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INTERNATIONAL SCIENTIFIC AND PROFESSIONAL CONFERENCE

POLITEHNIKA 2023

CONFERENCE PROCEEDINGS

Belgrade, 15th December 2023

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FOREWORD

The International Scientific and Professional Conference POLITEHNIKA 2023 represents the seventh edition of the POLITEHNIKA scientific and professional events, occurring biannually since its inaugural event in 2011. POLITEHNIKA 2023 upholds a distinguished tradition and commitment to integrating higher education and practical application across a diverse spectrum of disciplines represented by defined thematic scopes.

Organized with the patronage of the Ministry of Education of the Republic of Serbia, the Ministry of Environmental Protection of the Republic of Serbia, the Ministry of European Integration of the Republic of Serbia, the Directorate for Occupational Safety and Health, the Office for Dual Education and National Qualifications Framework, the Conference of Academies of Applied Studies in Serbia, the Chamber of Commerce of Serbia, the Chamber of Commerce of Belgrade, the Institute for Standardization of Serbia, the Association of Belgrade Architects, the City of Požarevac and the Tourist Organization of the City of Požarevac, POLITEHNIKA 2023 stands as a collaborative platform at the intersection of academia, governmental institutions and industry.

This year heralds a notable progression with its international status and the incorporation of 10 conference scopes. Expanding beyond the thematic domains featured in previous events, the Conference now encompasses Environment and Sustainable Development, Occupational Safety and Health and Fire Safety, Smart Management Systems, Graphic Engineering, Design, Traffic Engineering, Biotechnology and Healthcare, Mechanical Engineering, Ecotourism and Rural development, and Mechatronics. By engaging experts, emerging professionals, and practitioners from these domains, the conference unifies fields of study programs of the Academy of Applied Technical Studies Belgrade. The thematic scopes, coupled with the structure of the compiled papers in this Proceedings, exhibit a rich diversity and multidisciplinary approach, fundamentally contributing to a holistic examination and resolution of societal and scientific challenges.

Comprising over 220 peer-reviewed contributions, the Proceedings represent a substantial intellectual asset, aligning with the conference's overarching objective of fostering the exchange of knowledge, research findings, and professional experiences among experts from industry, research institutions, and higher education establishments.

The Proceedings of the International Scientific and Professional Conference POLITEHNIKA 2023 serve as a comprehensive snapshot of the current landscape within the thematic realms of the conference, offering both insights and directives for ongoing scientific and professional development. Moreover, they proffer concrete solutions to practical challenges grounded in contemporary trends and pertinent insights.

The Academy of Applied Technical Studies Belgrade extends its sincere appreciation to all conference supporters whose financial contributions played a pivotal role in its successful realization. Special acknowledgment is reserved for the authors of the papers, whose diligence and eagerness to present their work to a wider audience, alongside the reviewers and members of the International Scientific Committee, Program Committee and Organizational Committee, have collectively contributed to the triumph of the International Scientific and Professional Conference POLITEHNIKA 2023.

Belgrade, December 2023
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INTERNATIONAL SCIENTIFIC AND PROFESSIONAL CONFERENCE
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ENVIRONMENT AND SUSTAINABLE DEVELOPMENT. MECHATRONICS. OCCUPATIONAL SAFETY AND HEALTH AND FIRE SAFETY. SMART MANAGEMENT SYSTEMS. GRAPHIC ENGINEERING. DESIGN. TRAFFIC ENGINEERING. BIOTECHNOLOGY AND HEALTHCARE. MECHANICAL ENGINEERING. ECOTOURISM AND RURAL DEVELOPMENT.

