11º CONGRESSO NACIONAL DE CROMATOGRAFIA

20 anos CROMATOGRAFIA

11th NATIONAL MEETING ON CHROMATOGRAPHY

9|11 Dezembro 2019

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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 Méndez[1]
[1] Waters Corporation

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

Beatriz Andrade[1], Jorge Pereira[1], José Câmara[1,2]
[1] CQM – Centro de Química da Madeira, Universidade da Madeira, Campus Universitário da Penteada, 9020-105 Funchal, Portugal
[2] Faculdade de Ciências Exatas e da Engenharia, Universidade da Madeira, Campus Universitário da Penteada, 9020-105, Funchal, Portugal

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-canes subcritical water extracts and its utilization for production of a topical formulation***

Manuela M. Moreira[1], Francisca Rodrigues[1], Olena Dorosh[1,2], Diana Pinto,[1] Andreia F. Peixoto,[3] Paulo Costa[4], Simone Morais[1], Cristina Freire[3], Cristina Delerue-Matos[1]
[1] REQUIMTE/LAQV, Instituto Superior de Engenharia do Porto, Rua Dr. António Bernardino de Almeida 431, 4200-072, Porto, Portugal
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[3] REQUIMTE/LAQV, Dep. de Química e Bioquímica, Faculdade de Ciências, Universidade do Porto, Rua do Campo Alegre s/n, 4169-007 Porto, Portugal
[4] UCIBIO/REQUIMTE, MedTech-Laboratory of Pharmaceutical Technology, Department of Drug Sciences, Faculty of Pharmacy, University of Porto

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

Zeshan Aqeel[1], Felipe Silva[2], PhD. Jason Anspach[1], and Ryan Splitstone[1]
[1] Phenomenex, Inc., 411 Madrid Ave., Torrance, CA 90501 USA
[2] Phenomenex C/ Valgrande 8, planta 2.1.B., 28108 Alcobendas, Madrid, Spain

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

R. Arafah[1,2], A. Ribeiro[1,2], A. Rodrigues[2], L. Pais[1,2]
[1] Centro de Investigação de Montanha (CIMO), Instituo Politécnico de Bragança, Campus de Santa Apolónia, Apartado 1134, 5301-857 Bragança, Portugal
[2] Laboratory of Separation and Reaction Engineering, Department of Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias s/n, 4200-465 Porto, Portugal

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

A. Oliveira[1], A. Ribeiro[1,2], P. Brito[1], A. Queiroz[1]
[1] Centro de Investigação de Montanha (CIMO), Instituto Politécnico de Bragança, Campus de Santa Apolónia, 5300-253 Bragança, Portugal
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Index

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

A. Oliveira[1], A. Ribeiro[1,2], P. Brito[1], A. Queiroz[1]

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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.

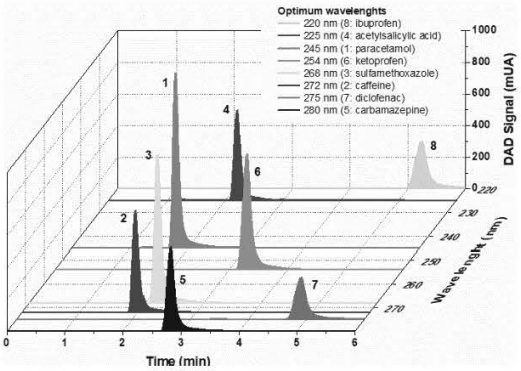

Figure 1. HPLC-DAD chromatograms of eight individual standards of pharmaceutical drugs using a Nucleosil 100-5 C18 column and 60%acetonitrile:40%water:0.01%trifluoracetic acid (pH=2.5) solvent composition. Numbers represent elution order.

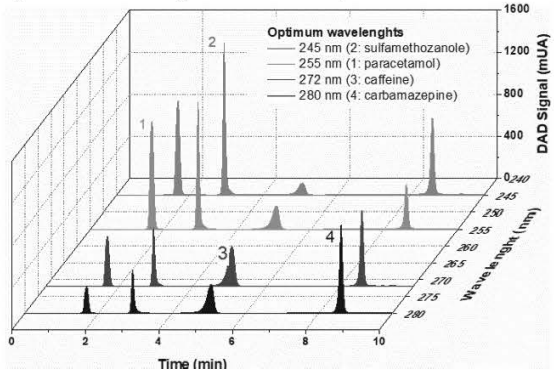

Figure 2. HPLC-DAD chromatograms of a standard mixture of four selected pharmaceutical drugs using a SiliaChrom XT C18 column and methanol:water:0.005%diethylamine (pH=10.6) solvent composition with gradient operation. Numbers represent elution order.

References:

1. Ebele A. J.; Abdallah M. A.; Harrad S. *Emerging Contaminants* **2017**, *3*, 1.

2. 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.