Sustainable Determination of VOC's in Water using Headspace GC-MS/MS

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Introduction

In modern analytical chemistry, increasing emphasis is placed on the development of methods aligned with the principles of green chemistry, while maintaining high sensitivity, selectivity, and reliability. This study presents the application of gas chromatography coupled with tandem mass spectrometry (GC-MS/MS) and Headspace technique for the identification and quantification of volatile organic compounds (VOCs) in water samples. VOCs are important indicators of pollution, originating from various sources such as industrial processes, landfills, petroleum products, and wastewater systems. Their presence in the environment is associated with numerous adverse effects on human health and aquatic ecosystems.

Methodology

The Headspace technique enables the extraction of volatile analytes from aqueous matrices without the use of organic solvents, thereby significantly reducing chemical waste generation and enhancing analytical safety. When combined with GC-MS/MS, this method provides highly specific and sensitive detection of target compounds at trace levels.

Principle of the method:

10 ml of water sample is transferred with a glass syringe into a 20 ml glass vial with stopper. Before use, the vial should be placed in a muffle furnace at 150 °C for one hour to eliminate any interference from volatile compounds.

A water sample of a certain volume is heated in a headspace vial closed with a special septum stopper. After reaching equilibrium between the aqueous and gas phases above the solution, a certain volume of the gas phase is transferred to a chromatographic column. Individual volatile hydrocarbons are separated by gas chromatography. Detection and quantification are performed by electron ionization (EI) mass spectrometry and the concentration in the sample is quantified using an external standard.

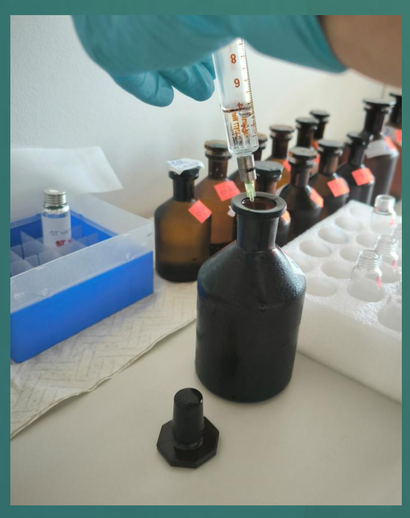
Water sampling and sample handling

Dark glass bottles with ground stoppers are used for water sampling. Plastic containers should be avoided during sampling, sample storage or extraction. Samples are stored at a temperature of up to 4 °C, protected from light, and the sample storage time should be shortened as much as possible. It is recommended to perform testing within 48 hours of sampling.

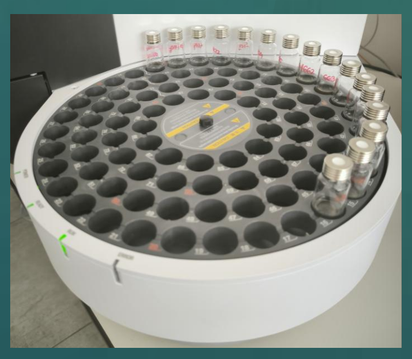
Picture 1. Dark glass bottles with ground stopper for VOC's

Picture 2. Shimadzu GCMS-TQ8050 NX for VOC's determination

Picture 3. Headspace vial



Picture 4. Sample preparation – transfer of samples with a glass syringe



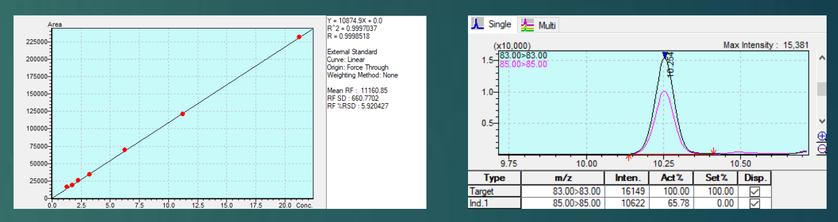
Picture 5. Headspace injector unit

Results

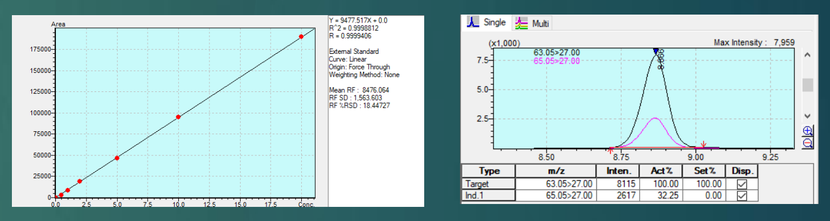
Table 1. shows validation data with LOD and LOQ values, RSD of preparation repeatability and recovery for clean, polluted and seawater.

