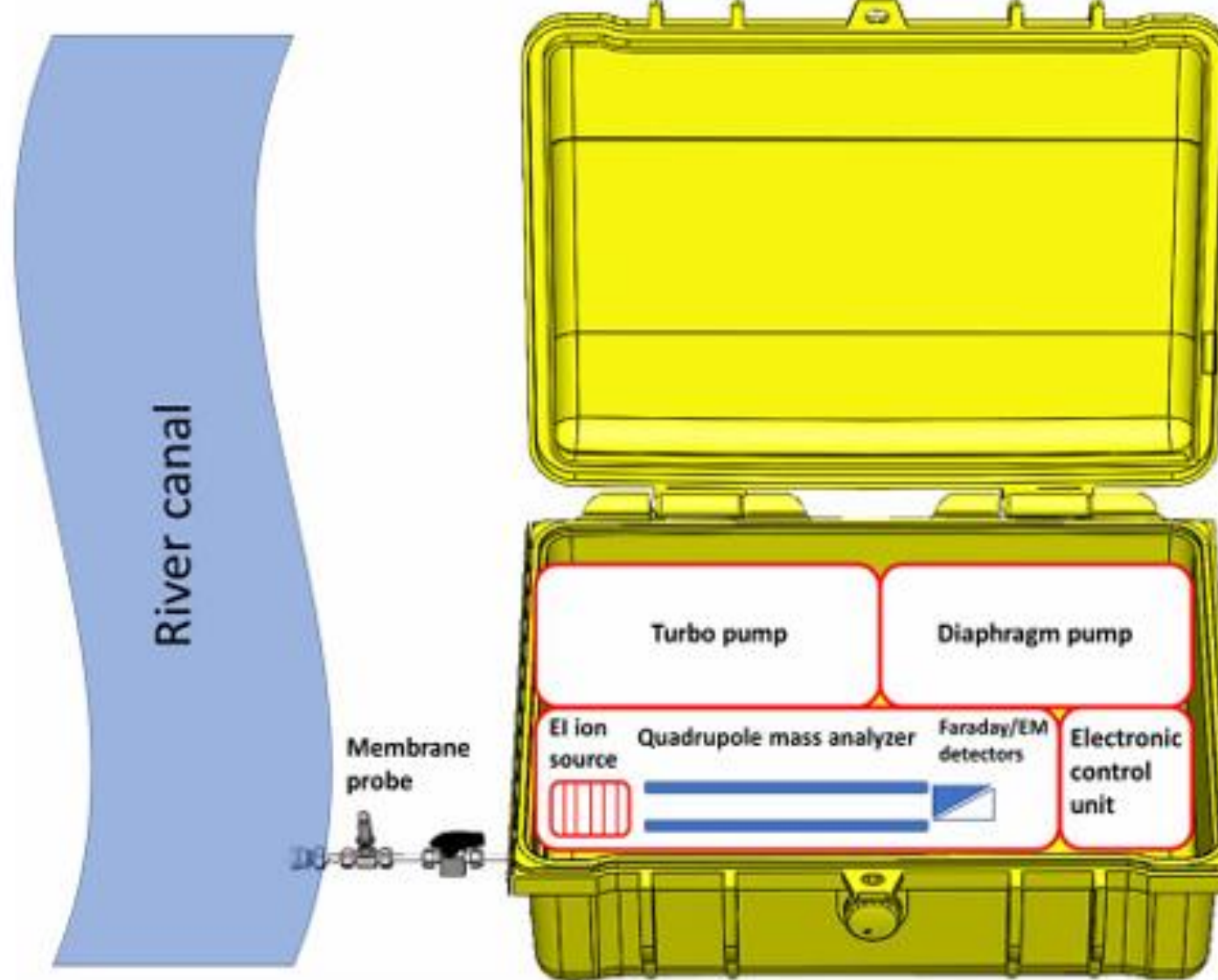


Fig. 1 Schematic of the MIMS system used for the river canal water monitoring.

The experimental setup utilized MIMS system (Fig. 1) designed for real-time monitoring of organic contaminants in water. The system employed a polydimethylsiloxane (PDMS) membrane inlet, which selectively allowed organic compounds to pass through while blocking water.

This facilitated the separation and detection of analytes using a quadrupole mass spectrometer with a mass range of 1–300 amu. The vacuum system consisted of a diaphragm pump and a turbomolecular pump, ionization was achieved through an open electron impact (EI) ion source with yttriated iridium filaments. The MIMS system was compact, with dimensions of 25 cm x 50 cm x 60 cm and a weight of 25 kg, making it suitable for field applications. The instrument provided unit mass resolution and a rapid response time of less than 0.5 seconds, ensuring near real-time analysis of water samples.