ANALYSIS OF BTEX IN SEDIMENTS BY PURGE-AND-TRAP GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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Abstract: *One of the most BTEX (benzene, toluene, ethyl benzene and a mixture of o-, m- and p-xylenes) compounds are prominent environmental pollutants commonly found in discharges and petroleum products. These volatile aromatic compounds (VOCs) are severely toxic to living organisms if contact is maintained. They originate from incomplete combustion of organic matter, and their major sources to the urban environment include vehicular emissions (mobile source), gasoline evaporation, coal burning and residential heating, and waste incineration (stationary sources). Water sediments are not well known as a source of BTEX. However, it is crucial to consider that they can act as a source, as well as sink, of organic compounds, especially in a water body located in an urban city exposed excessively to the above-mentioned sources of BTEX. In this study, we aimed to identify and quantitatively analyze VOCs compounds in sediment by using purge-and-trap (P&T) gas Chromatography-Mass Spectrometry (GC-MS).*
Keywords: VOCs; sediment analysis; GC-MS; toxicity

1. INTRODUCTION

With the rapid development of urbanization and industrialization during the last two decades, the number of pollutants has increased significantly, which can lead to severe environmental pollution in water bodies. Considering that pollutants, such as BTEX (benzene, toluene, ethyl benzene, o-, m-, p-xylenes), pose a high accumulation property in the body due to heavy exposure, they can cause various adverse health effects. In this regard, BTEX are discharged to aquatic environments from natural or anthropogenic sources and distributed between the aqueous phase and the sediments during their transport. Hence, investigating sediment contamination in terms of ecological and health risks of trace pollutants is crucial in predicting aquatic ecosystem risks [1,2].
BTEX are widely employed as degreasers and intermediates in chemical synthesis, fuels, and fuel additives. The presence of solvents in the environment, especially in water, has become a significant health concern for over three decades due to their potential carcinogenicity [3,4]. It is reported that BTEX in urban environments have harmful effects on the central nervous system, and benzene is classified by the International Agency for Research on Cancer (IARC) as a human carcinogen with pending assessment updates [5]. All the BTEX chemicals can produce neurological impairment, and exposure to benzene can additionally cause hematological effects including aplastic anemia and acute myelogenous leukemia. The critical nature of the neurotoxicity is reflected by the use of neurological

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impairment as the basis for 9 of 13 maximum residue levels (MRL) for BTEX chemicals [5]. The carcinogenic (leukemogenic) potential of benzene is well established, as indicated by its consensus classification as a human carcinogen by the National Toxicology Program (NTP 2001), U.S. Environmental Protection Agency (EPA) (IRIS 2001) [6], and International Agency for Research on Cancer (IARC).

BTEX predominantly originated from a wide array of products such as cleaning supplies, pesticides, building materials, office equipment, and craft materials, including glues and adhesives, photographic solutions, and tires. They are usually included in the composition of paints and lacquers, varnishes, and fuels and serve as precursors for producing other compounds. Aromatic hydrocarbons can be divided into monoaromatic hydrocarbons and polyaromatic hydrocarbons, whereas BTEX belongs to the group of monoaromatics with unique properties due to the delocalized electron density in benzene, including additional stabilization. BTEX are commonly found in crude oil and are used in geochemical investigations as direct indicators of the presence of oil and gas; they are easily volatile and can be degraded by microorganisms, which affect their precise measurement seriously.

Within this work, a procedure of gas chromatography-mass spectrometry (GC-MS), including purge and trap extraction (P&T), is used for the quantification of BTEX compounds in sediment samples [7,8]. The modification of several EPA standard methods for BTEX determination was made. Continuous monitoring of various parameters that may affect the performance of the method is necessary for ensuring an appropriate result quality. This primarily refers to specificity and selectivity, linearity, detection limit, precision, accuracy, and measurement uncertainty. It is emphasized how important it is to reflect the pollution status of BTEX comprehensively and evaluate the potential ecological risk of these analytes for water management in future studies.

2. MATERIAL AND METHODS

2.1 Reagents and laboratory supplies

The QA/QC of the analysis with scientifically acceptable results was ensured using the certified reference material (CRM) (Internal/Surrogate Standard Mix M-8260A-B-IS-SS-10X) for QC of VOCs analyses. CRM and all the reagent blanks were prepared by carrying out the entire analytical procedure and included as quality control samples. Concentrations were obtained as the average of three random measurements, whereas the analytical quality was evaluated by analysis of replicate. Methanol used for sediment sample preparation was of chromatographic grade. The solvent chromatogram showed no BTEX interfering peaks. The stock standard solutions were prepared in the appropriate solvent and stored in vials with Teflon-lined stoppers in the refrigerator. Intermediate mix standard solutions were prepared by diluting the stock standard solutions. Calibration standard solutions were prepared by diluting the intermediate standard solution. Since the exposure to BTEXs was to be minimized, pure standard materials and stock standard solutions were handled in a hood.