VOC	LOD, µg/l	LOQ, µg/l	RSD, %	Recovery, %	RSD, %	Recovery, %	RSD, %	Recovery, %
Chloroform	0,010	0,09	6,4	96,8	6,6	94,9	12,5	114,7
1,1-dichloroethane	0,004	0,03	4,8	102,7	3,7	90,3	5,6	94,3
Carbon tetrachloride	0,006	0,06	5,5	120,0	3,7	97,8	8,9	93,1
1,2-dichloroethane	0,005	0,05	10,1	105,3	4,7	91,9	3,1	95,3
Trichloroethene	0,002	0,02	4,2	118,3	4,7	105,1	5,7	116,9
			Clean water		Polluted water		Seawater	

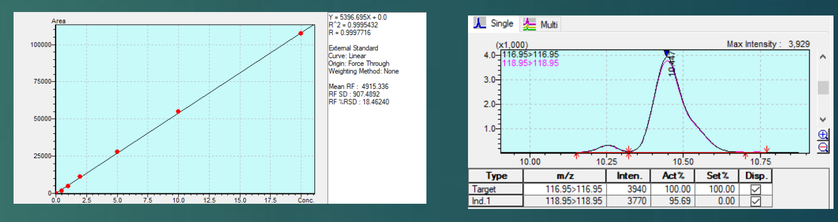
Table 1. Validation data for individual volatile compounds



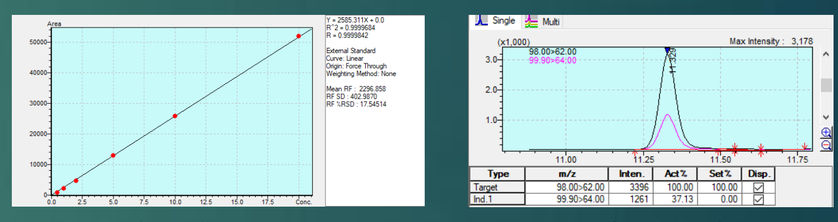
Picture 6. Calibration curve and chromatogram for chloroform



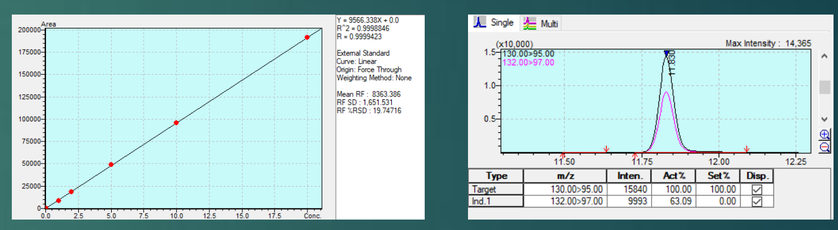
Picture 7. Calibration curve and chromatogram for 1,1-dichloroethane



Picture 8. Calibration curve and chromatogram for carbon tetrachloride



Picture 9. Calibration curve and chromatogram for 1,2-dichloroethane



Picture 10. Calibration curve and chromatogram for trichloroethene

Conclusion

The validation measurements included the determination of volatile organic compounds in drinking, waste and seawater, and from the obtained results it can be concluded that the method corresponds to its intended purpose and that it is applicable in laboratory conditions.

The application of GC-MS/MS combined with Headspace extraction has proven to be a highly effective method for the detection and quantification of volatile organic compounds (VOCs) in water samples. This analytical approach ensures high sensitivity and selectivity while fully aligning with the principles of green chemistry by minimizing the use of organic solvents and reducing chemical waste. Given the harmful effects of VOCs on human health and the environment, such methods represent a sustainable and reliable tool for routine water quality monitoring. In doing so, they contribute to reducing the ecological footprint of laboratory analyses and promote a more responsible approach to environmental and public health protection.

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