Sample analysis

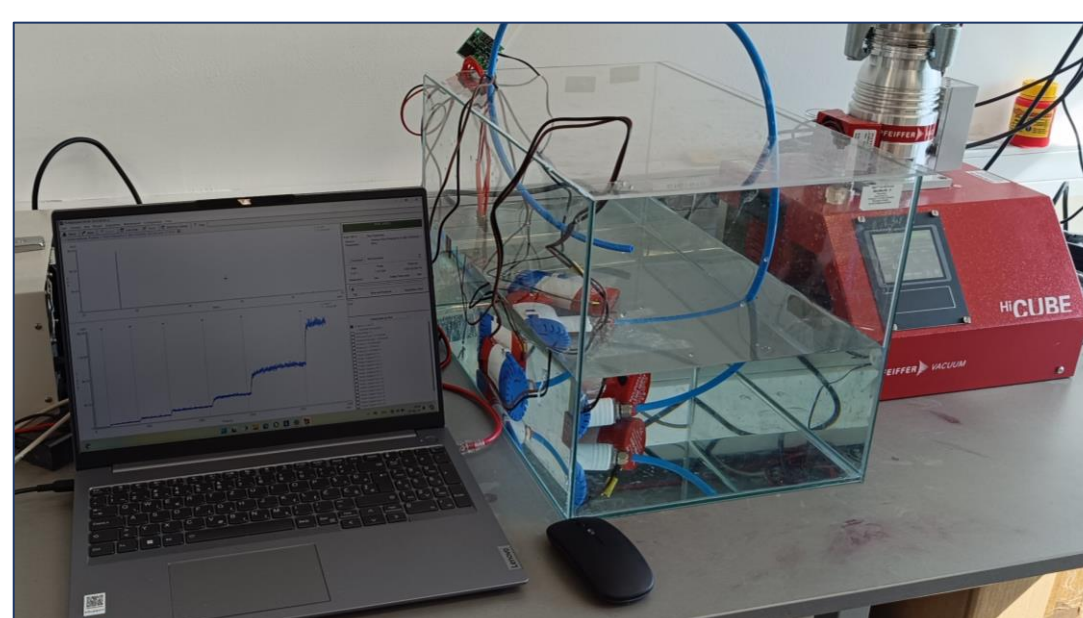


Fig. 2. Temperature-controlled aquarium with continuous mixing used for sample preparation.

A water analysis method was developed based on oil-in-water monitoring technique. During field tests on river canal water, a membrane sample probe was connected to the water flow system via a PA hose. The method was compared with a lab technique for quantifying benzene, toluene, and xylene (BTX) residues in water. Validation used DI water and DTD canal water, spiked with BTX standards at concentrations from 10 to 250 mg/L. Samples were prepared in a temperature-controlled aquarium with continuous mixing, and the membrane probe was inserted directly for analysis (Fig 2.). All measurements were taken at ambient temperature. The sample was introduced directly into the vacuum system.

Results and discussion

The analysis of BTX in water and the total time needed for the on-site analysis was ~10 min. During the validation study, all the tests were performed in the analytical range from 10–250 mgL⁻¹ to confirm the linearity, specificity, selectivity, accuracy, and precision of the method, and to determine the detection and quantification limits.

Selectivity of the method – Fig. 3 shows that the developed analytical method was able to distinguish the analyte(s) of interest from endogenous components in the matrix or other components in the sample, without interference. Fig. 2a displays the selectivity of the method when using DI water, while Fig. 2b illustrates the influence of the matrix on the selectivity of the method. After recording and enriching the canal water, it was observed that the selectivity of the method was at the same level as the DI water.

