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- III-O-5.** ULTRASONIC NEBULISER, A USEFUL TOOL FOR IMPROVING THE SENSIBILITY OF TRACE ELEMENT DETECTION IN SURFACE WATER WITH AXIAL VIEWING ICP-EOS SPECTROMETER
(Full paper will be published in *Journal of Environment and Ecology-JEPE*)
Gabriela Geanina Vasile, Nicoleta Mirela Marin,
Jana Petre, Liliana Valeria Cruceru 126
- III-O-6.** NEW LC-MS/MS METHOD FOR DETERMINATION OF EIGHT NITROSAMINES IN DRINKING WATER
(Full paper will be published in *Journal of Environment and Ecology-JEPE*)
Toma Galaon, Liliana Cruceru, Jana Petre, Luoana
Florentina Pascu, Vasile Ion Iancu, Marcela Niculescu 127
- III-O-7.** ASSESSMENT OF THE POTENTIAL ECOLOGICAL RISK WITH HEAVY METALS IN SURFACE SEDIMENTS FROM ACCUMULATION LAKES ON THE SECTOR INFERIOR OF THE OLT RIVER
Mihaela Iordache, Luisa Roxana Popescu,
Luoana Florentina Pascu, Ioan Iordache 128
- III-O-8.** OCCURRENCE OF SOME PESTICIDES IN THE DISSOLVED WATER PHASE OF THE DANUBE RIVER AND ITS THREE MAJOR TRIBUTARIES
Vasile Ion Iancu, Toma Galaon, Jana Petre, Liliana Cruceru,
Luoana Florentina Pascu 129
- III-O-9.** A NEW APPROACH FOR THE EXTRACTION OF ORGANIC COMPOUNDS FROM DIFFERENT TYPES OF CONTAMINATED WATERS AND SEWAGE SLUDGE
Andrei Niculae 130
- III-O-10.** RESEARCH ON THE RELATIONSHIP BETWEEN URBAN AIR POLLUTION AND NOISE LEVELS IN AREAS WITH HEAVY TRAFFIC
Mihai Bratu, Valeriu Danciulescu, Elena Bucur,
Andrei Vasile 131
-

- III-O-11.** DETERMINATION OF FORMIC AND ACETIC ACIDS IN INDOOR AIR
Raluca Diodiu, Elena Bucur 132
- III-O-12.** THE STRUCTURE OF THE BIOTIC COMMUNITIES IN AQUATIC ECOSYSTEMS FROM DANUBE RIVER
Elena Stanescu, Daniel Scradeanu, Irina Lucaciu,
Alina Roxana Banciu, Jana Petre, Bogdan Stanescu 133
- posters -** 135
- III-P-1.** ASPECTS OF THE UNCERTAINTY OF MEASUREMENT RESULTS BY GC – MS WITH APPLICATION TO PCNB – INTERNAL STANDARD USED IN ORGANOCHLORINE PESTICIDE DETERMINATION
Luminita Barbu, Emilia Teaca 135
- III-P-2.** ACID-BASE PROPERTIES OF SODIUM SALT OF 4-PHENYLSEMICARBAZONE 1,2-NAPHTHOQUINONE-4-SULFONIC ACID
Oksana Zagurskaya-Sharaevskaya, Igor Povar 136
- III-P-3.** VOLTAMMETRIC TECHNIQUES APPLIED TO THE STUDY OF [Cu₂(DH)₄ g,g-bipy] ELECTROCHEMICAL BEHAVIOR
Tatiana Cazac 137
- III-P-4.** BUFFER ACTION OF NATURAL SYSTEMS “IRON (III) MINERAL –SOIL SOLUTION”
Igor Povar, Oxana Spînu 138
- III-P-5.** STUDY THE ACID-BASE PROPERTIES OF PHARMACEUTICAL PREPARATION OF ENOXIL
Tudor Lupascu, Nina Tîmbaliuc 140
-

III-O-5. ULTRASONIC NEBULISER, A USEFUL TOOL FOR IMPROVING
THE SENSIBILITY OF TRACE ELEMENT DETECTION IN SURFACE
WATER WITH AXIAL VIEWING ICP-EOS SPECTROMETER

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Abstract

In order to analyze trace elements from surface water, drinking water and waste water an analytical method using Ultrasonic Nebulizer USN U5000AT⁺ equipment coupled with ICP-EOS Optima 5300 DV Perkin Elmer Spectrometer has been developed. Quality parameters (LOD, LOQ) allow the detection on sensitivity level imposed by European Legislation. Optimized parameters for simultaneous determination of Cd, Cr, Cu, Co, Ni and Pb in the range 1 to 5 µg/L were: power setting of 1400 W; auxiliary gas of 0.2 L/min; nebulizer gas of 0.75 L/min; sample uptake rate of 1.9 mL/min; plasma gas rate 15 L/min. Quantification limits obtained using fortified blanks indicated low values such as: 0.075 µg/L (Cd, Co); 0.10 µg/L for (Ni, Pb); 0.15 µg/L (Cr, Cu). The use of an USN as sample introduction system instead of Meinhardt Classic Nebulizer improves LOQ more than 10 times.

Keywords: ICP-EOS, metals, simultaneous determination, ultrasonic nebulizer, water quality

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III-O-6. NEW LC-MS/MS METHOD FOR DETERMINATION
OF EIGHT NITROSAMINES IN DRINKING WATER

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Abstract

A new sensitive, selective and accurate LC-MS/MS method with positive electrospray ionization was developed to detect eight nitrosamines from drinking water. The method separates all nitrosamines using a Zorbax Eclipse Plus C18 column (100 x 2.1 mm, 3.5 µm) kept at 15°C and a mobile phase consisting of aq. 0.005% formic acid and acetonitrile in the ratio 90/10 (v/v). A strong gradient applied in 8 minutes up to 90% ACN allowed analyte separation. A low mobile phase flow-rate of 0.2 mL/min was used to enhance ESI ionization. Collision energy, fragmentor and capillary voltages were optimized to enhance analyte S/N ratio. Optimization of LC-MS parameters generated low instrumental LOQ values between 0.05 – 1.84 µg/L. Detector response was linear in the range 1 – 100 µg/L ($R^2 > 0.99$) for all nitrosamines. SPE using activated charcoal cartridges was used to concentrate nitrosamines from drinking water. Overall method LOQs were situated in the range 1.23 – 4.12 ng/L. LC-MS/MS method was fully validated with respect to specificity, linearity, precision, accuracy and LOQ, and provided good results. The method was tested on six tap water samples collected from different regions of Bucharest and the determined nitrosamine total content ranged between 2.8 and 14.8 ng/L.

Keywords: drinking water, electrospray, LC-MS/MS, nitrosamines, solid-phase extraction