



11th NATIONAL MEETING ON CHROMATOGRAPHY

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Faculdade de Ciências e Tecnologia,
Universidade NOVA de Lisboa



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15:40 O10 *The Use of Ion Mobility-MS to Resolve and Discover Sample Complexity In Small Molecule Analysis*

Alberto Méndez¹

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16:05 O11 *Analysis of skin volatiles using a membrane-SPME/GC-MS approach to unveil putative biomarkers for neurodegenerative diseases*

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16.25 Coffee Break & Posters Session

AFTERNOON SESSION

Session 4 Chair: Cristina Dias - Universidade de Évora

17:00 O12 *Determination of the phenolic composition of vine-canapes subcritical water extracts and its utilization for production of a topical formulation*

Manuela M. Moreira¹, Francisca Rodrigues¹, Olena Dorosh^{1,2}, Diana Pinto,¹ Andreia F. Peixoto,³ Paulo Costa⁴, Simone Morais¹, Cristina Freire³, Cristina Delerue-Matos¹

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17:20 O13 *HPLC and UHPLC Selectivity – Finding a Selectivity Starting Point*

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17:45 O14 *Separation of Nadolol Racemates by High pH Reversed-Phase Fixed-Bed and Simulated Moving Bed Chromatography*

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18:05 O15 *Pharmaceutical drugs as emerging pollutants in aqueous media of Northeast Portugal*

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O15 Pharmaceutical drugs as emerging pollutants in aqueous media of Northeast Portugal

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Emerging pollutants are potentially toxic substances that although found in very small concentrations can produce hazard effects to the environment. Due to their very small concentrations they are not yet included in the water quality monitoring programs neither in national or international environmental control regulations. Pharmaceutical and Personal Care Products (PPCPs) represent an important group of emerging pollutants owing to increased worldwide consumption and to their inherent capacity to induce physiological harmful effects in very low doses, which raises several concerns related with the potential adverse effects on humans, animals and environmental systems.¹

In this work, it will be presented the development and validation of a complete experimental methodology proposed for the monitoring of pharmaceutical drugs. The method is based on solid phase extraction (SPE) followed by analysis with high performance liquid chromatography with a diode array detector (HPLC-DAD)². Experimental results obtained with two different columns will be presented. An analytical Nucleosil 100-5 C18 column, 150 mm x 4.6 mm, obtained from Macherey-Nagel for compounds with lower pKa values and a SiliaChrom XT C18 column, 4.6 mm x 250 mm, obtained from SiliCycle for compounds with higher pKa values. The method is validated by the analysis of real aqueous matrices samples obtained from different water media sources, such as, swimming pools, rivers and wastewater treatment plants. To extend the scope of the analytical method and thus obtain a broader study, several drugs were selected, belonging to five different pharmacological classes: non-steroidal anti-inflammatory (ibuprofen, acetylsalicylic acid, ketoprofen, naproxen and diclofenac), analgesic (paracetamol), antibiotic (sulfamethoxazole), an anticonvulsant (carbamazepine) and a central nervous system stimulator (caffeine). These compounds were selected due to their high level of use and medical prescription and, consequently, leading to a high probability of environmental contamination. Figure 1 shows the overlay chromatograms of individual drugs standards with Nucleosil 100-5 C18 column and a concentration of 100 ppm in the optimum wavelength. Figure 2 represents the chromatograms of a mixture of four selected drugs standards (sulfamethoxazole, paracetamol, caffeine and carbamazepine) with a SiliaChrom XT C18 column and 100 ppm concentration using the optimum wavelength for each compound.

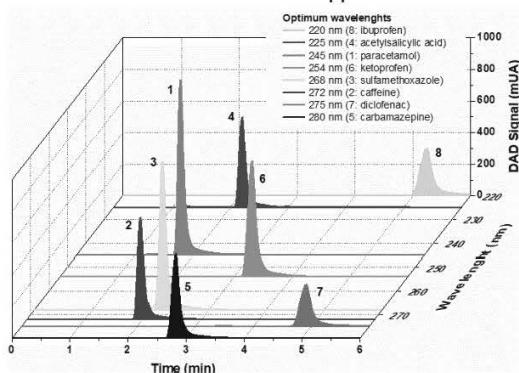


Figure 1. HPLC-DAD chromatograms of eight individual standards of pharmaceutical drugs using a Nucleosil 100-5 C18 column and 60%acetonitrile:40%water:0.01%trifluoracetic acid (pH=2.5) solvent composition. Numbers represent elution order.

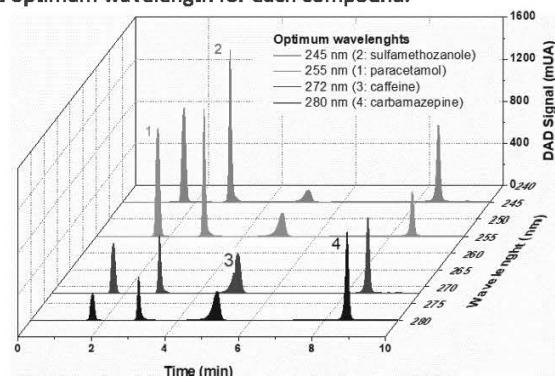


Figure 2. HPLC-DAD chromatograms of a standard mixture of four selected pharmaceutical drugs using a SiliaChrom XT C18 column and methanol:water:0.005%diethylamine (pH=10.6) solvent composition with gradient operation. Numbers represent elution order.

References:

- Ebele A. J.; Abdallah M. A.; Harrad S. *Emerging Contaminants* 2017, 3, 1.
- Oliveira A.; Nemoto A.; Ribeiro A.; Brito P.; Queiroz A. Extraction and quantification of pharmaceutical drugs in aqueous matrices. XXVI Encontro Nacional da Sociedade Portuguesa de Química, Porto, Julho de 2019, 229.