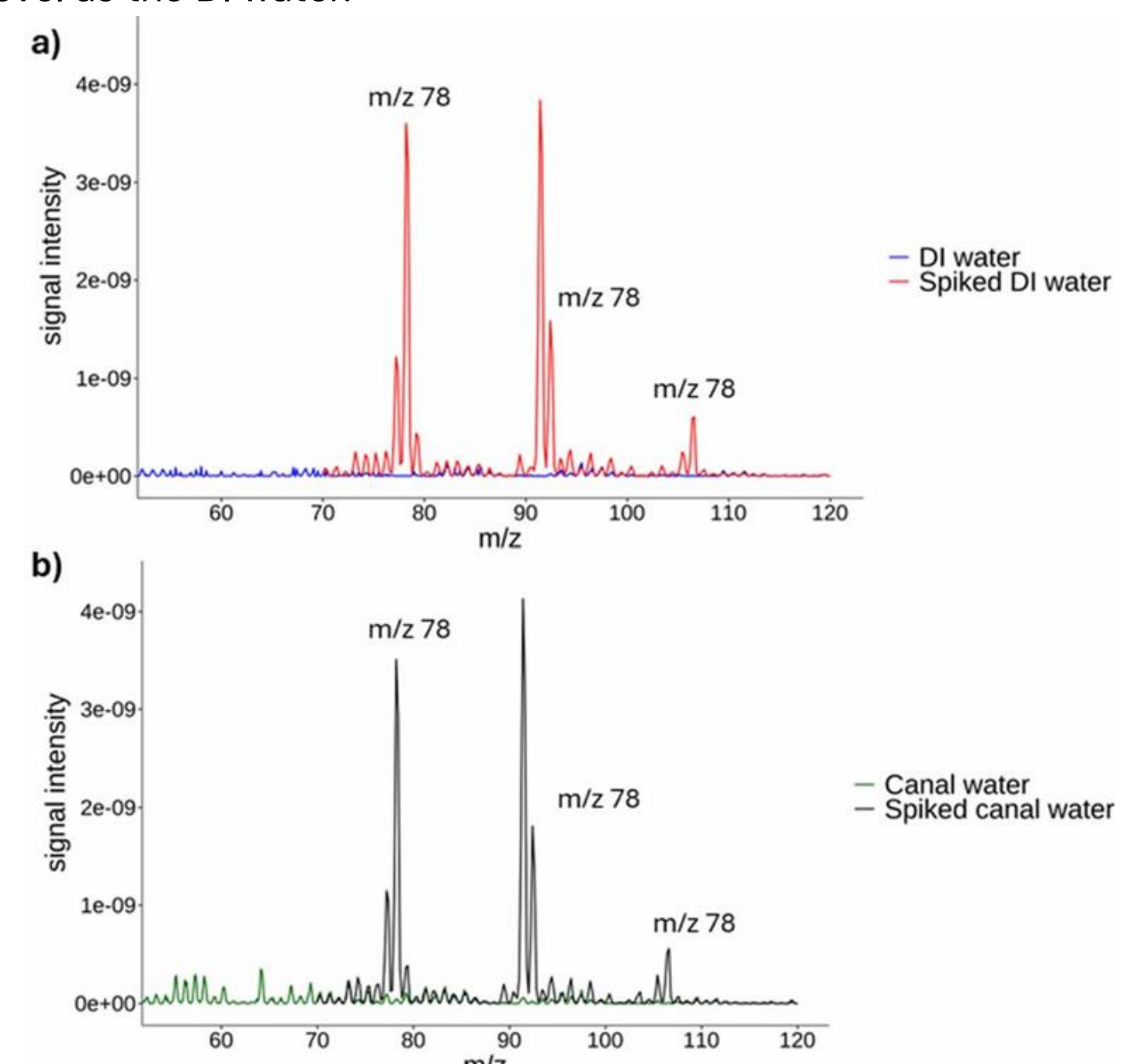


Fig. 3. Full scan of pure and spiked samples for (a) DI water and (b) canal water using the portable MIMS.

Limit of detection (LOD) and limit of quantification (LOQ)

– for target compounds, using the MIMS, analytical standard of 10 mgL⁻¹ was used, which was added to the water sample at a defined concentrate on range. Our results show (Table 2) that the LOQ values were significantly lower than the MRL values for irrigation water and slightly above the MRL values for drinking water.

Table 2. Results for the LOQ and LOD values obtained using the MIMS.

Analyte	MIMS (LOD), µg L ⁻¹	MIMS (LOQ), µg L ⁻¹
Benzene	4.88	16.27
Toluene	7.43	24.77
Xylene	7.46	24.85

In this study, we employed a portable MIMS device to create and validate a method for the fast quantitative analysis of BTX in river and irrigation canal water, covering a specific analytical range. The system's portability, rapid response, and accuracy make it a valuable instrument for environmental monitoring and the on-site detection of water contaminants.

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Acknowledgments: This work is supported through ANTARES project that has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement SGA-CSA. No. 739570 under FPA No. 664387 (https://doi.org/10.3030/739570). The authors acknowledge financial support from the Ministry of Education, Science, Technological Development and Innovation of the Republic of Serbia (Grant No. 451-03 66/2024-03/ 200358)