Sample containers of 40 ml t screw cap vials were used, each equipped with a Teflon-faced silicone septum. It was essential to wash vials and septa with detergent and rinse with tap and distilled water. The vials and septa were air-dried at room temperature, placed in an oven at 105^{o}C for 1 h, and cooled in a place free from organics.

2.2 Purge-and-Trap Gas Chromatography-Mass Spectrometry

Purge-and-Trap (P&T) Gas Chromatography-Mass Spectrometry (GC-MS) is the most widely used method in BTEX analysis [7]. A purge-and-trap system (Teledyne Tekmar ATOM XYZ) coupled with GC-MS (Agilent GC 7820A with MS 5977B) was used to analyze BTEX in sediment samples. After purging and cooling the trap with ultra-high-purity N_2, the samples were purged, trapped, and then introduced into GC-MS to measure BTEX [8,9].

2.2.1 P&T sampler method conditions

Purge: Prepurge 0.00 min; flow: 0 mL/min, Preheat Mix Speed: slow, sample preheat time: 0 min, Presweep time: 0.25 min, water vol: 10.0 mL, Sweep water time 0.25 min, Sweep water flow: 100 mL/min, Sparge vessel heather: off; Purge mix speed: Medium; purge time: 11.0 min, Purge flow: 40 ml/min, Purge and MCS Purge T = 20 °C; Dry Purge Time: 1.00 min, Dry Purge flow: 100 mL/min; Dry Purge T = 20 °C.
Desorb: Methanol Needle Rinse: Off; (V=0 mL); water Needle Rinse V = 7 mL; Sweep Needle time: 0.25 min; Desorb preheat T = 245 °C; GC Start Signal: Begin Desorb; Desorb time: 2.00 min; Drain flow: 300 mL/min, Desorb T = 250 °C.

2.2.2 GC-MS instrument method conditions

Column: Agilent DB-VRX, 20 m × 0.18 mm, 1 μm film, helium – 1 mL/min; oven profile: 35 °C, 4 min, 15 °C/min to 85 °C, 30 °C/min to 225 °C, 2 min hold, Run 14.0 min; Inlet: 180 °C, 120:1 Split, 19.752 psi; temperature: transfer line 225 °C; Source 230 °C; Quad 150 °C: scan: Range 35 m/z to 260 m/z, solvent delay: 0.5 min, normal scanning; gain: gain factor 10.00, autotune.

2.3 Interferences

Major potential contaminant sources in this kind of analysis are volatile materials in the laboratory and impurities in the inert purging gas and the sorbent trap. Using Teflon tubing, Teflon thread sealants, or flow controllers with rubber components in the purging device is avoided since such materials out-gas organic compounds, which will be concentrated in the trap during the purge operation. Interfering contamination may occur when a sample containing a low concentration of BTEX is analyzed immediately after a sample with high BTEX concentrations. Preventive action of thorough rinsing of the purging apparatus and sample syringes with two water portions was conducted, as well as one or more reagent blanks were analyzed to check for cross-contamination [9]. Another potential source of contamination from traces comes from organic solvents (i.e., the highest purity methanol), which was assessed before the standard preparation in methanol.

3. RESULTS AND DISCUSSION

The specificity and selectivity of the method were determined based on the retention times of peaks and ion characteristics for each analyte (quantifier and qualifier ions).

Table 1. Retention times and selected ions of the target analytes
(Masses for P&T GC-MS VOC and ISTD)

No.	Analyte	RT	Quantifier	Qualifier	Qualifier	Qualifier
1.	Benzene	5.632	78	52	39	77
2.	Toluene	7.827	91	65	39	92
3.	Ethyl benzene	9.003	91	106	65	51
4.	m – xylene**	9.134	91	106	77	51
5.	p – xylene*	9.134	91	106	77	51
6.	o - xylene	9.363	91	106	77	51

** m- and p-xylene (the same RT, as one peak), so the result is given as a total result for m, p-xylene*

Linearity was tested in a range of different concentrations for each analyte individually on a series of standard solutions prepared according to a standard procedure. Linearity is confirmed because the correlation coefficient (R^2) is greater than 0.999.

Table 2. Linearity with and the corresponding values of the coefficient correlation R^2

Analyte	y= Ax+B	R^2
Benzene	y = 212005.8 x + 2802.4	0.9996
Toluene	y = 292823.9 x + 4918.1	0.9994
Ethyl benzene	y = 104198.1 x + 3631.2	0.9992
m,p - xylene	y = 174957.8 x + 6926.3	0.9992
o - xylene	y = 86776.6 x + 3243.8	0.9991

The values of the correlation coefficient R^2 exceed the value of 0.99, so they can be considered sufficiently close to 1.0; therefore, the dependence can be considered linear. The detection limits were determined from seven repetitions of the concentration standard of 1.0 µg/l and based on the equation: MDL = S*t(n-1,1-alpha = 0.99) where: t(n-1,1-alpha = 0.99) – Student's t value for 99% confidence level and n-1 degrees of freedom, S – standard deviation of repeated analysis.

Table 3. Values of standard deviation and limit of detection (t=3.143 for n-1=6)

Analyte	STDEV	MDL
Benzene	0.012	0.04
Toluene	0.010	0.03
Ethyl benzene	0.012	0.04
m,p - xylene	0.013	0.04
o - Xylene	0.010	0.03

Precision and accuracy were determined by running 6 spiked samples at 0.1 µg/kg. Accuracy is determined by calculating the standard deviations and relative standard deviations.

Table 4. Parameters of precision and accuracy at the level of 0.1 µg/kg

Analyte	Mean	STDEV	RSD (%)	Bias	Bias (%)	Recovery (%)
Benzene	1.082	0.0823	7.6	0.082	8.2	108.2
Toluene	0.995	0.0938	9.4	-0.005	-0.5	99.5
Ethyl benzene	0.98	0.0901	9.2	-0.02	-2.0	98.0
m,p - xylene	0.947	0.0641	6.8	-0.053	-5.3	94.7
o - xylene	1.067	0.0963	9.0	0.067	6.7	106.7

The obtained results show that the requirements of the EPA are fulfilled because recoveries were in the range of 80-120%, and the relative standard deviations were less than 20%.

Table 5. Values of measurement uncertainties at the level of 0.1 µg/kg

Analyte	u(Rw), %	u(bias), %	u (%)	U (%)
Benzene	7.6	8.9	11.7	23.4
Toluene	9.4	4.0	10.3	20.5
Ethyl benzene	9.2	4.4	10.2	20.3
m,p - xylene	6.8	6.1	9.1	18.2
o - xylene	9.0	7.8	11.9	23.9

*where *u is the total (combined) measurement uncertainty, while the extended measurement uncertainty is U = u* 2*

There are no specified special EPA requirements for measurement uncertainty, but considering that it is determined based on accuracy and precision that meet the prescribed criteria, it can be considered that the values of measurement uncertainties are also satisfactory.

Table 6. Calibration, Accuracy, and Precision Data

Compound	Calibration			Accuracy and precision (n = 7, 0.1 µg/kg)[1]		
	Linearity RF (%RSD)	MDL (ppb)	Avg. RF	Avg. Conc. (ppb)	Accuracy (%)	Precision (%RSD)
Benzene	5.48	0.12	1.510	0.85	87	4.51
Toluene	3.39	0.13	1.24	0.95	95	4.44
Ethylbenzene	5.48	0.22	1.50	0.88	90	7.91
m-, p-xylene	9.22	0.43	1.15	1.7	89	7.51
o-xylene	6.57	0.20	1.17	0.89	88	7.03

Data from the seven 0.1 µg/kg samples

4. CONCLUSION

This study confirmed the capability of the use of the P&T system for the determination of BTEX concentrations in the sediment samples with a developed method by a GC-MS. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL, precision, and accuracy showed no interference from excessive water. The procedure of method verification showed that the obtained values are satisfactory and that the applied method can be used to analyze BTEX in sediment samples.

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